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Mechanical Construction**

**Title**

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**Thermo-Mechanical Behavior of Bio-Composite  
Materials Based on Date Palm Fibers**

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Presented by:

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## ملخص

تستكشف هذه الأطروحة للدكتوراه تطوير وتقييم أداء المواد المركبة الحيوية، وتحديدًا تلك المقواة بألياف نخيل التمر (DPFs) والجبس. وتبحث الأطروحة في إمكاناتها كبديل مستدامة في التطبيقات الهندسية، لا سيما في مجالي البناء والعزل. ووقد استخدمت مقاربة منهجية لتصنيع مواد مركبة حيوية (GF.s.m) بأحجام متفاوتة (s: I-III) من ألياف نخيل التمر (DPFs) ونسب كتلة (m: 0.5-20%) لكل حجم. وتُحلل الأطروحة بشكل شامل الخصائص الميكانيكية والفيزيائية والحرارية والكيميائية والمورفولوجية لهذه المواد المركبة الحيوية (GF.s.m). وتكشف النتائج الرئيسية عن وجود علاقة عكسية بين نسبة كتلة ألياف نخيل التمر (DPFs) وحجمها، مع تسليط الضوء على خصائص العزل الحراري المحسنة لألياف نخيل التمر (DPFs). وتُستخدم تقنيات التحليل الإحصائي والتحسين متعدد الأهداف (MOO) لتحديد حلول تصميمية مثلى للمواد المركبة الحيوية وفي نهاية المطاف، تساهم هذه الأطروحة في تعزيز المعرفة حول المواد المركبة الحيوية وتوضح إمكاناتها للتطبيقات الصديقة للبيئة وعالية الأداء في صناعات البناء والتصنيع.

**الكلمات المفتاحية:** ألياف نخيل التمر (DPFs)، الجبس، المواد المركبة الحيوية، الحجم، نسبة الكتلة، التحليل الإحصائي، والتحسين متعدد الأهداف.

## Abstract

This doctoral thesis explores the development and performance evaluation of bio-composite materials, specifically those reinforced with Date Palm Fibers (DPFs) and gypsum. The thesis investigates their potential as sustainable alternatives in engineering applications, particularly in construction and insulation. A systematic approach was employed to manufacture bio-composite materials (GF.s.m) with varying sizes (s: I-III) of Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) for each size. The thesis comprehensively analyzes the mechanical, physical, thermal, chemical, and morphological properties of these bio-composite materials (GF.s.m). Key findings reveal an inverse relationship between the mass ratio of Date Palm Fibers (DPFs) and size, while highlighting the enhanced thermal insulation properties of Date Palm Fibers (DPFs). Statistical analysis and Multi-Objective Optimization (MOO) techniques are used to identify optimal bio-composite materials design solutions. Ultimately, the thesis contributes to the growing knowledge of knowledge on bio-composite material and demonstrates its potential for eco-friendly, high-performance applications in the construction and manufacturing industries.

**Keywords:** Date Palm Fibers (DPFs), Gypsum, Bio-Composite Material, Size, Mass Ratio, Statistical Analysis, and Multi-Objective Optimization (MOO).

## Résumé

Cette thèse de doctorat explore le développement et l'évaluation des performances des matériaux bio-composites, notamment ceux renforcés par des Fibres de Palmier Dattier (FPDs) et du gypse. Elle étudie leur potentiel en tant qu'alternatives durables pour des applications d'ingénierie, notamment en construction et en isolation. Une approche systématique a été utilisée pour fabriquer des matériaux bio-composites (GF.s.m) avec différentes tailles (s : I-III) de Fibres de Palmier Dattier (FPDs) et différents rapports massiques (m : 05-20 %) pour chaque taille. La thèse analyse en profondeur les propriétés mécaniques, physiques, thermiques, chimiques et morphologiques de ces matériaux bio-composites (GF.s.m). Les principaux résultats révèlent une relation inverse entre le rapport massique des Fibres de Palmier Dattier (FPDs) et leur taille, tout en soulignant les propriétés d'isolation thermique améliorées de ces Fibres de Palmier Dattier (FPDs). Une analyse statistique et des techniques D'optimisation Multi-Objectifs (OMO) sont utilisées pour identifier les solutions de conception optimales pour les matériaux bio-composites. En définitive, cette thèse contribue à l'enrichissement des connaissances sur les matériaux bio-composites et démontre leur potentiel pour des applications écologiques et performantes dans les secteurs de la construction et de la fabrication.

**Mots Clés:** Fibres de Palmier Dattier (FPDs), Gypse, Matériau Bio-composite, Taille, Rapport de Masse, Analyse Statistique, et Optimisation Multi-Objectif.

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ قَالَ اللَّهُ تَعَالَى: "وَلَقَدْ ءَاتَيْنَا لُقْمَانَ الْحِكْمَةَ أَنْ اشْكُرْ لِلَّهِ وَمَنْ يَشْكُرْ فَإِنَّمَا يَشْكُرُ لِنَفْسِهِ وَمَنْ كَفَرَ فَإِنَّ اللَّهَ غَنِيٌّ حَمِيدٌ". سورة لقمان الآية رقم 12.

In the name of Allah, the Most Gracious and the Most Merciful Allah Almighty said: "Indeed, We blessed Luqmân with wisdom, 'saying', "Be grateful to Allah, for whoever is grateful, it is only for their own good. And whoever is ungrateful, then surely Allah is Self-Sufficient, Praiseworthy". Surah Luqman, verse 12.

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**Abderrahmane SAHRAOUI**

## **Dedication**

I would like to dedicate this thesis to my  
father, mother, brothers, friends,  
colleagues, comrades, and all those who have  
passed away. I hope that this thesis and all  
my future academic works will be a treasure  
for the Islamic nation.

**Abderrahmane SAHRAOUI**



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### Part 2

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# Statistical Analysis and Optimization Study

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## **List of Abbreviations**

|           |                                                                                                                                                         |
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| AFNOR     | French Standards Association.                                                                                                                           |
| ANOVA     | Analysis of Variance.                                                                                                                                   |
| ASTM      | American Society for Testing and Materials.                                                                                                             |
| ATR       | Attenuated Total Reflectance.                                                                                                                           |
| COLPA     | Cosider Lafarge Plasters Algeria.                                                                                                                       |
| DPF       | Date Palm Fiber.                                                                                                                                        |
| DPFs      | Date Palm Fibers.                                                                                                                                       |
| DPFM      | Date Palm Fiber Mesh.                                                                                                                                   |
| DPFR      | Date Palm Rachis Fibers.                                                                                                                                |
| DPFW      | Date Palm Fibers Waste.                                                                                                                                 |
| DPP       | Date Palm Petiole.                                                                                                                                      |
| EDS       | Energy Dispersive X-ray Spectroscopy.                                                                                                                   |
| EPS       | Expanded Polystyrene Waste.                                                                                                                             |
| FGD       | Flue-Gas Desulfurization.                                                                                                                               |
| FT-IR     | Fourier Transform Infrared Spectroscopy.                                                                                                                |
| GF.I.05   | Gypsum-Date Palm Fiber Bio-Composite Material with 0.080 mm Size and 05 % Mass Ratio.                                                                   |
| GF.I.10   | Gypsum-Date Palm Fiber Bio-Composite Material with 0.080 mm Size and 10 % Mass Ratio.                                                                   |
| GF.I.15   | Gypsum-Date Palm Fiber Bio-Composite Material with 0.080 mm Size and 15 % Mass Ratio.                                                                   |
| GF.I.20   | Gypsum-Date Palm Fiber Bio-Composite Material with 0.080 mm Size and 20 % Mass Ratio.                                                                   |
| GF.II.05  | Gypsum-Date Palm Fiber Bio-Composite Material with 0.315 mm Size and 05 % Mass Ratio.                                                                   |
| GF.II.10  | Gypsum-Date Palm Fiber Bio-Composite Material with 0.315 mm Size and 10 % Mass Ratio.                                                                   |
| GF.II.15  | Gypsum-Date Palm Fiber Bio-Composite Material with 0.315 mm Size and 15 % Mass Ratio.                                                                   |
| GF.II.20  | Gypsum-Date Palm Fiber Bio-Composite Material with 0.315 mm Size and 20 % Mass Ratio.                                                                   |
| GF.III.05 | Gypsum-Date Palm Fiber Bio-Composite Material with 0.500 mm Size and 05 % Mass Ratio.                                                                   |
| GF.III.10 | Gypsum-Date Palm Fiber Bio-Composite Material with 0.500 mm Size and 10 % Mass Ratio.                                                                   |
| GF.III.15 | Gypsum-Date Palm Fiber Bio-Composite Material with 0.500 mm Size and 15 % Mass Ratio.                                                                   |
| GF.III.20 | Gypsum-Date Palm Fiber Bio-Composite Material with 0.500 mm Size and 20 % Mass Ratio.                                                                   |
| GF.s.m    | Bio-Composites Reinforced with Different Sizes (s: I, II, and III) of Date Palm Fibers at Different Mass Ratios (m: 05, 10, 15, and 20%) for each Size. |
| HB        | Brinell Hardness.                                                                                                                                       |
| ISO       | International Organization for Standardization.                                                                                                         |
| MC        | Moisture Content.                                                                                                                                       |
| MDPF      | Mortar Date Palm Fibers.                                                                                                                                |
| MeanSq    | Mean Squares.                                                                                                                                           |
| MENA      | Middle East and North Africa.                                                                                                                           |
| MOO       | Multi-Objective Optimization.                                                                                                                           |
| NGP       | Neat Gypsum Plaster.                                                                                                                                    |

## List of Abbreviations

---

|       |                                                                                |
|-------|--------------------------------------------------------------------------------|
| OLS   | Ordinary Least Squares.                                                        |
| OPEFB | Oil Palm Empty Fruit Bunch.                                                    |
| PF    | Polyester Fibers.                                                              |
| PLA   | Polylactic Acid.                                                               |
| PP    | Polypropylene.                                                                 |
| RILEM | The International Union of Laboratories and Experts in Construction Materials. |
| RMSE  | Root Mean Squared Error.                                                       |
| RS2   | Reinforcement Sieve 2.                                                         |
| SE    | Standard Error.                                                                |
| SEM   | Scanning Electron Microscopy.                                                  |
| SumSq | Sum of Squares.                                                                |
| tStat | t-Statistic.                                                                   |
| WAR   | Water Absorption Ratio.                                                        |
| XRD   | X-Ray Diffraction.                                                             |

# **General Introduction**

## **General Introduction**

In recent years, there has been a growing demand for sustainable and eco-friendly materials across various engineering sectors. Composite materials, known for their enhanced mechanical, physical, and chemical properties, have emerged as a promising solution to meet these demands. These materials, which are formed by combining two or more distinct components, offer superior performance compared to individual materials. From construction and automotive to aerospace and biomedical applications, composite materials are increasingly being utilized to address specific performance needs.

The objectives of this doctoral thesis address the environmental and material challenges associated with the underutilization of palm waste in the Biskra region of southeastern Algeria. The thesis focuses on eliminating the harmful practice of burning palm waste by transforming it into a valuable resource for the production of bio-composite materials. These materials, made from Date Palm Fibers (DPFs) and gypsum, aim to reduce atmospheric carbon levels and minimize environmental pollution. The thesis investigates the influence of varying fiber sizes (s: I-III) and mass ratios (m: 5-20%) of Date Palm Fibers (DPFs) on the mechanical, physical, thermal, thermo-physical, chemical, and morphological properties of gypsum-based bio-composites (GF.s.m). The goal of this thesis is to produce a bio-composite material with superior properties, including enhanced mechanical strength, lightweight, low water absorption, mold resistance, and insulation. Additionally, the thesis explores the optimal fiber size and mass ratio combinations that will maximize the performance of these bio-composites for potential applications. This thesis seeks to fill the gap in understanding the relationship between fiber reinforcement and overall material properties, ultimately contributing to sustainable practices in material science and environmental preservation. This thesis is composed of six chapters grouped into three parts.

This first chapter explores composite materials in depth, with particular emphasis on bio-composite materials. It begins with an overview of composite materials, including their definition and various types: nano-based, matrix-based, reinforcement-based, and bio-composite-based composites. The section on bio-composites delves into the different types of bio-composites, emphasizing the role of natural fibers. Natural fibers are defined and categorized, and their physico-mechanical and chemical properties are analyzed, along with methods for extracting these fibers for use in bio-composite materials. The first chapter further investigates the manufacturing techniques for bio-composite materials, including hand

lay-up, extrusion, compression molding, vacuum infusion, and filament winding. A comprehensive review of the applications of bio-composites highlights their diverse uses across industries. Finally, the advantages and disadvantages of bio-composite materials are discussed, providing a balanced perspective on their potential and limitations. This first chapter aims to offer valuable insights for researchers and industry professionals interested in advancing the development and application of bio-composite materials.

This second chapter emphasizes the application of Date Palm Fibers (DPFs) and gypsum in the development of bio-composite materials, highlighting their potential applications, properties, and benefits. The second chapter provides a comprehensive overview of Date Palm Fibers (DPFs), including their definition, types, and properties, followed by various extraction methods. The paper examines the applications of Date Palm Fibers (DPFs), emphasizing their role in bio-composite materials, and outlines both the advantages and disadvantages of using these fibers in such materials. Similarly, the second chapter discusses gypsum, detailing its definition, types, and properties, as well as extraction methods and applications. The advantages and disadvantages of incorporating gypsum into bio-composite materials are also reviewed. The final section of the second chapter delves into existing literature on bio-composite materials composed of palm fibers and gypsum, offering insights into current research and practical applications. This second chapter aims to provide valuable knowledge for researchers and industry professionals interested in sustainable material development using natural fibers and minerals.

The date palm is an important crop, especially in the Middle East and North Africa (MENA). Its fibers are a useful byproduct of the date production sector. These fibers are strong, light, and long-lasting, making them great for use in bio-composites [1-3]. They have both environmental benefits and mechanical features that make them useful for many different things [3, 4]. In addition, the leaves, fruit stalks, and trunk of the date palm are the main sources of Date Palm Fibers (DPFs). These fibers are usually long, thick, and resistant to breaking down, which makes them a strong and long-lasting material for reinforcing composite structures. Date palm has a lot of cellulose, hemicellulose, and lignin in its fibers. These fibers make it strong, flexible, and able to connect with matrix materials like biodegradable resins or synthetic polymers [5-7].

Furthermore, Date Palm Fibers (DPFs) are biodegradable, non-toxic, and derived from renewable resources. They help reduce the environmental impact of traditional composites made from petroleum-based materials. The fibers have a favorable strength-to-weight ratio, which makes Date Palm Fiber (DPF) composites lighter than many other materials. This is useful in the automotive, construction, and

packaging industries. Since Date Palm Fibers (DPFs) are often waste products from date palm cultivation, they are relatively inexpensive and contribute to waste reduction, offering an affordable alternative to synthetic fibers like glass or carbon. When combined with appropriate matrix materials, Date Palm Fibers (DPFs) can offer mechanical properties comparable to those of conventional synthetic fibers [3, 8]. They enhance the tensile strength, stiffness, and durability of the composites. The production of Date Palm Fiber (DPF) bio-composites reduces reliance on non-renewable resources and helps mitigate carbon emissions, as the fibers are biodegradable and help sequester carbon during their lifecycle [9-11].

Meanwhile, gypsum, a naturally occurring mineral composed primarily of calcium sulfate dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), is increasingly being explored as a key component in bio-composites due to its sustainability and versatile properties. Traditionally used in construction materials, particularly in gypsum and drywall, gypsum has gained attention in the field of bio-composites for its potential to enhance the performance and environmental profile of composite materials.

In addition, in bio-composites, when mixed with natural fibers like flax, jute, or hemp, and matrix elements like biopolymers or resins, gypsum is employed as a filler or strengthening ingredient. Adding gypsum to composite formulations can make some attributes better, such as mechanical strength, fire resistance, and biodegradability. It can also help lower the material's overall environmental impact.

Furthermore, gypsum is a natural material that is very common, safe, and easy to recycle. It is thought to be an environmentally acceptable replacement for petroleum-based fillers and reinforcements that are typically used in composites, like synthetic resins or glass fibers. Gypsum's natural ability to resist fire is one of its best features. Gypsum makes bio-composites more resistant to heat and flame, which makes them suitable for use in construction materials and other applications that need to be fireproof. Gypsum can make bio-composites stronger in terms of their stiffness, compressive strength, and capacity to hold their shape [12-14]. Gypsum helps make composite materials stronger when blended with natural fibers and bio-based matrices [15, 16]. Gypsum can hold and release moisture because it is hygroscopic. This helps keep humidity levels in check in places where it is critical to control moisture, like in building materials. This hygroscopic property makes gypsum useful for bio-composite applications in construction. Gypsum-based bio-composites can break down in the environment, making them a more eco-friendly choice than typical composites that are difficult to recycle or take a long time to break down [17, 18]. Gypsum waste is typically made as a byproduct in industries like building and mining [19, 20].

## General Introduction

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It may be used to make bio-composites, which cuts down on waste and uses a resource that would otherwise end up in landfills [19, 21].

Meanwhile, gypsum-Date Palm Fiber (DPF) bio-composites are a new type of eco-friendly material that combines natural fibers from date palms (*Phoenix Dactylifera*) with gypsum matrices. This combination uses the structural benefits of natural fibers and the natural features of gypsum, such as being fire-resistant and insulating against heat, to make eco-friendly alternatives to traditional building materials. Research has shown that adding Date Palm Fibers (DPFs) to gypsum composites makes them stronger in terms of compressive and flexural strength [3, 4, 11, 22]. These composites also have better thermal insulation properties, which makes them good for building projects that save energy [11, 17, 23]. Studies show that gypsum-Date Palm Fiber (DPF) composites have good characteristics; however, they may not work as well when they absorb moisture [11, 17, 23, 24]. Researchers have looked into surface treatments of fibers to remedy this problem and make the composites endure longer [3, 17, 25-27]. Using Date Palm Fibers (DPFs), which are often thought of as agricultural waste, in gypsum composites cuts down on waste and promotes the use of renewable resources in construction materials [11]. Adding Date Palm Fibers (DPFs) to gypsum matrices is a promising technique to develop building materials that last longer and hold heat and cold better. More research on fiber handling and composite manufacturing will improve their utility in construction.

The third chapter focuses on a comprehensive study of the properties and performance of bio-composite materials made from Date Palm Fiber (DPF) and gypsum matrix, focusing on experimental testing methods. The materials section in the third chapter outlines the components used, including Date Palm Fiber (DPF), gypsum matrix, and the resulting bio-composite material. The methodology section in the third chapter describes the various experimental tests conducted to evaluate the bio-composite material's mechanical, physical, thermal, chemical, and morphological properties. Mechanical tests include the flexural (3-point) test, compressive strength test, Brinell hardness test, and Charpy impact strength test, which provide insight into the bio-composite material's strength and resilience. Physical properties are assessed through bulk density, water absorption, thickness swelling, and moisture content tests, while thermal conductivity is examined through thermal tests. Chemical analysis includes Energy Dispersive X-ray Spectroscopy (EDS) and Fourier Transform Infrared Spectroscopy (FT-IR) tests, contributing to a deeper understanding of the bio-composite material's composition and behavior. Scanning Electron Microscopy (SEM) is employed for morphological testing to examine the microstructure of the bio-composite materials. The third chapter covers the key findings and the potential implications for future research and applications in bio-composite material development.

The fourth chapter concentrates on the detailed results of various tests conducted on bio-composite materials made from Date Palm Fibers (DPFs) and gypsum matrix. The results are organized into distinct categories, starting with the mechanical properties, which include flexural strength and modulus, compressive strength, Brinell hardness, and Charpy impact strength. These tests provide a comprehensive assessment of the bio-composite material's strength and durability under various loading conditions. The physical properties are also evaluated, with results for bulk density, water absorption, thickness swelling, and moisture content, providing insights into the bio-composite material's behavior in different environmental conditions. Thermal properties are explored through tests on thermal conductivity, thermal diffusivity, and specific heat, while the thermo-physical relationship between thermal conductivity and bulk density is also investigated. Chemical analysis is performed using Energy Dispersive X-ray Spectroscopy (EDS) and Fourier Transform Infrared Spectroscopy (FT-IR) to examine the elemental composition and molecular structure of the bio-composite material. Finally, morphological results obtained from Scanning Electron Microscopy (SEM) provide detailed images of the bio-composite material's microstructure. The fourth chapter synthesizes the key findings, highlighting the potential applications and implications of these bio-composite materials for various industries.

The fifth chapter concentrates on a detailed overview of the process of data collection, statistical analysis, and regression modeling, with a focus on the application of regression models for Multi-Objective Optimization (MOO). The first section of the fifth chapter discusses the collection of data, followed by an introduction to the regression models used in the analysis, including linear, quadratic, and pure-quadratic models. The fifth chapter outlines the process for estimating model coefficients, as well as the calculation of Standard Errors (SE), t-statistics, and p-values, which are critical for understanding the significance of the coefficients. Model fit statistics, such as the Root Mean Squared Error (RMSE), R-squared ( $R^2$ ), adjusted R-squared ( $R^2_{Adj}$ ), F-statistic, and p-value for the F-statistic, are used to evaluate the performance of the regression models. The Analysis of Variance (ANOVA) is also explored, detailing the Sum of Squares (SumSq), degrees of freedom, and Mean Squares (MeanSq), among other factors. Multi-Objective Optimization (MOO) is introduced, focusing on Pareto optimality, Pareto fronts, and trade-offs, to provide insights into the optimization of multiple objectives simultaneously. The MATLAB code used to implement the regression models, including data collection, regression analysis, model evaluation, and predictions, is also provided. The fifth chapter concludes with a summary of the findings and the final output from the regression models and optimization process.



## General Introduction

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The sixth chapter is devoted to a detailed analysis of the Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) of bio-composite materials, incorporating statistical analysis, model fit statistics, and Analysis of Variance (ANOVA) results for each property. The first section of the sixth chapter focuses on Charpy impact strength (G), providing statistical results, model fit statistics, and Analysis of Variance (ANOVA) outcomes, followed by the predicted results for this mechanical property. A similar approach is applied to water absorption (WAR), where statistical analysis, model fit statistics, Analysis of Variance (ANOVA) results, and predicted outcomes are discussed. The thermal conductivity ( $\lambda$ ) section also follows the same structure, offering insights into the statistical results, model fit statistics, Analysis of Variance (ANOVA), and predicted results. The sixth chapter concludes with Multi-Objective Optimization (MOO) results, detailing the optimization objective, Pareto optimal solutions, limiting solutions, and the best compromise solution. This comprehensive sixth chapter aims to provide a better understanding of the performance and optimization of bio-composite materials, with implications for their application in various industries.

Ultimately, this thesis contributes to the growing body of knowledge in the field of bio-composite materials, offering a comprehensive analysis of the development, characterization, and optimization of bio-composites made from Date Palm Fibers (DPFs) and gypsum. Through this work, we aim to contribute to the advancement of sustainable material science and promote the use of eco-friendly materials in engineering applications.

In conclusion, as shown in **Figure 01**. The following diagram explains the most important aspects of the three parts of the thesis:

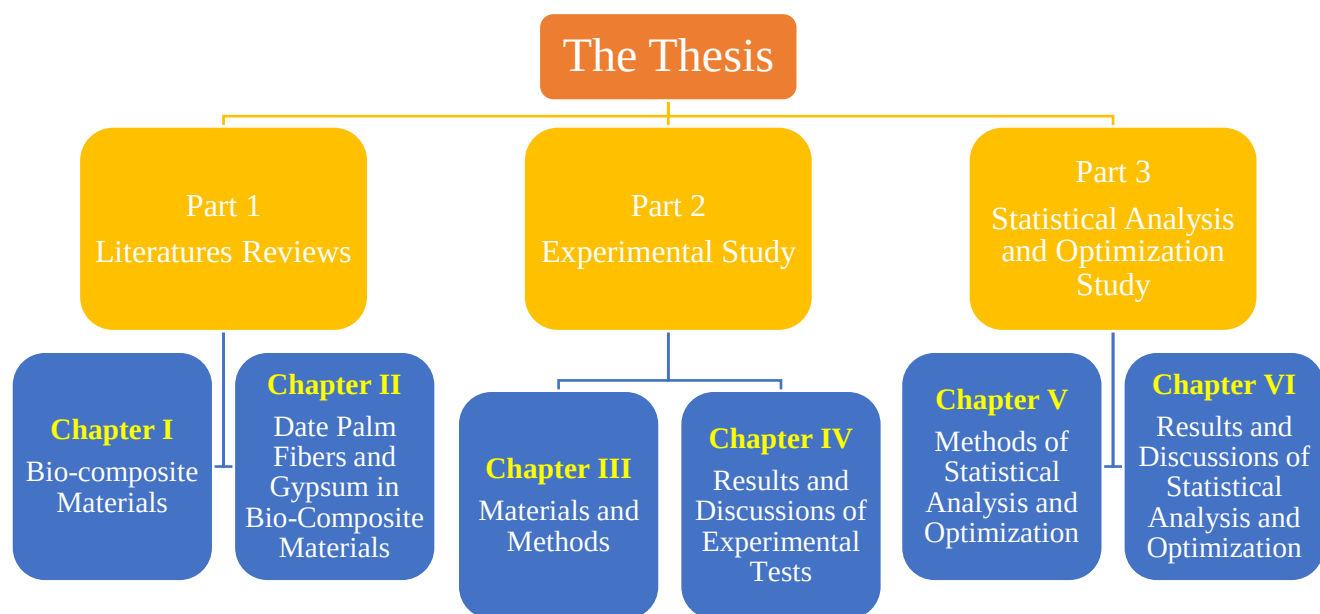


**Figure 01. Graphical Abstract of the Thesis.**

**Part 1**

**Literature Reviews**

## Introduction

The growing global demand for sustainable and eco-friendly materials has catalyzed significant advancements in the field of composite materials. Composites, defined as materials formed by combining two or more distinct components, offer superior mechanical, physical, and chemical properties that are unattainable by individual materials. These materials are widely utilized across industries such as construction, automotive, aerospace, and biomedical fields. Among the various types of composite materials, bio-composites, which integrate natural fibers into the matrix, have gained particular attention due to their environmentally friendly characteristics and potential for sustainable production.

The first part of this thesis focuses on the exploration of composite materials, particularly bio-composites, which are at the forefront of sustainable material science. The first chapter introduces the concept of composite materials, detailing their various types, including matrix-based, reinforcement-based, and nano-based composites. A special emphasis is placed on bio-composites, with a focus on the role of natural fibers and their properties, such as mechanical and chemical characteristics. The methods of extracting natural fibers for bio-composites are explored, alongside traditional and modern manufacturing techniques, including hand lay-up, extrusion, and compression molding.

The second chapter narrows its focus to specific components used in bio-composite materials, namely Date Palm Fibers (DPFs) and gypsum. Date Palm Fibers (DPFs), a natural by-product of the date palm tree, are highlighted for their abundance, biodegradability, and favorable mechanical properties, which make them valuable as a reinforcement material in composites. Gypsum, a versatile and easily extracted mineral, is also explored for its role in bio-composites, particularly in construction and building applications. The second chapter delves into the properties of both materials, their extraction methods, and their respective advantages and challenges in bio-composite formulation.

Ultimately, this first part of the work offers a comprehensive review of their properties, benefits, limitations, and applications, setting the stage for deeper investigations into the optimization and future potential of these materials. By examining the foundations of bio-composite materials, the first part provides a critical overview of their role in addressing the demand for sustainable, eco-friendly materials in various industrial sectors.

The first part can be summarized by the hierarchy, as shown in [Figure 02](#).



**Figure 02. Graphical Abstract of the First Part of the Thesis.**

# Chapter I

## Bio-composite Materials

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## **Introduction**

The increasing demand for sustainable and eco-friendly materials has led to the emergence of composite materials, which offer superior properties for various engineering applications. Composite materials, defined as materials made by combining two or more distinct materials, are designed to achieve enhanced mechanical, physical, and chemical properties that are not present in the individual components alone. These materials have found widespread use across a range of industries, from construction and automotive to aerospace and biomedical applications.

Composite materials can be classified into several types, based on their matrix, reinforcement, and nano-components. Matrix-based composites, reinforcement-based composites, and the more recent innovations in nano-based composites represent a wide array of material choices for different performance needs. Among these, bio-composite materials, which incorporate natural fibers into the matrix, are gaining significant attention due to their environmentally friendly attributes and the potential for sustainable production methods.

This first chapter delves into the concept of composite materials, focusing on bio-composites, which are particularly relevant in the context of sustainable materials science. It begins with a definition of composite materials and explores the different types, emphasizing the importance of natural fibers in the composition of bio-composites. Further, the first chapter investigates the properties of natural fibers, including their physico-mechanical and chemical characteristics, and examines the various methods of extracting these fibers for use in bio-composites.

The manufacturing processes for bio-composites are also discussed, from traditional methods such as hand lay-up and extrusion to advanced techniques like compression molding and filament winding. These methods play a crucial role in the fabrication of bio-composites, enabling their application in a wide range of industries. Furthermore, the advantages and challenges of bio-composite materials are critically assessed, offering insights into their future potential.

Ultimately, this first chapter provides a comprehensive overview of composite materials, particularly bio-composites, setting the foundation for a deeper exploration of their applications, benefits, and limitations in subsequent chapters.



## I.1. Definition of Composite Materials

Two or more different materials (Matrix and Reinforcement) are mixed to form composite materials, as shown in [Figure I.01](#). Producing a new material with qualities unlike those of the separate parts. Composite materials aim to increase performance using the constituent materials' advantages, such as strength, weight, durability, and resistance to heat or corrosion [28]. Usually, a composite material is made up of [29, 30]:

- 1. Matrix:** The foundation material, usually a polymer, metal, or ceramic, binds the components together and shapes the final result;
- 2. Reinforcement:** The foundation material, usually a whisker, particle, fiber, or structural material, inserted in the matrix to improve mechanical qualities like strength or stiffness, is called reinforcement.



**Figure I.01. Constitution of Composite Material [31].**

## I.2. Types of Composite Material

Composite materials can be categorized depending on their matrix material and the reinforcing material used. The composite materials, as shown in [Figure I.02](#), they divided into four types: Nano-based composites, matrix-based composites, reinforcement-based composites, and bio-composites-based composites.

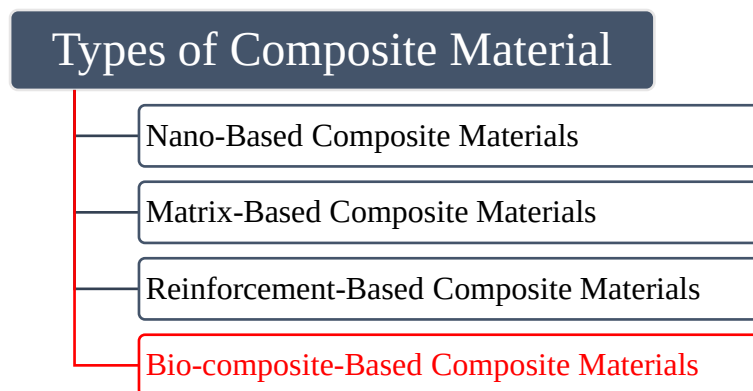


**Figure I.02. Types of Composite Material [30].**

The following is a thorough discussion of the key types of composite materials:

### I.2.1. Nano-Based Composite Materials

Nano-based composite materials are a leading material science; these composites have possible uses in many sectors, including electronics, aircraft, healthcare, energy storage, and environmental engineering [32].

The nano-based composite materials are a family of composite materials that combine nanoparticles into a polymer, metal, or ceramic matrix, as shown in [Figure I.03](#) [33]. They hence improve mechanical, thermal, electrical, and chemical qualities over the base material by itself [34].

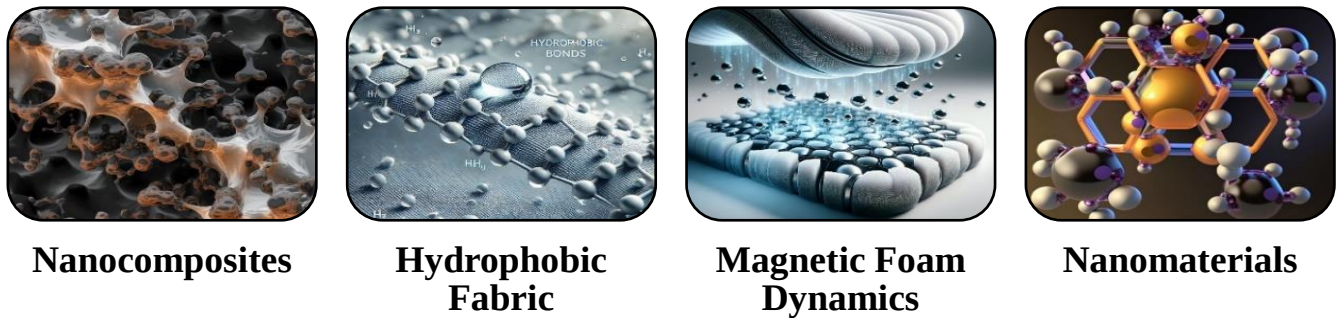


**Figure I.03. The Nano-Based Composite Materials.**

Ultimately, every type of nanocomposite has possible uses with several unique benefits. Choosing the right nanoparticles and matrix materials helps researchers customize the composites' characteristics to fit specific performance criteria in different industrial and technical domains.

### I.2.2. Matrix-Based Composite Materials

Leading materials research, these composites might be used in many sectors, including gas turbines, rocket engines, automotive, aircraft, and sports equipment [28].

The matrix-based composite materials consist of two separate phases: a matrix and a reinforcement, as shown in [Figure I.04](#). While the reinforcement improves the material's mechanical, thermal, electrical, or other qualities, the matrix is the continuous phase that holds the reinforcement in place and shapes the material. By binding the reinforcement together, the matrix guarantees that the composite material acts as a single, solid entity [28].



**Ceramic  
Matrix**



**Polymeric  
Matrix**



**Ceramic  
Matrix**



**Polymeric  
Matrix**

**Figure I.04. The Matrix-Based Composite Materials.**

Ultimately, selecting suitable matrix materials and reinforcements shows how composite materials may be customized for specific performance requirements. The engineers and material scientists use this categorization to choose the most appropriate composite for particular uses.

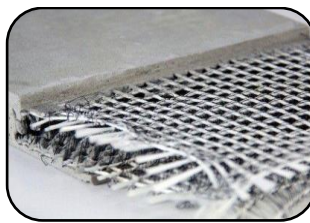
### **I.2.3. Reinforcement-Based Composite Materials**

Leading materials research, these sophisticated composites might be used in numerous sectors, including gas turbines, rocket engines, automotive, aviation, aerospace, and high-temperature settings [35].

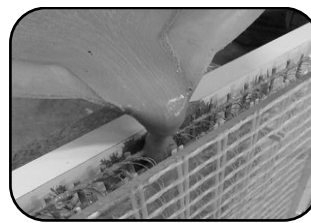
The reinforcement-based composite materials consist of a matrix phase with a reinforcement phase inserted therein, as shown in [Figure I.05](#) [35]. Reinforcement mainly causes the improved mechanical, thermal, electrical, or other performance characteristics of the material [36]. The reinforcement in these composites is usually made of high-strength, stiffness, or other desirable qualities, and its function is to improve the general performance of the composite material [30].



**Synthetic Macro  
Fibers-Concrete**



**Concrete Textile**



**Beton Campus**



**Lisocore-Light  
Construction**

**Figure I.05. The Reinforcement-Based Composite Materials.**

Ultimately, this categorization of reinforcement-based composite materials helps direct material choice based on the needs of specific uses by balancing considerations like strength, weight, and durability.

### I.2.4. Bio-composite-Based Composite Materials

Leading materials research shows that these sophisticated composites may be used in many industries, including packaging, automotive, agriculture, and construction [37]. The bio-composites are composite materials made of renewable natural fibers and bio-based matrices. They are manufactured in a variety of forms, as shown in [Figure I.06](#) [38]. Designed to reduce environmental impact and improve sustainability relative to conventional composite materials, depending on synthetic fibers and petroleum-derived matrix, they constitute a type of "Green Materials" [39]. Offering lightweight, strong, and environmentally responsible alternatives across many industries, including automotive, construction, packaging, and textiles, bio-composites combine the mechanical properties of fibers with the durability of a matrix [40].

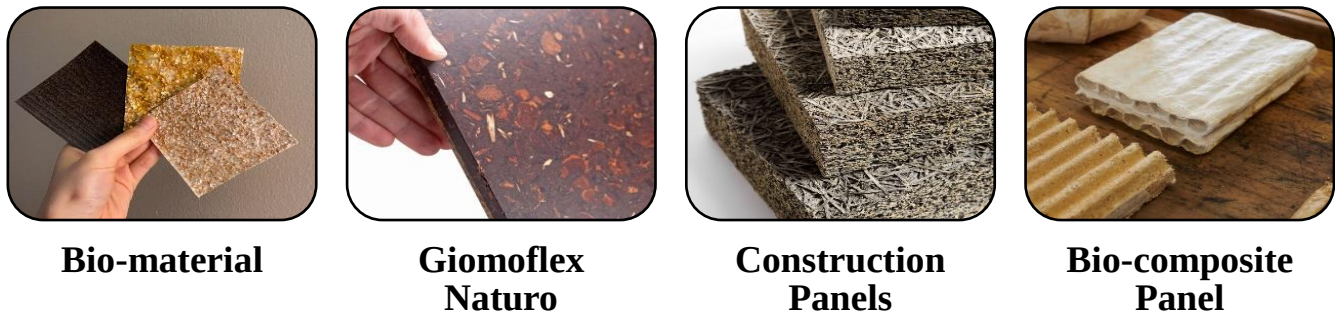


**Figure I.06. The Bio-composite-Based Composite Materials.**

Ultimately, this categorization of bio-composite-based composite materials facilitates material selection according to specific application requirements by balancing factors such as strength, weight, durability, and long-term environmental sustainability.

To conclude, engineers and scientists are working to develop more sustainable materials with particular characteristics for many applications across several sectors, including automobiles, aerospace, marine, furniture, agriculture, and packaging. They have to classify them into four types to help them identify them depending on their intended characteristics.

### I.3. Types of Bio-composite-Based Composite Materials

An important field of study is bio-composites, materials made of bio-based and occasionally synthetic elements, as they have possible sustainability in many sectors [41]. [Figure I.07](#). Separates bio-composites into three primary types.

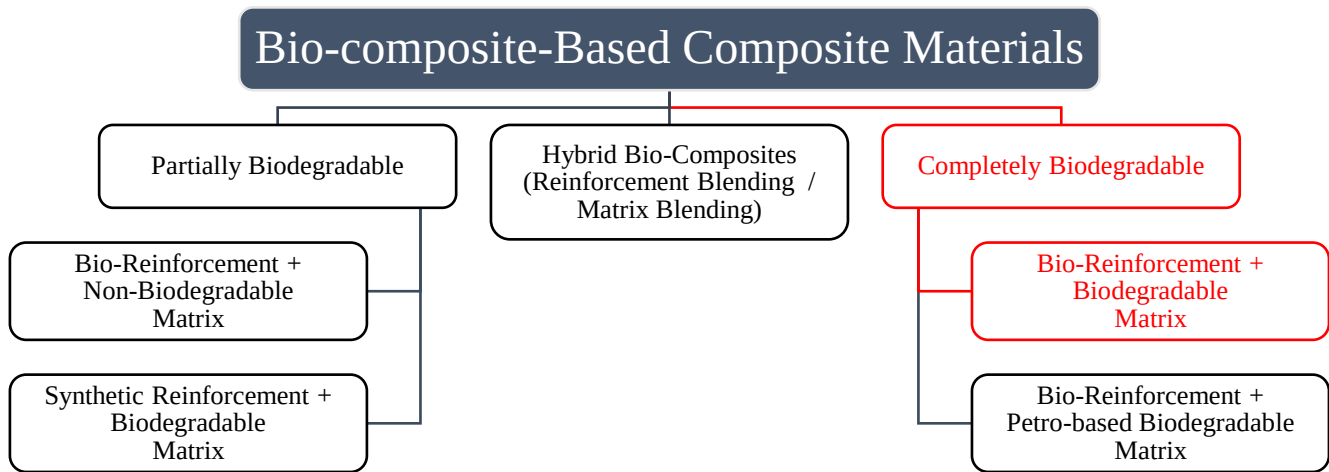


**Figure I.07. Types of Bio-composite-Based Composite Materials [42].**

The primary types of bio-composite-based composite materials are comprehensively addressed in these aspects:

- 1. Partially Biodegradable Bio-Composites:** The bio-composites of this type are not completely biodegradable [43]. Their development uses components including:
  - a. Bio-Reinforcement with Non-Biodegradable Matrix:** This subcategory includes bio-composites where the reinforcing is bio-based (Like Natural Fibers), but the matrix is non-biodegradable (Like Synthetic Polymers) [44];
  - b. Synthetic Reinforcement with Biodegradable Matrix:** The matrix is biodegradable (e.g., Bio-Based Polymers), and synthetic materials are used as reinforcement [37].
- 2. Hybrid Bio-Composites:** The hybrid bio-composites are combinations in which the matrix and reinforcing components are combined [45]. After that, there are two types of hybrid bio-composites [45]:
  - a. Reinforcement Blending:** Where a composite combines synthetic reinforcements with bio-based ones;
  - b. Matrix Blending:** Where synthetic and bio-based matrices are used.
- 3. Completely Biodegradable Bio-Composites:** This category emphasizes completely biodegradable substances. These composites are the most sustainable, leaving the least environmental effect after usage by breaking down into natural components [46]. The types of combinations are [46]:

- a. **Bio-Reinforcement with Biodegradable Matrix:** These composites are very sustainable, as both the matrix and the reinforcement are bio-based and biodegradable;
- b. **Bio-Reinforcement with Petro-based Biodegradable Matrix:** This consists of a biodegradable matrix from petrochemical origins combined with bio-based reinforcement. Although the matrix is biodegradable, petrochemical sources could cause environmental issues.

Ultimately, this categorization of bio-composite-based composite materials facilitates material selection according to specific application requirements by balancing factors such as strength, weight, durability, and long-term environmental sustainability.

To conclude, while bio-composite-based composite materials are the perfect choice for lowering waste and reliance on non-renewable resources, partially biodegradable composites might affect performance and sustainability.

In conclusion, engineers and scientists are working to develop more sustainable materials with particular characteristics for many applications across several sectors, including automobiles, aerospace, marine, furniture, agriculture, and packaging. They have to classify them into four types to help them identify them depending on their intended characteristics.

## I.4. Natural Fibers

Valued for their sustainability, natural fibers are commonly employed in many applications because of their strength, flexibility, and eco-friendly quality [47]. The following are the key discussions on natural fibers:

### I.4.1. Definition of Natural Fibers

The natural fibers have animal, plant-vegetable, and mineral [48], as shown in [Figure I.08](#). The being biodegradable and regenerative, they provide an environmentally viable alternative to synthetic fibers [47]. The natural fibers are often used in textiles, construction, automotive, and many other industries [49].



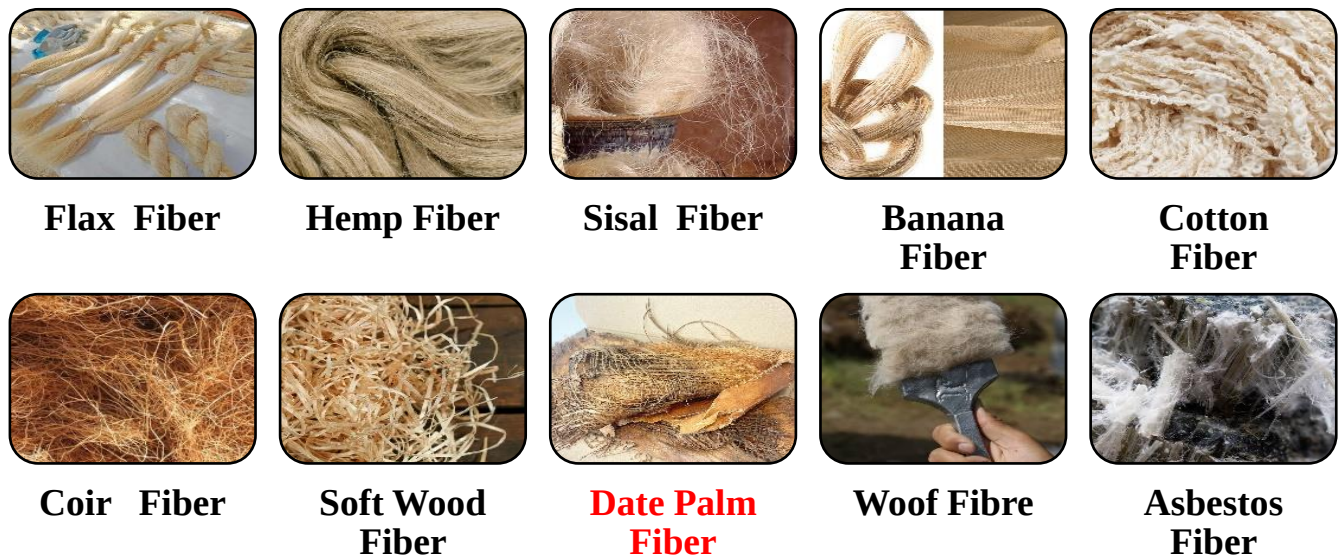


**Figure I.08. Natural Fibers-Reinforced in Composite Materials.**

### I.4.2. Types of Natural Fibers

**Figure I.09.** Shows a methodical categorization of natural fibers depending on their source. It classifies natural fibers into three primary categories [48]: animal, plant-vegetable, and mineral. Relevant to materials science, textile engineering, and natural fiber research, this classification helps to understand fiber qualities, uses, and sources.

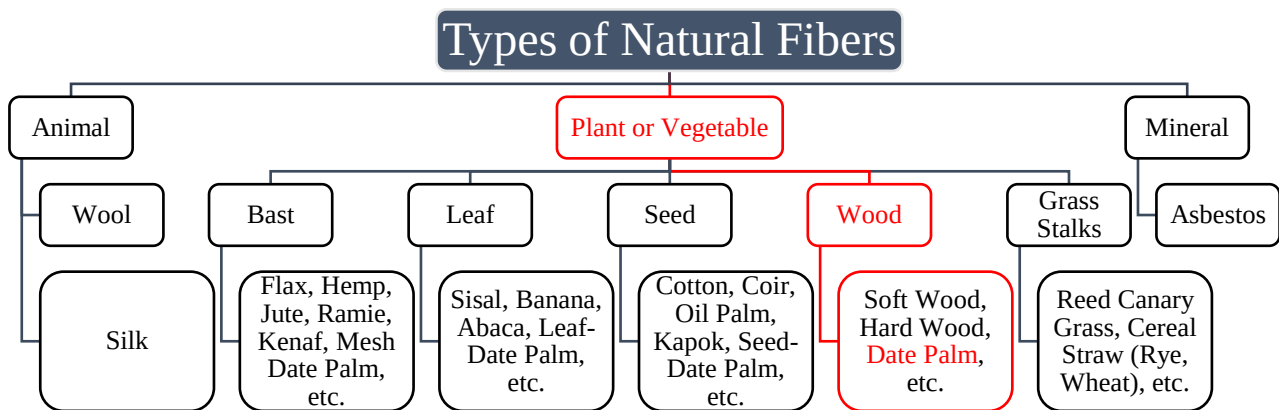


**Figure I.09. Types of Natural Fibers [50].**

The key types of natural fibers are listed below:

- 1. Animal Fibers:** These come from animals and consist of silk and wool [48]. Protein-based fibers are well-known for their flexibility, warmth, and moisture-wicking qualities [51];

- 2. Plant or Vegetable Fibers:** These are fibers made from cellulose sourced from various plant parts [52]. This group is divided into:
- a. Bast Fibers:** Sourced from the inner bark of several plants [53]. Among them are meshed date palms, kenaf, ramie, jute, hemp, and flax. Well known for their strength and longevity [54];
  - b. Leaf Fibers:** From the leaves of plants [55]. Sisal, banana, abaka, and leaf-date palm are among the examples. Often used in mats and ropes, leaf fibers are somewhat rough [55];
  - c. Seed Fibers:** Taken from the seed hairs of plants [56]. Among them are seed-date palm, kapok, oil palm, coir, and cotton. Because of its softness and breathability, cotton is the most often used seed fiber worldwide [57];
  - d. Wood Fibers:** Taken from the wood of certain trees [58]. These consist of trunk-Date Palm Fibers (DPFs), hardwood, and softwood. Though rare in textile uses, wood fibers are utilized in the paper and composite sectors;
  - e. Grass Stalk Fibers:** Derived from grass-like plants like reed canary grass, and cereal straw (Rye, Wheat) [59]. They used in handicrafts and composites, certain fibers have specialized uses.
- 3. Mineral Fibers:** Asbestos is a typical mineral fiber [60]. Historically, asbestos was employed for its heat resistance; currently, it is prohibited mostly because of health concerns.

Ultimately, this hierarchical classification of natural fibers shows the variety of fibrous elements present in natural sources. It is a helpful guide for academics, textile engineers, and material scientists trying to maximize fiber selection for specific industrial uses, environmental objectives, and performance standards.

### I.4.3. Properties of Natural Fibers

The properties of natural fibers determine their suitability for many applications, such as textiles, composite materials, and environmentally sustainable products [61]. The classification of properties of natural fibers, as shown in [Figure I.10](#), understanding these features optimizes fiber selection to get the desired performance for each application.



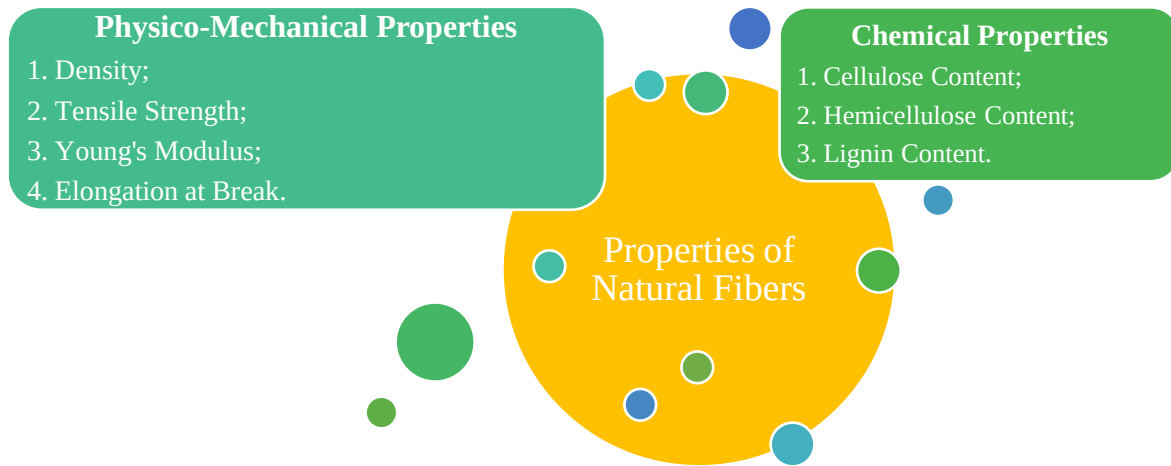


**Figure I.10. Classification of Properties of Natural Fibers.**

Natural fiber properties are defined as follows:

#### I.4.3.1 Physico-Mechanical Properties

The choice of a certain fiber must consider the requisite physico-mechanical capabilities and aspects like sustainability, affordability, and the environmental ramifications of manufacturing and disposal. The spectrum of tensile strengths, Young's moduli, densities, and elongation at break values demonstrates that natural fibers may be customized to meet diverse technical requirements based on their intended performance attributes. **Table I.01.** Shows a varied collection of natural fibers, each possessing unique physico-mechanical qualities that render them appropriate for various applications in construction, automotive, textiles, and composites.

**Table I.01. Physico-Mechanical Properties of Many Prevalent Natural Fibers [62-66].**

| Natural Fiber    | Density [g/cm <sup>3</sup> ] | Tensile Strength[MPa] | Young's Modulus [GPa] | Elongation at Break [%] |
|------------------|------------------------------|-----------------------|-----------------------|-------------------------|
| Abaca            | 1.50                         | 980                   | -                     | -                       |
| Bagasse          | 1.20                         | 20-290                | 19.7-27.1             | 1.1                     |
| Banana           | 1.35                         | 355                   | 33.8                  | 53                      |
| Coir             | 1.25                         | 220                   | 6                     | 15-25                   |
| Cotton           | 1.51                         | 400                   | 12                    | 3-10                    |
| <b>Date Palm</b> | <b>1.17-1.45</b>             | <b>28.52</b>          | <b>5-27</b>           | <b>20-50</b>            |
| Flax             | 1.40                         | 88-1500               | 60-80                 | 1.2-1.6                 |
| Hemp             | 1.48                         | 580-900               | 70                    | 1.6                     |
| Henequen         | 1.40                         | 430-580               | -                     | 3-4.7                   |
| Jute             | 1.46                         | 400-800               | 10-30                 | 1.8                     |
| Kenaf (Bast)     | 1.20                         | 295                   | -                     | 2.7-6.9                 |
| Kenaf (Core)     | 0.21                         | -                     | -                     | -                       |
| OPEFB            | 0.70-1.55                    | 248                   | 3.2                   | 2.5                     |
| Pineapple        | 1.50                         | 170-1672              | 82                    | 1-3                     |
| Ramie            | 1.50                         | 500                   | 44                    | 2                       |
| Sisal            | 1.33                         | 600-700               | 38                    | 2-3                     |

**Table I.01.** Shows the comparison of natural fiber density, tensile strength, Young's modulus, and elongation at break, which can be properties as follows:

- 1. Density:** The Fibers like abaca ( $1.5 \text{ g/cm}^3$ ) and pineapple ( $1.5 \text{ g/cm}^3$ ) possess a greater density, suggesting they may be heavier, perhaps influencing their applications where weight is crucial [67]. The Kenaf (Core) fiber, possessing a density as low as ( $0.21 \text{ g/cm}^3$ ), is markedly less dense, rendering it advantageous for lightweight applications such as insulation or cushioning [68].
- 2. Tensile Strength:** The Flax fiber has a strong tensile strength (88-1500 MPa), making it suitable for composite reinforcement [69]. The Bananas (355 MPa) and hemp (500-900 MPa) fibers are applied as durable fibers in the construction and automotive sectors [70]. However, coir (220 MPa) and bagasse (20-290 MPa) have lower tensile strengths and are less appropriate for high-stress applications, although they may help with mat or packing.
- 3. Young's Modulus:** The Linen's Young's modulus (60-80 GPa) makes it stiffer than other fibers, making it ideal for composite reinforcement where rigidity is crucial. Hemp, with a Young's modulus of 70 GPa, is both stiff and suitable for reinforcement [71]. Due to their lower values, coir (6 GPa) and Oil Palm Empty Fruit Bunch (OPEFB) (3.2 GPa) are more ideal for flexible or non-structural applications [72].
- 4. Elongation at Break:** The Banana fiber's (53%) elongation at break indicates its excellent elasticity and capacity to endure massive deformations. Its flexibility may make it appealing in textiles and other applications. The Date Palm Fiber's (DPFs) elasticity ranges from 20-50%, which is still higher than that of most other fibers. The Coconut fiber (15-25%) and cotton (3-10%) have moderate elongation and reduced extensibility.

Ultimately, these qualities indicate distinct elements of the natural fiber's performance, making them crucial for engineering and industrial selection.

#### I.4.3.2. Chemical Properties

**Figure I.11.** Shows the interrelation of cellulose, hemicellulose, and lignin in maintaining the structural integrity of plant cell walls. Each component serves a unique yet synergistic function: cellulose imparts stiffness, hemicellulose offers flexibility and support, and lignin augments strength and resilience against environmental pressures [73]. These three elements are crucial to the mechanical characteristics of plants, including their capacity to develop vertically and endure diverse environmental adversities.

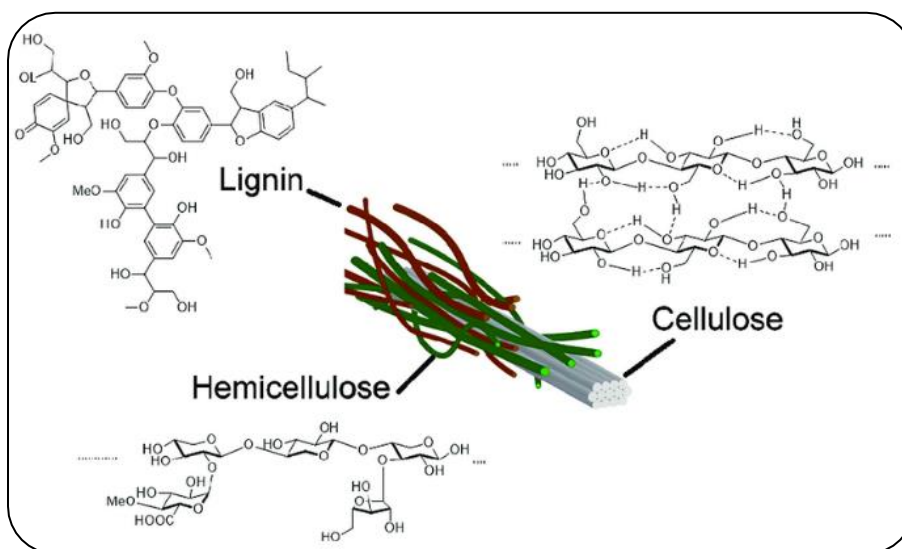


**Figure I.11. Components of Natural Fibers: Cellulose, Hemicellulose, and Lignin [74].**

The proportions of cellulose, hemicellulose, and lignin in several standard natural fibers constitute their chemical properties. [Table I.02](#). Shows data on the variations in the biochemical properties of natural fibers.

**Table I.02. Chemical Properties of Many Prevalent Natural Fibers [7, 62].**

| Natural Fiber    | Cellulose wt [%] | Hemicellulose wt [%] | Ligning wt [%] |
|------------------|------------------|----------------------|----------------|
| Abaca            | 56-63            | 20-25                | 7-9            |
| Bagasse          | 55.2             | 16.8                 | 25.3           |
| Bamboo           | 26-43            | 30                   | 21-31          |
| Coir             | 32-43            | 0.15-0.25            | 40-45          |
| Curaua           | 73.6             | 9.9                  | 7.5            |
| <b>Date palm</b> | <b>44</b>        | <b>26</b>            | <b>11.5</b>    |
| Flax             | 71               | 18.6-20.6            | 2.2            |
| Hemp             | 68               | 15                   | 10             |
| Jute             | 61-71            | 14-20                | 12-13          |
| Kenaf            | 72               | 20.3                 | 9              |
| Oil Palm         | 65               | -                    | 29             |
| Pineapple        | 81               | -                    | 12.7           |
| Ramie            | 68.6-76.2        | 13-16                | 0.6-0.7        |
| Rice Husk        | 35-45            | 19-25                | 20             |
| Rice Straw       | 41-57            | 33                   | 8-19           |
| Sisal            | 65               | 12                   | 9.9            |
| Wheat Straw      | 38-45            | 15-31                | 12-20          |

Where; wt: Weight in [%].

[Table I.02](#). Presents the disparities in the biochemical content of natural fibers and their prospective applications contingent upon this composition. All components are essential to the fiber's mechanical qualities and industrial applications, such that:

- 1. Cellulose Content:** Cellulose is a fundamental structural element of plant fibers, enhancing strength and stiffness [75]. The cellulose concentration in the specified fibers varies significantly, with pineapple exhibiting the greatest cellulose level at 81%. Additional fibers with elevated cellulose content are Curaua (73.6%) and Ramie (68.6-76.2%). These cellulose-rich fibers are expected to have superior tensile strength and endurance, rendering them appropriate for textile and composite material applications [76]. In contrast, fibers such as Bamboo (26-43%), Coir (32-43%), and Rice Husk (35-45%) exhibit reduced cellulose content, suggesting they may possess lower rigidity and enhanced flexibility, thereby rendering them more appropriate for applications where pliability is paramount, such as in the manufacturing of nonwoven materials or bio-composites [77].
- 2. Hemicellulose Content:** Unlike cellulose, hemicellulose is an amorphous polymer that imparts flexibility [78]. It possesses reduced tensile strength but facilitates the adhesion of cellulose fibers [79]. The Date Palm (26%) and Rice Straw (33%) have considerable hemicellulose content, indicating a compromise between flexibility and structural integrity [80]. Coir fiber is an anomaly, with an unusually low hemicellulose concentration (0.15-0.25%), which may result in reduced flexibility but perhaps increased rigidity and strength due to its elevated lignin level (40-45%) [81].
- 3. Lignin Content:** Lignin is a binding agent for cellulose fibers, providing structural stiffness and resistance to degradation [82]. Fibers such as Coir (40-45%) and Bagasse (25.3%) have notably elevated lignin concentrations. This may enhance their durability and water resistance; nevertheless, lignin's toughness may complicate the processing into fine fibers or textiles [83]. Flax and Ramie, with less lignin concentration (About 2.2% and 0.6-0.7%, Respectively), may facilitate the processing into high-quality textile products due to the diminished effort needed to eliminate lignin and reveal cellulose [84].

To conclude, the diverse chemical compositions of various fibers highlight the necessity of fiber types according to the intended application. Comprehending the ratios of cellulose, hemicellulose, and lignin in each fiber is crucial for enhancing their application in textiles, bio-composites, and biodegradable goods. Future research and development may concentrate on refining processing procedures to augment these fibers' mechanical qualities and sustainability.

#### I.4.4. Extraction Methods of Natural Fibers

**Figure I.12.** Shows the many natural fiber extraction methods, grouped into four primary types: mechanical, chemical, biological, and physical retting procedures [85]. The fiber extraction is fundamental in the manufacture of natural fibers for textile, composite, and industrial uses, affecting fiber quality, yield, and environmental effects [86].

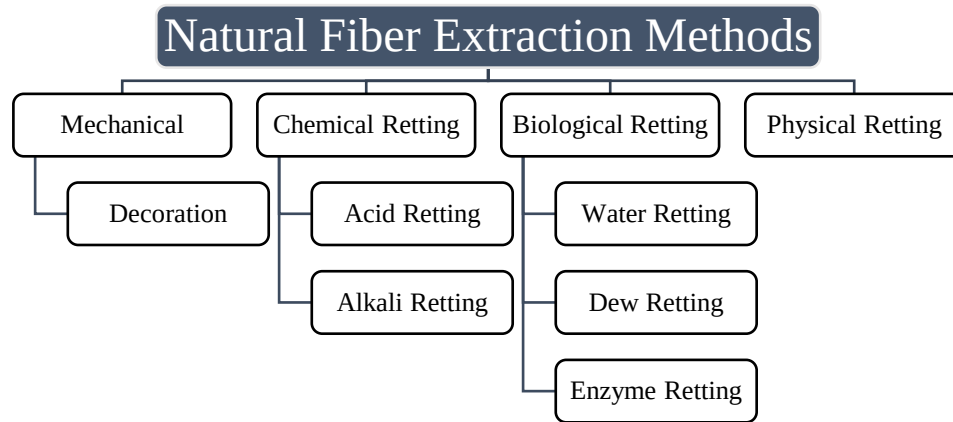


**Figure I.12. Extraction Methods of Natural Fibers [85].**

The key types of extraction methods for natural fibers are listed below:

- 1. Mechanical Methods:** This is a physical procedure to extract plant material's fibers [87]. This type is split into:
  - a. Decortication:** A standard mechanical technique called decortication removes fibers from the stalk or other areas by crushing, hammering, or scraping [87].
- 2. Chemical Retting Methods:** These methods break down the pectin and other compounds, attaching the fibers to the plant matrix using chemicals [49]. This type is divided into:
  - a. Acid Retting:** Dissolves non-cellulosic substances using acidic solutions (e.g., Sulfuric Acid) [88];
  - b. Alkali Retting:** Degrades hemicellulose and lignin using alkaline chemicals, including sodium hydroxide [89].
- 3. Biological Retting Methods:** Often seen as more ecologically benign, they depend on microbial activity to break down pectin and other binding agents [90]. This type is divided into:
  - a. Water Retting:** Plant materials are immersed in water, where anaerobic bacteria break down the binding agents [91];

- b. **Dew Retting:** Spread throughout the field and exposed to moisture and dew, plants undergo retting by aerobic bacteria and fungi [92];
  - c. **Enzyme Retting:** Isolated enzymes (e.g., Pectinases) are used to target binding materials specifically for a regulated and efficient procedure [93].
- 4. Physical Retting:** This type covers additional physical or enzymatic activities like enzyme retting, which is mentioned here [92]. The enzyme retting, on the other hand, is occasionally classified under biological procedures because of its biochemical character [93]. The physical methods could include operations like steam treatments or ultrasonic treatments to support fiber separation without severe chemicals [94].

Ultimately, the choice of fiber extraction method directly affects fiber quality, strength, fineness, and environmental sustainability. Industries using natural fibers in textiles, bio-composites, and other sustainable products must first grasp these methods.

#### I.4.4.1. Fundamental Aspects of Extracting Natural Fibers for Using in Bio-Composite Materials

Due to our pressing requirement for functional bio-composite materials, certain aspects must be observed when extracting natural plant fibers. **Figure I.13.** Shows the fundamental aspects of extracting natural fibers for bio-composite materials.

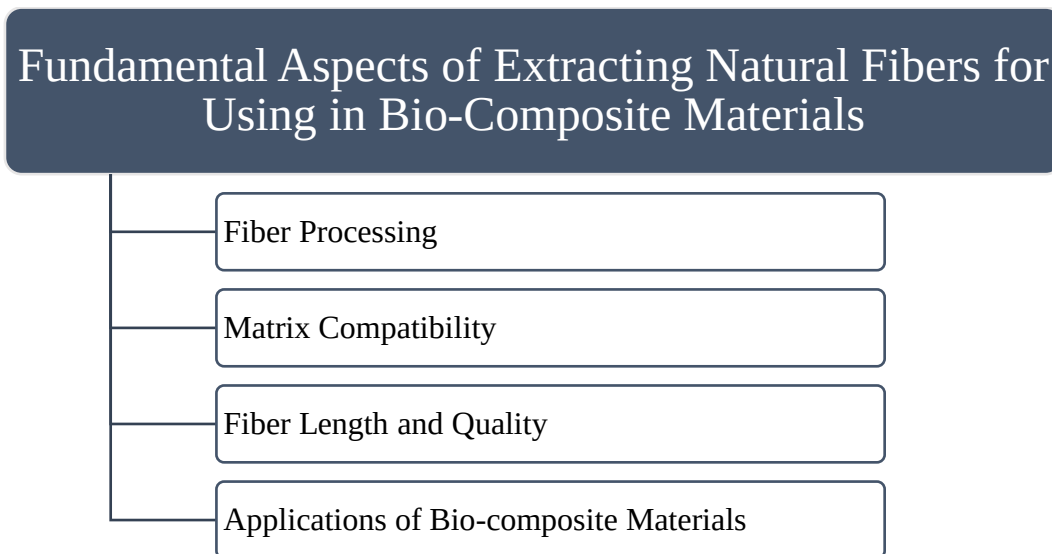


**Figure I.13. Fundamental Aspects of Extracting Natural Fibers for Using in Bio-Composite Materials.**

The fundamental aspects of extracting natural fibers for bio-composite materials are as below:

- 1. Fiber Processing:** Post-extraction, fibers often undergo processing, which includes cleaning, drying, and chemical treatments, such as alkali treatment, to enhance their compatibility with the matrix (Matrix or Polymers) [95];
- 2. Matrix Compatibility:** The bio-composite materials are produced by combining natural fibers with bio-based matrices, such as poly-lactic acid and starch matrix, or thermo-plastic polymers, like polypropylene and polyethylene. Bonding agents and other fiber surface treatments can enhance the adhesion between fibers and matrix [96];
- 3. Fiber Length and Quality:** The length and quality of the fibers fundamentally influence the durability and longevity of the final composite product. Fibers are regularly cut or treated to the requisite length according to the application [97];
- 4. Applications of Bio-composite Materials:** Extracted fibers offer a more sustainable alternative to synthetic materials and facilitate the manufacturing of bio-composite materials across several sectors, including automotive, construction, packaging, and consumer goods [38].

Ultimately, these aspects are essential for extracting fibers from natural plants to achieve the desired qualitative qualities of the bio-composite materials.

## I.5. Manufacturing Methods of Bio-composite Materials

The bio-composite materials manufacturing methods require the use of bio-materials that can act as a matrix, reinforcement, or both. These methods differ in their ease of application and intended uses.

**Figure I.14.** Shows the most common manufacturing methods for bio-composite materials.

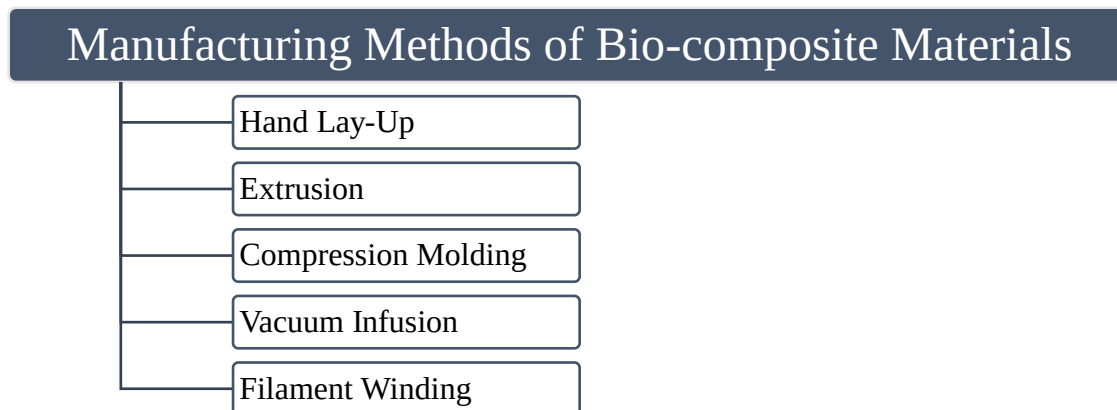


**Figure I.14. Manufacturing Methods of Bio-composite Materials.**

These aspects identify several methods for manufacturing bio-composite materials and highlight their transformation into materials usable in diverse applications:

### I.5.1. Hand Lay-Up

The hand lay-up Method for producing eco-friendly bio-composite materials for many applications is a vital process. **Figure I.15.** Shown is the manufacturing method called bio-composite material hand lay-up produces bio-composite materials composites manufactured by mixing natural fibers (Such as Flax, Hemp, Jute, or Bamboo) with bio-matrix (Like Polylactic Acid PLA or Other Biodegradable Matrix) [98]. The hand lay-up method puts fiber reinforcement layers into a mold and then applies matrix to saturate the fibers [98]. The procedure is straightforward for applications in sectors like automotive, construction, and consumer products and is often employed in low-to-medium manufacturing numbers [99].



**Fabric-Formed  
Tea Table**



**Rolling Layers**



**Fibreglass  
Products**



**Marine Epoxy  
Paints**

**Figure I.15. Hand lay-up of Bio-Composite Materials.**

Ultimately, hand lay-up for bio-composite materials finishes using natural fibers, including abaca, hemp, flax, and sisal, to create lightweight, sustainable, strong materials relevant across many industries, including automotive, building, packaging, and textiles.

### I.5.2. Extrusion

The extrusion method for manufacturing eco-friendly bio-composite materials for various applications is a critical procedure. The manufacturing bio-composite material extrusion is the method whereby a bio-composite made of natural fibers such as wood, hemp, or flax combined with a matrix is forced through a mold or die to create a continuous shape or profile, as shown in **Figure I.16** [100]. The manufacturing components for the automotive, construction, and packaging sectors as well as pipes, sheets, films, and other goods, this approach is frequently applications [101].

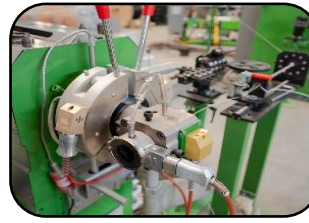




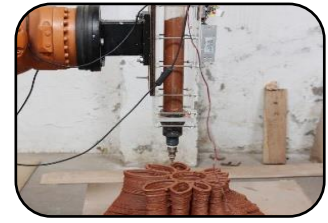
**Extrusion-  
Plant Walls**



**Extrusion-  
Ceramic**



**Extrusion-  
Optical Fiber**



**Extrusion-  
Clay Pots**

**Figure I.16. Extrusion of Bio-Composite Materials.**

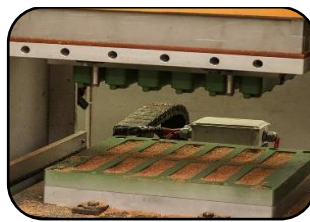
Ultimately, extrusion bio-composite materials utilize natural fibers like abaca, hemp, flax, and sisal to produce lightweight, sustainable, and robust materials applicable across several sectors, including automotive, construction, packaging, and textiles.

### I.5.3. Compression Molding

The compression molding method for manufacturing sustainable bio-composite materials for diverse applications is an essential process. The compression molding employs heat and pressure to shape bio-composite materials into designated shapes [102], as shown in [Figure I.17](#). Typically consisting of natural fibers (Such as Flax, Hemp, or Jute, etc.), bio-composite materials are placed into a heated mold during this process and combined with biodegradable matrices (Such as PLA, PP, Epoxy, etc.) [103]. The closed mold exerts pressure on the material, facilitating its flow to fill the mold cavity.



**Press  
Mouldings**



**Cork  
Molding**



**Cork  
Molding**



**Mushroom  
Packaging**

**Figure I.17. Compression Molding of Bio-Composite Materials.**

Ultimately, compression molding is a flexible and environmentally beneficial manufacturing method that applies natural fibers to create a wide spectrum of durable and sustainable goods. Industries such as automotive, construction, packaging, and consumer products depend on it extensively.

### I.5.4. Vacuum Infusion

The vacuum infusion method for producing sustainable bio-composite materials for various purposes is a critical procedure. **Figure I.18.** Shows that bio-composite material vacuum infusion is a manufacturing method for producing bio-composed materials. These materials integrate natural fibers (e.g., Sun, Ganja, or Jute) with the bio-based matrix. During the vacuum infusion method, natural fiber reinforcement is distributed in a form, and a vacuum is applied to extract the matrix in fiber layers, guarantee homogeneous saturation, and make a bio-composite material [104].



**Vacuum Manual Machine**



**Vacuum Forming a Mold**



**Vacuum Gauge and Valve**



**Vacuum Bagging**

**Figure I.18. Vacuum Infusion of Bio-Composite Materials.**

Ultimately, vacuum infusion is a versatile and organic-friendly manufacturing method that applies natural fibers to generate durable objects.

### I.5.5. Filament Winding

An important method for producing sustainable bio-composite materials for many applications is filament winding. The winding continuous fibers, usually, natural fibers like flax, hemp, or jute, impregnated with a bio-based matrix (Like Polylactic Acid PLA or Epoxy) around a spinning mandrel (Cylindrical Mold), bio-composite material filament winding is a manufacturing method application to produce bio-composite materials, as shown in **Figure I.19** [105]. The method is planned for producing cylindrical or pipe-shaped overall structures such as wide pipes, tanks, and pressure containers and is known for creating strong, light, and long-term components [106].



**Filament Winding-  
Storage Tank**



**Filament Winding-  
Carbon Fiber**



**Filament Winding-  
Machine**



**Filament Winding-  
Carbon Fiber**

**Figure I.19. Filament Winding of Bio-Composite Materials.**

Ultimately, filament winding is a versatile and organic-friendly manufacturing method that applies natural fibers to generate durable objects.

In conclusion, the methods of manufacturing natural fibers into bio-composites, whether by hand lay-up, extrusion, compression molding, vacuum infusion, or filament winding, are essential for producing bio-composites with distinctive properties, thus enabling them to establish themselves in various fields where they can be relied upon [107].

## **I.6. Applications of Use Bio-composite Materials**

Made from sustainable resources, bio-composite materials combine matrix material bioplastics or biodegradable matrix with natural fibers like hemp, flax, or jute [108]. These materials provide a sustainable substitute for conventional bio-composites composed of synthetic fibers and polymers and they have various applications, as shown in [Figure I.20](#).

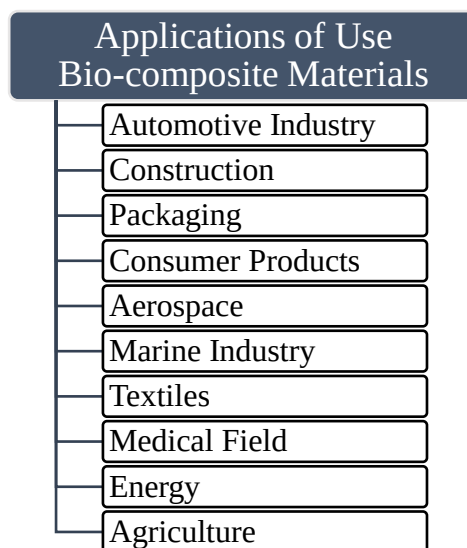


**Figure I.20. Applications of Use Bio-composite Materials.**

The Key applications for bio-composite materials include:

1. **Automotive Industry:** Bio-composites are applications in car exteriors and interiors for parts like door panels, dashboards, and seats. They cut carbon emissions, increase fuel economy, and help lighten vehicle weight, as shown in [Figure I.21](#) [109];
2. **Construction:** Flooring, paneling, and insulation require bio-composites. Their inherent qualities make them strong acoustic and thermal insulators. Furthermore, they provide a more sustainable substitute for traditional building materials, as shown in [Figure I.21](#) [110];
3. **Packaging:** Bio-composites are becoming popular in applications like packaging because of their biodegradability and recyclability. When these materials replace conventional plastic packaging, the environmental impact is decreased, as shown in [Figure I.21](#) [111];
4. **Consumer Products:** Bio-composites provide ecologically beneficial, lightweight qualities and durability and may be applications to create objects like furniture, sporting goods, and personal accessories, as shown in [Figure I.21](#) [112];
5. **Aerospace:** The aerospace sector is exploring bio-composites for manufacturing airplane parts, including structural and interior components. Bio-composites' lightweight character helps to support environmental sustainability and fuel economy, as shown in [Figure I.21](#) [113];
6. **Marine Industry:** Boat hulls, decks, and other parts call for bio-composites. Their low environmental effect and corrosion resistance enable them to lower the usage of materials derived from petroleum in maritime projects, as shown in [Figure I.21](#) [114];
7. **Textiles:** Natural fibers mixed with bio-based matrix provide strong, biodegradable, sustainable textiles in the textile business. These materials are applied to produce clothing, upholstery, and other fabric-based goods, as shown in [Figure I.21](#) [115];
8. **Medical Field:** Bio-composites find possible applications in medical equipment, particularly for biodegradable implants and prostheses. Due to their biocompatibility, these materials can promote patient recovery and help to lower the need for operations, as shown in [Figure I.21](#) [116];
9. **Energy:** Bio-composites are applied in the renewable energy industry, for example, in wind turbine blades, where strength and lightweight are vital. They might also be used in energy storage systems, as shown in [Figure I.21](#) [117];

**10. Agriculture:** Various agricultural applications for bio-composites include biodegradable plant pots, which, when broken down, improve soil quality and help to lower waste, as shown in **Figure I.21** [118].

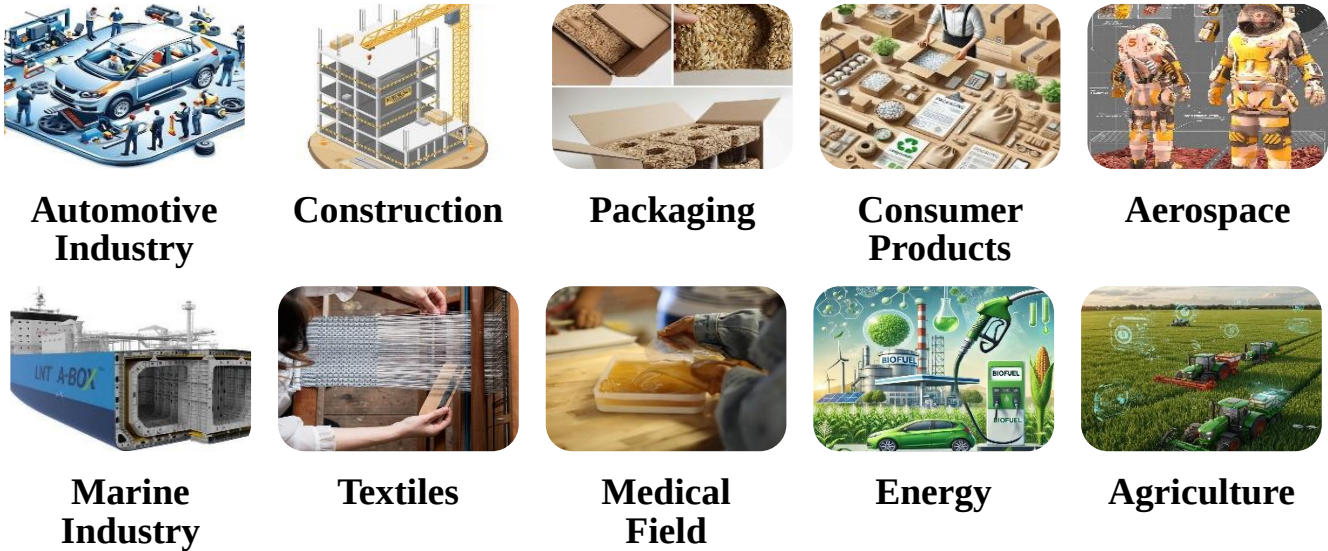


**Figure I.21. Applications of Using Bio-composite Materials.**

Ultimately, driven by sustainability objectives and the necessity of ecologically benign substitutes, these applications draw attention to the expanding interest and possibilities for bio-composites in several sectors.

## **I.7. Advantages and Disadvantages of Using Bio-composite Materials**

Although the manufacturing and general acceptance of bio-composite materials have significant environmental and sustainability advantages and disadvantages, as shown in **Figure I.22**, several key issues must be resolved.





**Figure I.22. Advantages and Disadvantages of Using Bio-Composite Materials.**

The overview of the advantages and disadvantages of bio-composite materials is summarized as follows:

### **I.7.1. Advantages of Using Bio-composite Materials**

There are several advantages through which bio-composites can be utilized on the ground, the most important of which are summarized in the following aspects:

- 1. Improving Material Performance:** Improved bonding agents and methods to increase the adhesion between natural fibers and bio-based matrix can greatly improve the mechanical qualities of bio-composites. In addition, natural fibers combined with synthetic or sophisticated materials like graphene or carbon fibers can produce hybrid bio-composites with enhanced performance, durability, and strength [119];
- 2. Advanced Processing Technologies:** Novel processing methods, such as nanotechnology and 3D printing, solve present industrial problems. These methods can increase the accuracy and efficiency of bio-composite fabrication and allow more complex and customized designs [119];
- 3. Sustainability and Recycling Innovations:** Crucially, developing bio-composite materials with higher biodegradability and recyclability will be vital. More effective recycling methods that allow the application of materials at the end of their life cycle and bio-composites that break down quickly without manufacturing toxic residues depend on research to accomplish these goals [120];

4. **Development of High-Performance Bio-based Matrix:** Improving bio-based matrix with stronger heat resistance, impact strength, and weathering resistance would let bio-composites operate better in demanding applications [119];
5. **Integration with Emerging Industries:** Promising possibilities for bio-composites in developing sectors like biotechnology (e.g., Medical Devices and Implants) and renewable energy (e.g., Wind Turbine Blades, and Energy Storage) are shown. These research disciplines will help find fresh applications for bio-composites and more effective manufacturing methods [121].
6. **Standardization and Quality Control:** Standardized testing and bio-composite certification methods help to ensure ongoing performance and quality. This will inspire trust in bio-composite materials and allow them to be accepted in industrial applications [37];
7. **Cost Reduction:** Bio-composites will become less expensive as research advances and more effective manufacturing procedures are developed, increasing their competitiveness with conventional synthetic materials. This will call for bettering supply networks for raw materials and increasing manufacturing capacity [122];
8. **Consumer Awareness and Market Acceptance:** Increasing awareness of the benefits of bio-composite should increase demand as consumers and companies respect stability. Promoting their environmental benefits and performance functions for market entry would be important [122].

Ultimately, through the aforementioned aspects, these advantages will provide a promising future for bio-composite materials in their exploitation, environmental conservation, and application in various fields.

### I.7.2. Disadvantages of Using Bio-composite Materials

There are many disadvantages facing the application and exploitation of bio-composites on the ground, the most important of which are summarized in the following aspects:

1. **Material Properties:** The mechanical qualities of bio-composites are sometimes less than those of synthetic composites. Some high-performance applications may require more than their strength, stiffness, and durability can provide. In addition, natural bio-composite fibers are

hygroscopic. Hence, they collect moisture and can cause dimensional instability, deterioration, and, with time, decreased mechanical qualities [123];

2. **Variability in Raw Materials:** Geographic location, growth circumstances, and harvesting methods may all greatly affect the qualities of natural fibers. Given this diversity, standardizing bio-composite materials for mass manufacturing is challenging [123];
3. **Processing Challenges:** Making bio-composites can be trickier than with conventional composites. Fiber-matrix adhesion, processing conditions, and the integration of natural fibers with bioplastics might challenge the assurance of homogeneous quality and performance [124];
4. **Cost:** The cost of manufacturing bio-composites can be greater than that of conventional composites depending on elements like raw material procurement, processing, and the evolution of specialized technology. In some markets, this reduces the competitiveness of bio-composites [125];
5. **Degradation and Stability:** Under some circumstances, such as UV radiation or excessive humidity, biodegradable materials are prone to aging and breakdown. This can restrict their usage in long-term applications, particularly in outdoor or demanding surroundings [123];
6. **Recycling and End-of-Life Disposal:** Although biodegradability helps make bio-composites ecologically favorable, recycling and end-of-life disposal problems remain. Research and development on recycling bio-composite materials, especially those using bioplastics, is underway [126];
7. **Limited Commercialization and Adoption:** The adoption of bio-composite materials in the aerospace, building, and automotive sectors is still restricted. This is usually the result of ignorance, worries regarding material performance, and the requirement to invest in new manufacturing technology [123].

Ultimately, the disadvantages confronting bio-composites are many yet surmountable. The bio-composites enable the realization of their objectives to save the environment and repurpose resources for practical applications in daily life.

To conclude, even if bio-composites provide sustainable, ecologically acceptable substitutes that might transform sectors, material performance, pricing, processing, and recycling still present major difficulties. Future success and general acceptance of bio-composite materials depend on creative research and development addressing these disadvantages.



## Conclusion

In conclusion, this first chapter has provided an in-depth exploration of composite materials, with a particular focus on bio-composites, which are at the forefront of sustainable material science. Composite materials, by definition, combine two or more distinct materials to create a new material with superior properties. The classification of composite materials into various types, including nano-based, matrix-based, and reinforcement-based composites, demonstrates the versatility and potential of these materials in addressing diverse engineering needs.

The emphasis on bio-composites has highlighted the growing interest in natural fibers as sustainable reinforcement agents. Natural fibers offer a unique combination of environmental benefits, mechanical performance, and biodegradability, making them an attractive alternative to traditional synthetic fibers. This first chapter has also outlined the essential properties of natural fibers, including their physico-mechanical and chemical characteristics, which play a vital role in determining their suitability for use in bio-composites.

Moreover, the various methods of fiber extraction and the different manufacturing techniques, such as hand lay-up, extrusion, and filament winding are critical in shaping the performance and application of bio-composites. These manufacturing processes, each with its own advantages and limitations, enable the fabrication of bio-composite materials with tailored properties suitable for a range of industries, from automotive to construction.

The discussion of the advantages and disadvantages of bio-composite materials has provided a balanced view of their potential. While bio-composites offer significant environmental and economic benefits, challenges related to cost, durability, and performance under certain conditions must still be addressed.

Ultimately, the development and widespread application of bio-composites will depend on continued research into enhancing their properties, improving manufacturing techniques, and overcoming the inherent challenges they present. This first chapter sets the stage for the subsequent exploration of bio-composite applications and further research into optimizing their use in modern engineering and material science.

# Chapter II

## Date Palm Fibers and Gypsum in Bio-Composite Materials

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## **Introduction**

The demand for sustainable materials has prompted extensive research into bio-composite materials, particularly those derived from natural fibers and locally available resources. One such promising combination is the use of Date Palm Fibers (DPFs) and gypsum, both of which offer distinct advantages for the development of bio-composites. This second chapter introduces the individual components, Date Palm Fibers (DPFs) and gypsum, highlighting their significance, properties, and applications within the realm of bio-composite materials.

Date Palm Fibers (DPFs), a by-product of the date palm tree, have been gaining attention due to their abundance, biodegradability, and favorable mechanical properties. These fibers are available in various types, each with unique characteristics that influence their suitability for use in bio-composites. This second chapter explores the properties of Date Palm Fibers (DPFs), focusing on their mechanical, chemical, and physical attributes that make them a valuable reinforcement material in composite formulations. Additionally, we discuss the extraction methods employed to obtain these fibers and examine their applications in multiple industries, including construction and automotive.

Gypsum, a naturally occurring mineral, is another key component in the formation of bio-composite materials. Known for its versatility and ease of extraction, gypsum is widely used in construction and building materials, often in the form of plaster and board. The second chapter provides an overview of gypsum, outlining its types, properties, extraction methods, and applications in both traditional and modern industries. The benefits of incorporating gypsum into bio-composites are examined, along with its potential limitations.

The combination of Date Palm Fibers (DPFs) and gypsum in bio-composites presents an innovative approach to sustainable material science. This second chapter critically evaluates the advantages and disadvantages of using Date Palm Fibers (DPFs) and gypsum as components in bio-composite materials. It highlights their respective strengths, such as natural availability, biodegradability, and cost-effectiveness, while also addressing the challenges associated with their use, such as durability and processing difficulties.

Furthermore, in this second chapter, a comprehensive review of the literature on bio-composite materials composed of Date Palm Fibers (DPFs) and gypsum is presented, summarizing previous studies and identifying research gaps. This sets the foundation for further investigations into optimizing the performance and applications of bio-composite materials based on these resources. The exploration of

these materials contributes to the broader field of sustainable construction and the development of eco-friendly materials for various industrial applications.

Ultimately, this second chapter serves as an introduction to the fundamental concepts and background necessary for understanding the role of Date Palm Fibers (DPFs) and gypsum in bio-composite materials, paving the way for deeper discussions in subsequent sections.

## II.1. Definition of Date Palm Fibers

**Figure II.01.** Shown are the extracted fibers naturally occurring in several sections of the date palm tree, mostly from its leaves, fronds, and stalks [6]. Date Palm Fibers (DPFs) are strong, long-lasting, biodegradable fibers, which are a green substitute for synthetic ones [6]. High tensile strength, lightweight nature, and moisture resistance define Date Palm Fibers (DPFs), which fit a broad spectrum of applications, including traditional applications like weaving and handicraft, and modern applications in bio-composites, textiles, and building materials [5]. Functional and environmentally friendly, the fibers can also be used to make ropes, mats, baskets, and brushes [127].

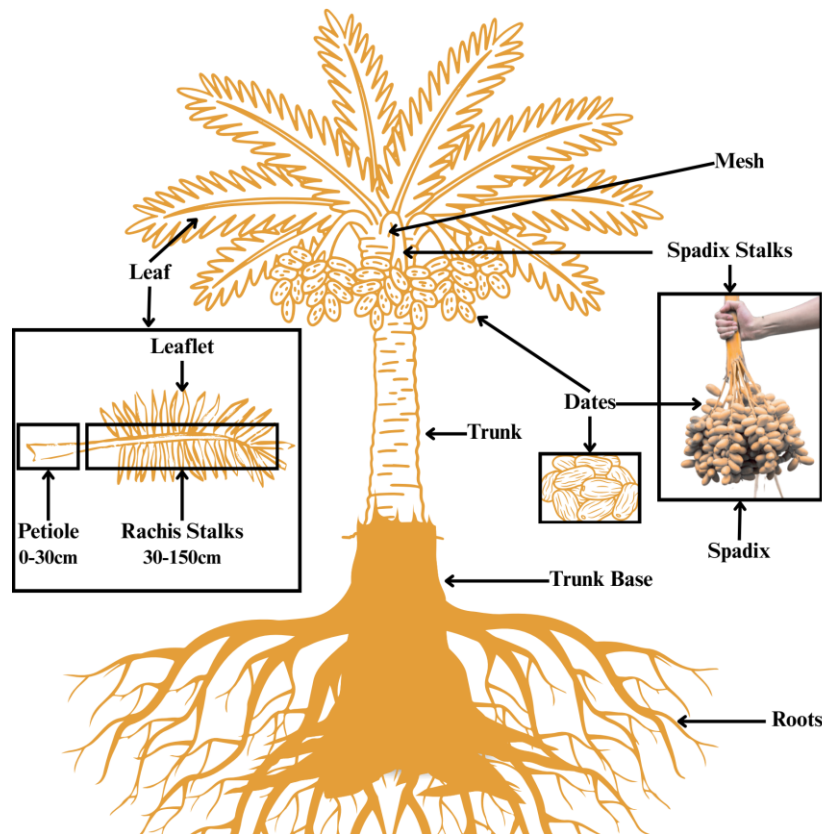


**Figure II.01. Sections of the Date Palm Tree.**

## II.2. Types of Date Palm Fibers

Derived from different sections of the date palm tree, Date Palm Fibers (DPFs) vary in quality and application based on their source. These fibers have been traditionally used in construction and manufacturing, as well as in modern applications such as bio-composites and textiles. The following list outlines the various types of Date Palm Fibers (DPFs):

- 1. Leaf Fibers (Fronds Fibers):** These fibers are taken from the fronds and leaves of the date palm tree [128], as shown in [Figure II.02](#). They are often used for their strength, durability, and moisture resistance [129]. Despite their coarse and quite stiff nature, they are perfect for ropes, carpets, and other woven goods, as they have outstanding tensile strength [130];
- 2. Spathe Fibers:** Extracted from the spathe, the protective leaf covering envelops the newborn fronds [131], as shown in [Figure II.02](#). These fibers have shorter lengths and finer quality than frond fibers. Though less often utilized, they have a smoother feel and are nonetheless robust and flexible [131];
- 3. Petiole Fibers:** Derived from the petioles, the leaf stalks link the leaves to the stalks [132], as shown in [Figure II.02](#). Although these fibers are somewhat coarse and strong, their flexibility lets them be employed for specific structural purposes [132]. They are usually stronger than mesh fibers but are less durable than those from the fronds [133];
- 4. Rachis Fibers:** The date palm tree leaves' Rachis (Centre Stalks) provides Date Palm Rachis Fibers (DPFRs), as shown in [Figure II.02](#). The Date Palm Rachis Fibers (DPFRs) is the primary axis supporting the separate leaflets of the palm leaves [134];
- 5. Mesh Fibers:** Date Palm Fiber Mesh (DPFM) are the fibrous substance on the stem and base of the fronds (Leaves) [63], as shown in [Figure II.02](#). Because of their natural strength, durability, and availability, these fibers have historically been employed in many civilizations for practical and sustainable applications [3];
- 6. Root Fibers:** Drawn from the date palm tree's roots, as shown in [Figure II.02](#). Among all the Date Palm Fibers (DPFs), they are both the best and the worst [135]. Although these fibers are often not as strong or durable as other types of Date Palm Fibers (DPFs), they offer considerable flexibility [135].

**Date Palm-Leaf****Date Palm-Spathe****Date Palm-Petiole****Date Palm-Rachis****Date Palm-Mesh****Date Palm-Root**

**Figure II.02. Locations of Palm Fiber Extraction used as Reinforcement for Bio-composite Materials.**

Ultimately, every sort of Date Palm Fiber (DPF) has special qualities that fit various applications [5]. Depending on the portion of the tree from which the fibers are taken, their strength, flexibility, texture, and durability change. These fibers are being investigated increasingly as demand for sustainable materials rises for usage in textiles, bio-composites, and other environmentally friendly goods, therefore providing value to a hitherto underused resource [127].

### **II.3. Properties of Using Date Palm Fibers**

Date Palm Fibers (DPFs) have unique properties that make them useful for many applications. They are natural and biodegradable and have various properties that suit conventional and contemporary usage, as shown in [Figure II.03](#).

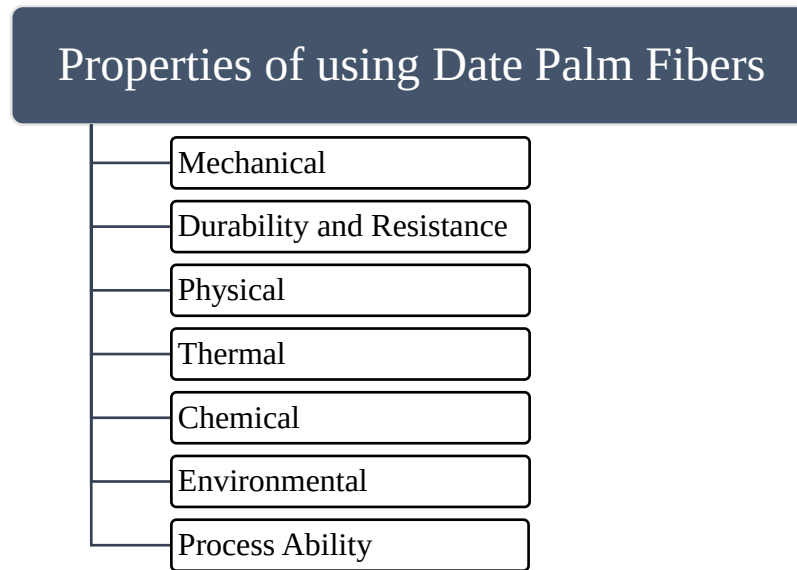


**Figure II.03. Properties of using Date Palm Fibers.**

The main properties of Date Palm Fibers (DPFs) are listed below:

**1. Mechanical Properties:** It will be as follows:

- a. **Tensile Strength:** The relatively high tensile strength of Date Palm Fibers (DPFs), particularly those from the fronds and petioles, makes them robust and able to resist tensile strength and ductility [4];
- b. **Flexibility:** Although Date Palm Fibers (DPFs) are strong, especially the finer fibers from the mesh and spathe, they are highly flexible [4];
- c. **Hardness:** Tending to be stiffer than other kinds of Date Palm Fibers (DPFs), leaf fibers are appropriate for applications needing stiffness, including building materials, ropes, and baskets [127, 136].

**2. Durability and Resistance:** It will be as follows:

- a. **Biodegradability:** A natural fiber, Date Palm Fibers (DPFs) biodegrade and decay under environmental circumstances [3]. Especially in creating sustainable products, this makes them an environmentally favorable substitute for synthetic fibers;

**3. Physical Properties:** It will be as follows:

- a. **Density:** Date Palm Fibers' (DPFs) comparatively low weight helps them be used in light bio-composite products [137]. Their low density is desirable in automotive and building applications, where weight savings are crucial [138, 139];

**4. Thermal Properties:** It will be as follows:

- a. **Insulation:** Date Palm Fibers (DPFs) offer excellent thermal insulation, especially the thicker ones from the leaves and petioles [140]. They can be used in building materials and other applications that require heat resistance [141];

**5. Chemical Resistance:** It will be as follows:

- a. **Alkaline and Acid Resistance:** Though more prone to disintegration by strong acids or bases, Date Palm Fibers (DPFs) exhibit some tolerance to acidic and alkaline environments. They are usually stable in neutral pH settings [142];

**6. Environmental Properties:** It will be as follows:

- a. **Renewability:** Date palms are a sustainable resource. They may be constantly gathered without creating long-term environmental harm. The tree creates the fibers organically. Thus, they are a sustainable material [143];
- b. **Carbon Footprint:** Being a natural fiber, Date Palm Fibers (DPFs) have a smaller carbon footprint than synthetic materials, particularly in environmentally friendly goods like bio-composites and biodegradable packaging [10, 144];
- c. **Waste Reduction:** Using Date Palm Fibers (DPFs) helps to lower agricultural waste. Rather than throwing away or burning the by-products of date palm agriculture, the fibers are recycled into useful materials, supporting a circular economy [145, 146].

**7. Process Ability:** It will be as follows:

- a. **Ease of Extraction:** Simple mechanical methods allow for easy extraction of Date Palm Fibers (DPFs) from the many tree components. This enables large-scale manufacturing without the need for sophisticated or costly extraction methods [26];
- b. **Spinning and Weaving:** Date Palm Fibers (DPFs), particularly those from the leaves and mesh, are appropriate for spinning into yarns and weaving into textiles. The fibers can be employed in robust, flexible textiles for many items, such as mats, ropes, and bags [127];
- c. **Compatibility with Bio-Matrix:** Date Palm Fibers (DPFs) complement biodegradable matrix and bioplastics, improving their possibilities in creating bio-composites. This compatibility makes them a good candidate for application in sustainable manufacturing [147].

Ultimately, the qualities of Date Palm Fibers (DPFs) make them useful for a wide range of modern and traditional uses. They are a sustainable alternative to synthetic fibers since they are strong, flexible,



resistant to moisture, and biodegradable. This is especially true for making eco-friendly products and bio-composites. With increased knowledge and enhancement of the properties of Date Palm Fibers (DPFs), they may contribute even more notably to sectors oriented on sustainability and minimizing environmental effects.

## II.4. Extraction Methods of Date Palm Fibers

Depending on the tree component being utilized (e.g., Fronds, Petioles, Spathe, or Rachis) and the intended application, many methods are used to extract Date Palm Fibers (DPFs), as shown in [Figure II.04](#).

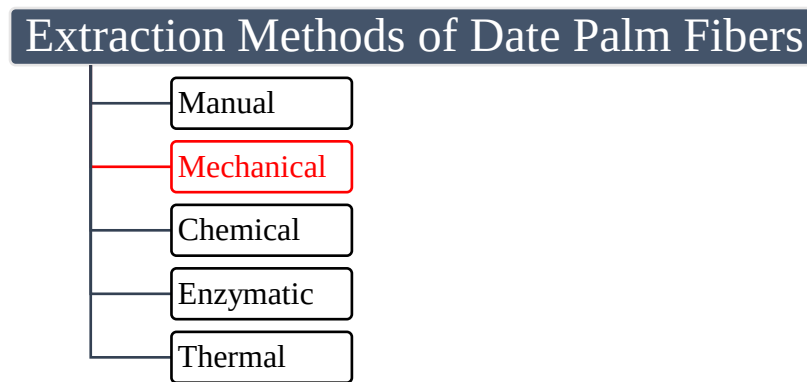


**Figure II.04. Extraction Methods of Date Palm Fibers.**

Usually, the aim is to isolate the fibers from the plant material while preserving their strength, flexibility, and other desirable qualities. The primary extraction methods for Date Palm Fibers (DPFs) are as follows:

- 1. Manual Extraction:** Usually, fibers are hand-picked from many sections of the date palm tree, particularly the fronds and petioles. Adult fronds of the date palm are then cut from the tree. After that, removing the outer layers of the fronds exposes the underlying fibrous structure. The fibers are manually separated once the fronds are peeled, either by hand or with simple tools like knives, combs, or scrapers. In addition, the procedure leaves the long, strong fibers by eliminating the non-fibrous material. Typically, the fibers are dried to stop deterioration and increase their lifetime after extraction [148];
- 2. Mechanical Extraction:** Especially in bio-composites and industrial applications, mechanical extraction methods are more effective for the large-scale manufacture of Date Palm Fibers (DPFs). Usually, this approach employs machinery meant to harvest fibers from plant material. The date palm's fronds, petioles, or spathes are cut into smaller bits. This stage facilitates

the breakdown of the plant material, hence facilitating fiber extraction. The chopped pieces are then put into mechanical scrapers or beaters that separate the fibers from the woody or non-fibrous portions of the plant. Some machines apply mechanical agitation to the plant material to release the fibers. After that, sifting, screening, or air classification removes the fibers from the residue. The shorter, less valuable fibers are discarded or used for something else, but the delicate, long fibers are kept for extra processing. Typically, the fibers that are taken out are dried to stop them from absorbing moisture, which might lower their quality over time [11];

- 3. Chemical Extraction (Retting):** Chemical extraction, sometimes known as retting, breaks down the lignin and hemicellulose, binding the plant tissue fibers using chemical solutions or fermentation. Often, this method is employed to separate fibers from fronds or the Rachis (Middle Layer) of date palm stalks. After that, the plant material (Fronds, Rachis, or Spathe) is first soaked in water or treated with chemical solutions such as alkali (Sodium Hydroxide) or acid. Soaking softens the tissue and loosens the fibers from the surrounding plant material. After that, the plant material is left to ferment or soak for several days. During this stage, microbes or chemicals break down the non-fibrous sections of the plant, making it easier to get the fibers out. After retting, the softened plant material has the fibers taken out. You can do this with mechanical methods like scraping, pounding, and hard work. After that, the removed fibers are washed to remove any chemical residues still there, and then dried [149];
- 4. Enzymatic Extraction:** Enzymatic extraction uses natural enzymes to break down the lignin and other elements holding the fibers together. This approach is more environmentally friendly than chemical extraction because it applies biodegradable enzymes rather than severe chemicals. Then, certain enzymes like cellulases or pectinases are used on the plant material fronds, petioles, or Rachis. These enzymes break down hemicellulose and pectin, which makes it easier for fibers to stick together. The plant material is soaked in a solution with the enzymes for a set amount, anywhere from a few hours to a few days. After the enzymatic treatment, mechanical methods like combing, scraping, or sifting separate the fibers. The fibers that were extracted are cleaned and dried properly to get rid of any leftover enzymes [150];
- 5. Thermal Extraction:** Thermal extraction applies heat to decompose the lignin and soften the plant material, facilitating the fiber extraction. Usually, this approach is used with other mechanical or chemical methods to enhance fiber output and quality. After that, the Date Palm Fiber (DPFs) (Fronds, Petioles, or Spathes) is subjected to high temperatures by steaming or using

hot air to soften the plant material. Mechanical methods, such as hammering or scraping, break the fiber after heat treatment. Heat makes fiber more flexible, so it recovers quickly. The fibers dry up to maintain strength and reduce moisture absorption [8].

Ultimately, different methods for different applications allow for the extraction of Date Palm Fibers (DPFs). While mechanical and chemical processes are more appropriate for large-scale, commercial manufacturing, manual extraction is usually employed for small-scale, traditional items. Enzymatic and thermal extraction technologies are better for the environment and work just as well as chemical-heavy approaches. As more people want sustainable resources, the growth of fiber extraction technologies will increase. These methods will be essential for using Date Palm Fibers (DPFs) best.

## II.5. Applications of Using Date Palm Fibers

Strong, durable, and sustainable Date Palm Fibers (DPFs) come from several sections of the palm tree and have many applications. As shown in [Figure II.05](#), these fibers are increasingly being used in conventional and modern applications.

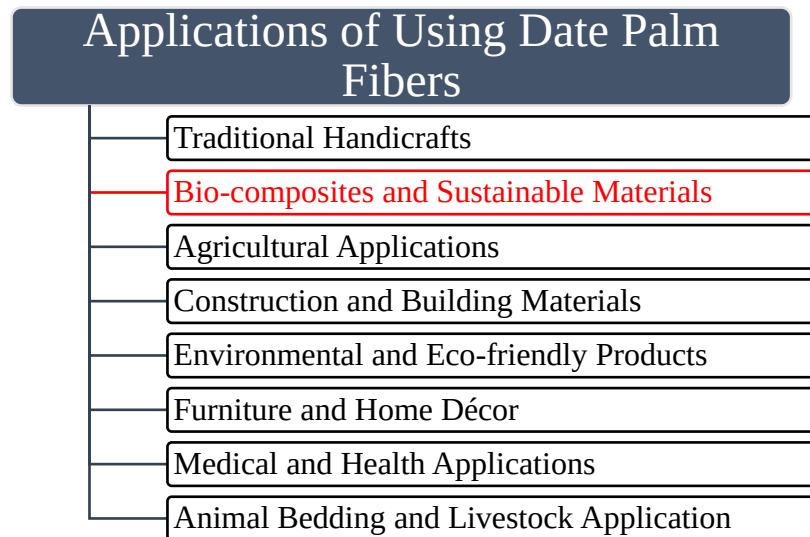


**Figure II.05. Applications of Using Date Palm Fibers.**

Some critical applications of Date Palm Fibers (DPFs) are listed below:

**1. Traditional Handicrafts:** As shown in [Figure II.06](#). It will be as follows:

- a. Baskets and Mats:** The most typical historical use for Date Palm Fibers (DPFs) is making baskets, mats, and other woven things [127]. The long, sturdy frond strands are often woven into complicated patterns to make valuable and strong goods;

- b. Ropes and Twine:** Ropes, twine, and other cordage are usually made from Date Palm Fibers (DPFs), especially those from the petioles (Leaf Stalks) [127]. Their strength and adaptability make these items very sought-after;
  - c. Bridges and Roofs:** Date Palm Fibers (DPFs) have historically been used to build temporary bridges and roofs in rural regions, as they are highly resilient to environmental wear and strain [151].
- 2. Bio-composites and Sustainable Materials:** As shown in [Figure II.06](#). It will be as follows:
  - a. Bio-composite Materials:** Date Palm Fibers (DPFs) are increasingly being used to make bio-composites. When mixed with bioplastics, matrix, or biodegradable polymers, these fibers make long-lasting materials used in building, the car industry, and packaging. These bio-composites may be utilized to create environmentally friendly packaging materials, structural panels in buildings, and interior components in automobiles [3, 152];
  - b. Textiles and Fabrics:** Textiles and fabrics may be made from the finer fibers of the date palm's spathe and Rachis. These fibers are soft, lightweight, and biodegradable, which makes them appropriate for upholstery, sustainable clothes, and other fabric-based items [2];
  - c. Bio-based Filaments for 3D Printing:** Date Palm Fibers (DPFs) are mixed with bioplastics to make 3D printing filaments. This makes it possible to create tailored, environmentally friendly products for the automotive, aerospace, and medical sectors [153].
- 3. Agricultural Applications:** As shown in [Figure II.06](#). It will be as follows:
  - a. Mulching and Soil Erosion Control:** Date palm fibers help stop soil erosion in farming. Spread across the top of the soil, they help keep moisture and stop wind and water erosion. This is particularly beneficial in dry areas where date palms are cultivated [154];
  - b. Compost and Organic Fertilizer:** Date Palm Fibers (DPFs) are a biodegradable substance commonly found in composting. Decomposing organically, they help provide rich organic material that may be utilized as fertilizer to enhance soil quality [155];
- 4. Construction and Building Materials:** As shown in [Figure II.06](#). It will be as follows:
  - a. Insulation Materials:** Date Palm Fibers (DPFs) are natural materials that keep heat in. Because they are good at keeping heat and sound out, they can be used instead of standard insulating materials, especially in eco-friendly building projects [156];

- b. Reinforced Concrete:** In some places, Date Palm Fibers (DPFs) are mixed with concrete to make it stronger and more durable [157]. The fibers make the concrete stronger and longer, which makes it suitable for use in low-cost houses, floors, and other building parts;
  - c. Roofing Materials:** Date Palm Fibers (DPFs) provide a strong and weatherproof roof. They can be utilized as thatching material or woven into panels. They are often used as a roofing material component in conventional and rural buildings [127].
- 5. Environmental and Eco-friendly Products:** As shown in [Figure II.06](#). It will be as follows:
  - a. Biodegradable Packaging:** Using Date Palm Fibers (DPFs) in biodegradable packaging is becoming increasingly common [10]. These fibers make strong packing materials that can be used instead of plastic items like shipping materials, wrappers, and food containers;
  - b. Biodegradable Straws and Utensils:** Researchers increasingly consider Date Palm Fibers (DPFs) a substitute for single-use plastics like cutlery and drinking straws. These items provide a sustainable way to cut plastic waste [158].
- 6. Furniture and Home Décor:** As shown in [Figure II.06](#). It will be as follows:
  - a. Furniture:** Date Palm Fibers (DPFs) are strong and flexible, which helps the furniture industry. They are often used with wood or metal frames to make chairs, tables, and other home decor pieces, which makes them light and long-lasting [159];
  - b. Decorative Items:** Date Palm Fibers (DPFs) produce handcrafted art pieces, wall hangings, lampshades, and other ornaments. Woven, twisted, or braided, these products may display the fibers' inherent texture in complex designs [160].
- 7. Medical and Health Applications:** As shown in [Figure II.06](#). It will be as follows:
  - a. Wound Dressings:** Date Palm Fibers' (DPFs) inherent qualities, namely their softness and absorbency, make them suitable for medicinal applications. Sometimes, Date Palm Fibers (DPFs) have been used in wound dressings to provide a biodegradable, non-toxic substitute for synthetic materials [161];
  - b. Filtration Materials:** Because of their delicate texture, Date Palm Fibers (DPFs) are suitable for filtering applications such as water filters or air purifiers. They also help create natural and sustainable filtration systems by capturing pollutants and particles [162, 163].
- 8. Animal Bedding and Livestock Application:** As shown in [Figure II.06](#). It will be as follows:

- a. **Bedding Material:** Date Palm Fibers (DPFs) are used to make bedding for animals [164]. The fibers soak up water, keeping the air moist and the animal's homes dry;
- b. **Animal Feed:** The fibrous material may sometimes be treated and utilized as roughage in animal feed, providing a sustainable choice for animal nutrition [165].



**Traditional  
Handicrafts**



**Sustainable  
Materials**



**Agricultural  
Applications**



**Construction  
Materials**



**Eco-friendly  
Products**



**Furniture and  
Home Décor**



**Medical  
Applications**



**Animal Bedding  
and Livestock**

**Figure II.06. Applications of Using Date Palm Fibers.**

Ultimately, Date Palm Fibers (DPFs) have several applications in several sectors because of their natural qualities, sustainability, and adaptability. From conventional handicrafts and agricultural applications to contemporary applications in bio-composites, textiles, and eco-friendly goods, Date Palm Fibers (DPFs) are becoming vital in driving toward more sustainable manufacturing methods. The applications of Date Palm Fibers (DPFs) are predicted to grow more as the need for ecologically friendly materials rises, helping create more sustainable goods in many industries.

## **II.6. Advantages and Disadvantages of Using Date Palm Fibers in Bio-composite Materials**

Though they have many advantages, employing Date Palm Fibers (DPFs) in different contexts presents particular disadvantages. Maximizing the potential of these fibers in contemporary sectors depends on overcoming these disadvantages and working with advantages, as shown in [Figure II.07](#).

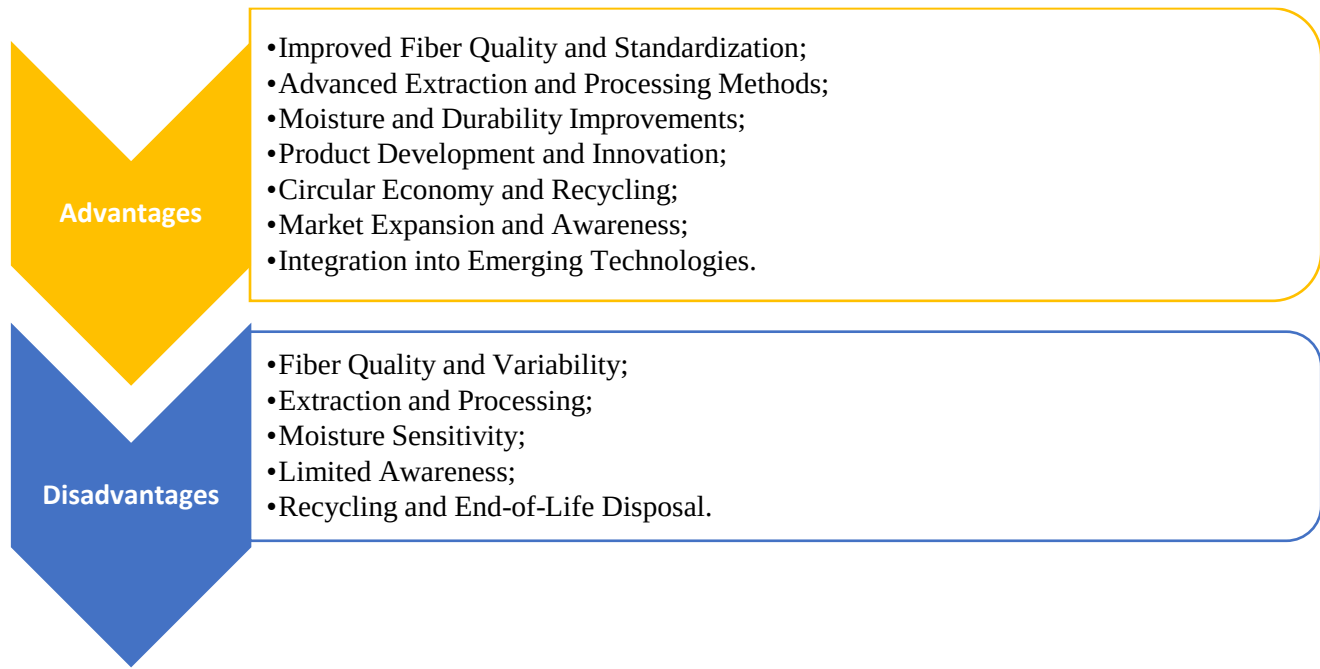


**Figure II.07. Advantages and Disadvantages of Using Date Palm Fibers in Bio-Composite Materials.**

The summary of advantages and disadvantages of using Date Palm Fibers (DPFs) in bio-composite materials in the following aspects:

### II.6.1. Advantages of Using Date Palm Fibers in Bio-composite Materials

The advantages of Date Palm Fibers (DPFs) seem bright, particularly as businesses emphasize eco-friendly solutions and sustainability. Among the possible advantages for the growth and more widespread application of Date Palm Fibers (DPFs) in bio-composite materials are the following aspects:

- 1. Improved Fiber Quality and Standardization:** It will be as follows:
  - a. Genetic Improvement of Date Palms:** Studies on breeding date palm trees with more consistent fiber qualities, for example, longer and stronger fibers, could significantly enhance the general performance of Date Palm Fibers (DPFs) in several applications [166];
- 2. Advanced Extraction and Processing Methods:** It will be as follows:
  - a. Eco-friendly Treatments:** Ongoing studies on non-toxic, eco-friendly therapies, e.g., plant-based or enzymatic methods for enhancing fiber-matrix bonding, might help lower the expense and environmental effects of chemical treatments [167].
- 3. Moisture and Durability Improvements:** It will be as follows:



- a. **Waterproofing Treatments:** Studies on natural or bio-based waterproofing treatments might strengthen the moisture resistance of Date Palm Fibers (DPFs), enhancing their performance when moisture sensitivity is a problem [137];
- b. **Hybrid Bio-composites:** Combining Date Palm Fibers (DPFs) with other fibers or materials such as synthetic fibers, bioplastics, or nanomaterials could enhance their mechanical characteristics, moisture resistance, and general performance in demanding applications [9].

**4. Product Development and Innovation:** It will be as follows:

- a. **New Applications:** Creating creative goods that employ Date Palm Fibers (DPFs) could help increase their application beyond conventional applications. For instance, including palm fibers in the manufacture of car components, packaging materials, or sophisticated construction materials might promote market acceptance [168];
- b. **Sustainable Packaging:** Date Palm Fibers (DPFs) provide a chance to produce biodegradable packaging solutions as the worldwide need for sustainable packaging grows [10]. Studies on how these fibers may replace plastic in single-application items, shipping materials, and food packaging would greatly help cut plastic waste.

**5. Circular Economy and Recycling:** It will be as follows:

- a. **Upcycling Waste Materials:** Using Date Palm Fiber (DPF) waste, such as short fibers or extraction process leftovers, in lower-value applications (e.g., Composite Panels or Biodegradable Plastic Alternatives) might help reduce total waste and foster a circular economy [10, 145].

**6. Market Expansion and Awareness:** It will be as follows:

- a. **Market Education:** The increasing use of Date Palm Fibers (DPFs) in sustainable applications will depend on how well the advantages of these fibers, like their low environmental impact and biodegradability, are promoted. Teaching businesses and consumers about the possibilities of Date Palm Fibers (DPFs) can help generate new demand in the packaging, automotive, and building industries [3];

**7. Integration into Emerging Technologies:** It will be as follows:

- a. **3D Printing and Advanced Manufacturing:** Growing additive manufacturing (3D Printing) uses Date Palm Fibers (DPFs) as a substitute for artificial filaments to produce environmentally friendly, tailored components in the aerospace, automotive, and medical sectors [169, 170].



- b. Innovative Materials:** Studies on Date Palm Fibers (DPFs), which are part of innovative materials and have characteristics that can be regulated or changed in reaction to external stimuli, could create new opportunities in sectors like building and electronics [26, 170].

Ultimately, through the aspects above, these advantages will provide a promising future for using Date Palm Fibers (DPFs) in bio-composite materials in their exploitation, environmental conservation, and application in various fields.

### II.6.2. Disadvantages of Using Date Palm Fibers in Bio-composite Materials

Although Date Palm Fibers (DPFs) offer several advantages, they also have several disadvantages. Getting beyond these disadvantages is necessary to get the most out of these fibers in modern fields. Among the main disadvantages are:

#### 1. Fiber Quality and Variability: It will be as follows:

- a. Inconsistent Quality:** Date Palm Fiber (DPF) quality can differ significantly depending on the section of the tree from which it is harvested fronds, petioles, Rachis, etc. and the environmental circumstances where the tree grows [171]. Variability like this makes it difficult to standardize fibers for industrial usage on a broad scale;
- b. Length and Strength:** While Date Palm Fibers (DPFs) from some regions of the tree (Like Fronds) are long and strong, others (Like from the Root) may be shorter or weaker. Their variable length and strength can influence the effectiveness of fibers in many applications, particularly in high-strength composite materials [11].

#### 2. Extraction and Processing: It will be as follows:

- a. Complex Extraction Methods:** Eliminating fibers from date palms, especially from tough spots like the petioles and spathes, can be challenging. You might need special instruments to do this. Mechanical procedures might not work and could harm the fibers, but conventional manual methods take a lot of time [172, 173];

#### 3. Moisture Sensitivity: It will be as follows:

- a. Hygroscopic Nature:** Date Palm Fibers (DPFs) naturally absorb moisture from the air [174]. This can make things less stable in size, less strong, and even cause mold to grow, especially in humid places [175]. Controlling the humidity in things made from Date Palm Fiber (DPF) ensures they last and operate well.

#### 4. **Limited Awareness:** It will be as follows:

- a. **Lack of Awareness:** Even though Date Palm Fibers (DPFs) are better for the environment, many industries still rely on synthetic and petroleum-based products. We should discuss the benefits of using natural fibers like date palms for long-lasting uses [176];

#### 5. **Recycling and End-of-Life Disposal:** It will be as follows:

- a. **Recycling Challenges:** Date Palm Fibers (DPFs) can break down naturally, but have not yet been recycled into new materials or items [145]. To promote circular economies and reduce waste, we need to ensure that date palm goods can be recycled easily.

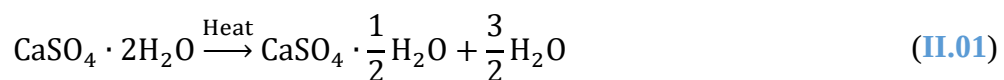
Ultimately, the disadvantages of using Date Palm Fibers (DPFs) in bio-composites are many yet surmountable. The bio-composites enable the realization of their objectives to save the environment and repurpose resources for practical applications in daily life.

To conclude, Date Palm Fibers (DPFs) have much promise as a long-lasting, eco-friendly alternative to manufactured materials. However, these fibers still have disadvantages, such as inconsistent quality, harrowing extraction, and sensitivity to moisture. Further research, improved manufacturing methods, and the development of new products are making it possible for them to be used by more people. Date Palm Fibers (DPFs) have a bright advantage because they are becoming more widely used in bio-composites, fabrics, packaging, building, and other fields that care about the environment and sustainability.

## II.7. Definition of Gypsum

Compared to calcium sulfate dehydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), gypsum is a naturally occurring mineral [177]. This soft mineral, often white or colorless, may appear gray, yellow, or pink due to impurities [178-180]. Gypsum crystallizes in evaporation conditions, causing water to evaporate and accumulating salts [181]. It is used in many areas, including construction, industry, and agriculture [182, 183].

In addition, it is important in agriculture as it acts as soil, increasing drainage, water retention, and soil structure [184]. The gypsum can be converted to gypsum Paris ( $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ ) applications in art, medicine, and construction repair on heating [185, 186], as shown in Equation (II.01) [187].



Ultimately, gypsum is a highly efficient thermal insulation matrix and possesses diverse properties that make it a preferred choice for the manufacture of bio-composites.

## II.8. Types of Gypsum

Depending on its source, processing, and intended application, gypsum has several types. The primary kinds of gypsum are classified according to their natural and synthetic sources and characteristics, as shown in [Figure II.08](#).

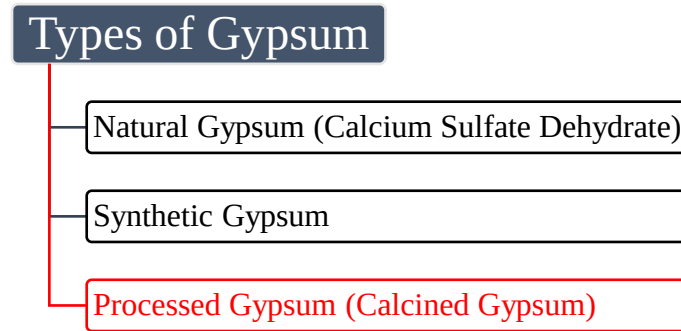


**Figure II.08. Diagram-Types of Gypsum.**

The following is a summary of the key types:

1. **Natural Gypsum (Calcium Sulfate Dehydrate):** Natural mineral resources provide this gypsum [188]. Though it may be processed to raise its grade for particular applications, it is usually mined and utilized without significant change, and they have many types, such as:
  - a. **Phosphor-Gypsum:** The phosphate fertilizer sector produces this as a byproduct. It contains calcium sulfate dehydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) but might include impurities like phosphorus and trace elements [189]. It is commonly applied in road building and farming (See [Figure II.09](#)) [189, 190];
  - b. **Selenite Gypsum:** Evaporitic deposits often provide a natural gypsum in salty settings. It has precise, crystal-like shapes. Its purity and beauty make it suitable for decorative applications and occasionally for gypsum manufacturing (See [Figure II.09](#)) [191];
  - c. **Alabaster:** A large, finely grained kind of gypsum. Often white, transparent, or colored (Yellow, Brown, or Pink). Its smooth texture and simplicity of carving make it primarily for sculpting and ornamental arts (See [Figure II.09](#)) [192].
2. **Synthetic Gypsum:** Industrial operations produce synthetic gypsum, specifically, power plants' flue gas desulfurization (FGD) process. Other industrial activities, including the manufacture of phosphoric acid or the paper sector, might also cause it [193], and they have many types, such as:

- a. **Flue-Gas Desulfurization (FGD) Gypsum:** Made when coal-burning power stations' exhaust gases lose sulfur dioxide ( $\text{SO}_2$ ). Essentially the same as natural gypsum (Calcium Sulfate Dehydrate), but with fewer contaminants. Often used in the building sector, especially for making gypsum boards and drywall (See [Figure II.09](#)) [194];
  - b. **Phosphor-gypsum:** The process of producing phosphate fertilizer creates a byproduct. Contains calcium sulfate but might have minute quantities of radioactive materials, including radon [189]. Though its usage in construction is controlled because of possible radiation issues, it is sometimes employed in road building or for agricultural applications (See [Figure II.09](#)) [189, 190].
3. **Processed Gypsum (Calcined Gypsum):** Heated (Calcination) processed gypsum loses water and changes its characteristics [195]. Construction and industrial applications often make use of these processed forms, and they have many types, such as:
- a. **Gypsum of Paris (Calcined Gypsum):** Heated to around  $150^\circ\text{C}$  ( $302^\circ\text{F}$ ), gypsum drives out part of the water, creating this. Calcium sulfate hemihydrate ( $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ ) is the outcome [196]. Used for manufacturing dental molds, casting, gypsum walls, and molding. When combined with water, it hardens fast and is commonly employed in art and building (See [Figure II.09](#)) [180];
  - b. **Gypsum Board (Drywall):** This item consists of calcined gypsum, mostly calcium sulfate hemihydrate, pressed between two sheets of paper. It is often used to build interior walls and ceilings in houses and businesses (See [Figure II.09](#)) [197];
  - c. **Gypsum Cement:** A result of calcined gypsum combined with other substances and used for castings, precast molds, and other applications needing high-density, fast-setting cement (See [Figure II.09](#)) [198].



**Figure II.09. Types of Gypsum.**

Ultimately, every type of gypsum has particular applications depending on its purity, composition, and the amount of processing it undergoes.

## II.9. Properties of Using Gypsum

A common and flexible substance, gypsum has many key physical, chemical, and mechanical properties that make it useful in many sectors, including building, agriculture, and industry, as shown in [Figure II.10](#).

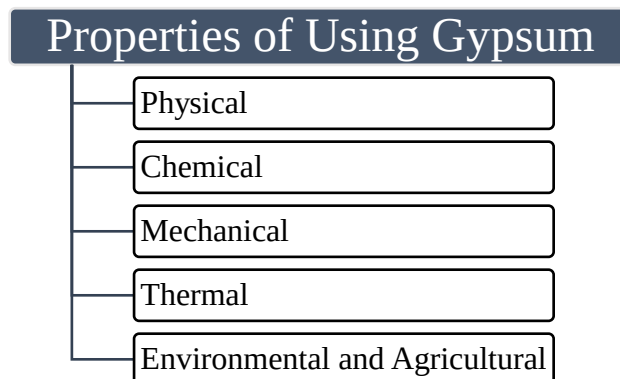


**Figure II.10. Properties of Using Gypsum.**

The main properties of gypsum are listed below:

**1. Physical Properties of Gypsum:** It will be as follows:

- a. **Color:** Gypsum is usually white or colorless, but it can also be yellow, gray, brown, or pink if it has iron or organic matter in it. You may sometimes tell what minerals are in gypsum by looking at its color. For instance, iron can change its color to red or yellow [180, 199, 200];
- b. **Hardness:** With a Mohs hardness of two, gypsum is a somewhat soft mineral. A fingernail can scratch it. Though some varieties, like Gladstone, might be more solid and compact, gypsum is smooth and fine-grained in its pure form [201];
- c. **Crystal Structure:** The gypsum occurs in monoclinic crystal systems, usually as prismatic, tabular, or bladed crystals. In its natural state, gypsum can also be found as fibers or large clumps. A kind of gypsum called selenite is famous for its clear crystals that may grow in a lengthy, column-like shape [202];
- d. **Cleavage:** Perfect cleavage along the (010) plane in gypsum allows it to split into thin sheets. The gypsum is utilized in drywall and other gypsum applications partly because of its cleavage quality, which allows for simple cutting into thin, consistent sheets [203];
- e. **Density:** The gypsum's density is around 0.9-1.4 g/cm<sup>3</sup>, which is somewhat low. Its lightweight makes it perfect for building (e.g., Drywall) and as an agricultural resource [204];
- f. **Solubility:** The gypsum is only marginally soluble in water; temperature and ion concentration affect solubility. It dissolves in water to free sulfate and calcium ions, causing it to saturate and facilitating agricultural and soil enhancement applications [205].

**2. Chemical Properties of Gypsum:** It will be as follows:

- a. **Chemical Composition:** The gypsum's chemical formula is  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . Its composition is calcium sulfate dehydrate. The main chemical ingredient of gypsum is calcium sulfate ( $\text{CaSO}_4$ ).  $\text{H}_2\text{O}$  Integral to its structure, gypsum includes two water molecules [206];
- b. **Reactivity:** Heating (Calcining) gypsum to around 150°C causes it to lose some of its water content and form calcium sulfate hemihydrate (Gypsum of Paris) ( $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ ) [207]. Mixing the gypsum of Paris with water causes it to rehydrate and generate gypsum again, enabling its application in applications needing setting and hardening [208];
- c. **PH:** The gypsum is either neutral or slightly alkaline. Its lack of significant acidic or basic qualities makes it perfect for agricultural soil conditioning [209];

- d. **Non-Flammability:** Gypsum is fire-retardant and non-flammable, making it perfect for use in fire-resistant drywall and construction materials. Two water molecules in gypsum serve as a fireproofing agent by helping absorb heat under fire exposure, preventing more material from burning [210].

**3. Mechanical Properties of Gypsum:** It will be as follows:

- a. **Compressive Strength:** Gypsum has moderate compressive strength (2-7 MPa), which makes it useful for building, especially in drywall and gypsum. The compressive strength of gypsum depends on whether it is raw, calcined, or treated [14].
- b. **Bending Strength:** Compared to other building materials, including concrete or steel, gypsum has relatively low bending strength [211]. It is usually used in gypsum or wallboard applications lacking a significant structural load [197]. Under pressure, gypsum can be brittle and breakable [212];
- c. **Moisture Resistance:** The water in gypsum's crystal structure allows it to absorb humidity from the atmosphere. Hydration is reversible, though gypsum will release water under high heat exposure [213].

**4. Thermal Properties of Gypsum:** It will be as follows:

- a. **Heat Insulation:** With its excellent thermal insulation qualities, gypsum helps develop energy-efficient structures. It delays the movement of heat, keeping indoor temperatures low and lowering energy consumption [214];
- b. **Thermal Conductivity:** Because it is a poor heat conductor, gypsum is an excellent material for constructing insulation [215];
- c. **Fire Resistance:** The gypsum's water molecules absorb heat, helping to stop fires from spreading. Often used in fire-resistant drywall, gypsum improves building safety during fires [216].

**5. Environmental and Agricultural Properties:** It will be as follows:

- a. **Soil Conditioning:** Gypsum is a typical soil conditioner in farming, especially for sodic soils, which are soils that have much salt in them [217]. By replacing sodium ions with calcium ions, you can improve the structure, drainage, and root penetration of the soil. Gypsum lowers the soil's alkalinity without changing the pH considerably [218];
- b. **Biodegradability:** The gypsum is a natural mineral that is biodegradable in its unprocessed state. It may be returned to the environment safely and without damaging ecosystems [219].

Ultimately, from drywall to soil conditioning, gypsum's properties make it perfect for various building, agriculture, and industrial applications. Its non-toxic character and capacity to absorb heat and moisture increase its flexibility as a construction material.

## II.10. Extraction Methods of Gypsum

Gypsum extraction is the method of getting gypsum, calcium sulfate dehydrate,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , from natural deposits or industrial by-products. There are several ways to get gypsum, mainly mining from natural resources and synthesizing from industrial by-products, as shown in [Figure II.11](#).

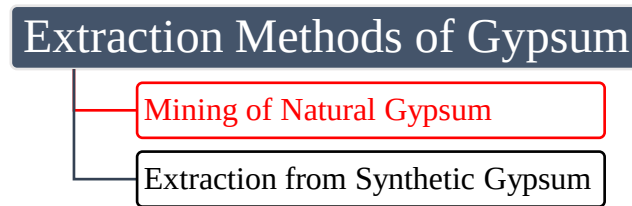


**Figure II.11. Extraction Methods of Gypsum.**

The extraction methods for gypsum are briefly described below:

- 1. Mining of Natural Gypsum:** It will be as follows:
  - a. Open-Pit Mining (Surface Mining):** Earth's surface, this is the most common way to remove it. Open-pit mining is digging big, shallow holes where gypsum reserves are near the surface. Using bulldozers or other heavy equipment, the first stage removes the overburden soil and rock layers covering the gypsum. Drilling, blasting, and excavating remove the gypsum layer once uncovered. Usually found in enormous, readily available quantities, the gypsum fits the required criteria for industrial applications; the gypsum is crushed and filtered into smaller pieces following extraction. Trucks, conveyors, or rail carry the extracted gypsum to processing facilities for more refinement (See [Figure II.12](#)) [220];
  - b. Underground Mining:** Deep subterranean gypsum resources call for underground mining methods. Reaching the gypsum ore with this approach calls for shaft or tunnel excavation, making it more involved. Vertical shafts or slanted tunnels are built to reach the gypsum deposit. Various methods, such as room and pillar mining (Leaving Pillars of Gypsum to Support the Ceiling) or cut and fill mining (Removing Gypsum in a Way That Allows for Controlled Backfilling of the Mined Area), are used to harvest gypsum from the subsurface deposit. Mined gypsum is brought to the surface for processing (See [Figure II.12](#)) [221].



**2. Extraction from Synthetic Gypsum:** As a byproduct of industrial processes, synthetic gypsum is important to extract since it helps reduce waste and utilize other resources. Some of the most important ways to get synthetic gypsum are:

- a. Flue-Gas Desulfurization (FGD):** Flue-Gas Desulfurization (FGD) is used by coal-fired power plants to remove sulfur dioxide ( $\text{SO}_2$ ) from the exhaust gases that come from burning coal. Synthetic gypsum produced by this technology is chemically comparable to natural gypsum. A slurry of lime ( $\text{CaO}$ ) and water in the scrubber system absorbs sulfur dioxide ( $\text{SO}_2$ ) from the exhaust gases. Calcium sulfate ( $\text{CaSO}_4$ ) is produced by the reaction of sulfur dioxide with calcium oxide (Lime); this then interacts with water to create calcium sulfate dehydrate (Gypsum). Filtration or settling removes the gypsum from the slurry; then, it is dried and treated to satisfy the required quality (See [Figure II.12](#)) [222, 223];
- b. Phospho-Gypsum:** Produced from phosphate rock, phosphor-gypsum is a by-product of phosphoric acid manufacture. Used in fertilizer manufacture, the phosphorus extraction from phosphate ore generates it. Sulfuric acid treats phosphate rock to create phosphoric acid. The by-product of this process is phosphor-gypsum, mostly made of calcium sulfate but possibly containing minute quantities of radioactive elements like radon. Filtration removes the phosphoric acid from the phosphor-gypsum. Although its usage may be limited in specific applications due to potential radioactivity, it can be dried and processed in agricultural settings or buildings (See [Figure II.12](#)) [224].

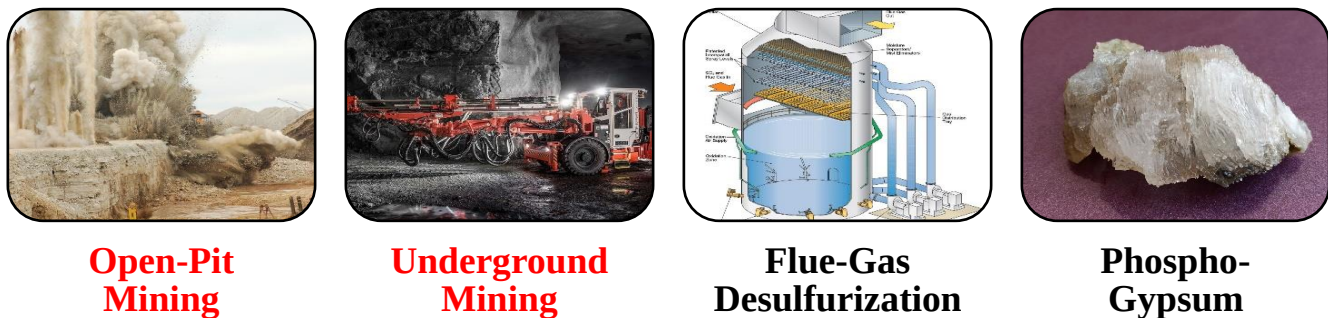


**Figure II.12. The Extraction Methods of Gypsum.**

Ultimately, many methods are used to get gypsum, depending on where it comes from and what the sector needs [180]. Flue-Gas Desulfurization (FGD) and phosphor-gypsum are important ways to make synthetic gypsum, although open-pit and underground mining are the most common ways to get natural gypsum [222, 223]. Environmental circumstances, cost-efficiency, and the required quality of the gypsum will guide the selection of the extraction method.

## II.11. Applications of Using Gypsum

Because of its unique qualities of lightweight, fire resistance, and thermal insulation, gypsum is a flexible material with several applications in many sectors. [Figure II.13](#). Shows the main applications of gypsum in several areas.

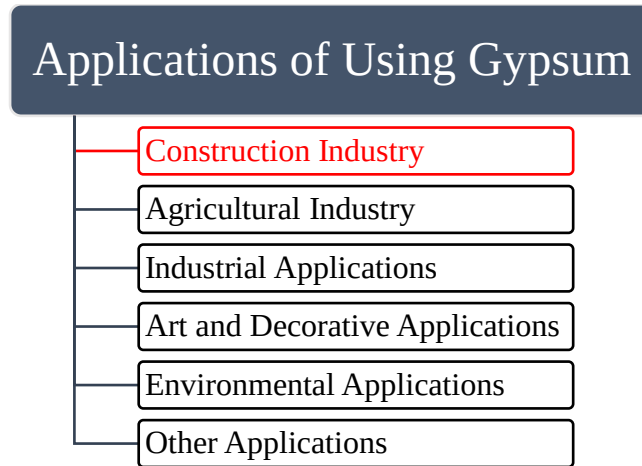


**Figure II.13. Applications of Using Gypsum.**

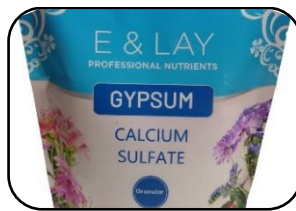
The following aspect is a summary of the main applications of gypsum in several areas:

- 1. Construction Industry:** As shown in [Figure II.14](#). It will be as follows:
  - a. Drywall (Gypsum-board):** A basic material in construction interiors, drywall or gypsum board, is usually made from gypsum [225];
  - b. Cement Manufacturing:** Portland cement's setting time is controlled by adding gypsum [198];
  - c. Fire-Resistant Materials:** When used in building materials, gypsum offers fire resistance because of its water crystallization [226];
  - d. Insulation:** With strong thermal insulation qualities, gypsum is helpful in energy-efficient buildings [214].
- 2. Agricultural Industry:** As shown in [Figure II.14](#). It will be as follows:
  - a. Soil Amendment:** Often, gypsum is utilized as a soil conditioner to enhance soil structure [209];
  - b. Improving Crop Yield:** The gypsum gives plants calcium and sulfate, among other vital elements [184].
- 3. Industrial Applications:** As shown in [Figure II.14](#). It will be as follows:

- a. **Flue-Gas Desulfurization (FGD):** Many applications of synthetic gypsum are generated as a by-product of Flue-Gas Desulfurization (FGD) in power plants [204];
  - b. **Water Treatment:** Particularly in wastewater treatment, various water treatment procedures employ gypsum [227].
- 4. Art and Decorative Applications:** As shown in [Figure II.14](#). It will be as follows:
- a. **Sculptures and Molds:** The gypsum of Paris (Calcined Gypsum) is used for sculpting and molding [228, 229];
  - b. **Architectural Details:** Architectural decoration in structures makes a great application of gypsum-based materials [230].
- 5. Environmental Applications:** As shown in [Figure II.14](#). It will be as follows:
- a. **Wastewater Treatment:** Wastewater treatment applications include gypsum to extract phosphate from industrial effluent or sewage [231];
- 6. Other Applications:** As shown in [Figure II.14](#). It will be as follows:
- a. **Dental Materials:** In dentistry, gypsum is used to create casts and molds [232];
  - b. **Food Additive:** Some goods use gypsum as a food additive (E516) [233].



**Construction  
Industry**



**Agricultural  
Industry**



**Industrial  
Applications**



**Art and  
Decorative**



**Environmental**



**Dental  
Materials**

**Figure II.14. Applications of Using Gypsum.**

Ultimately, the adaptability and stability of gypsum make it an essential material for many different regions. Its ability to increase thermal insulation, fire resistance, sound insulation, and soil quality guarantees that it will be a vital factor for industrial applications, agriculture, and building

[209, 214, 234, 235]. Whether gypsum board, fertilizer, or artistic sculpture, gypsum provides many benefits in both practical and creative domains.

## II.12. Advantages and Disadvantages of Using Gypsum in Bio-Composite Materials

Although frequently employed in many sectors, gypsum presents certain mining, processing, and application difficulties in particular situations. Research and invention still provide possible answers, but gypsum's sustainability and adaptability keep pushing its progress. Maximizing the potential of this material in contemporary sectors depends on overcoming these disadvantages and working with the advantages, as shown in [Figure II.15](#).

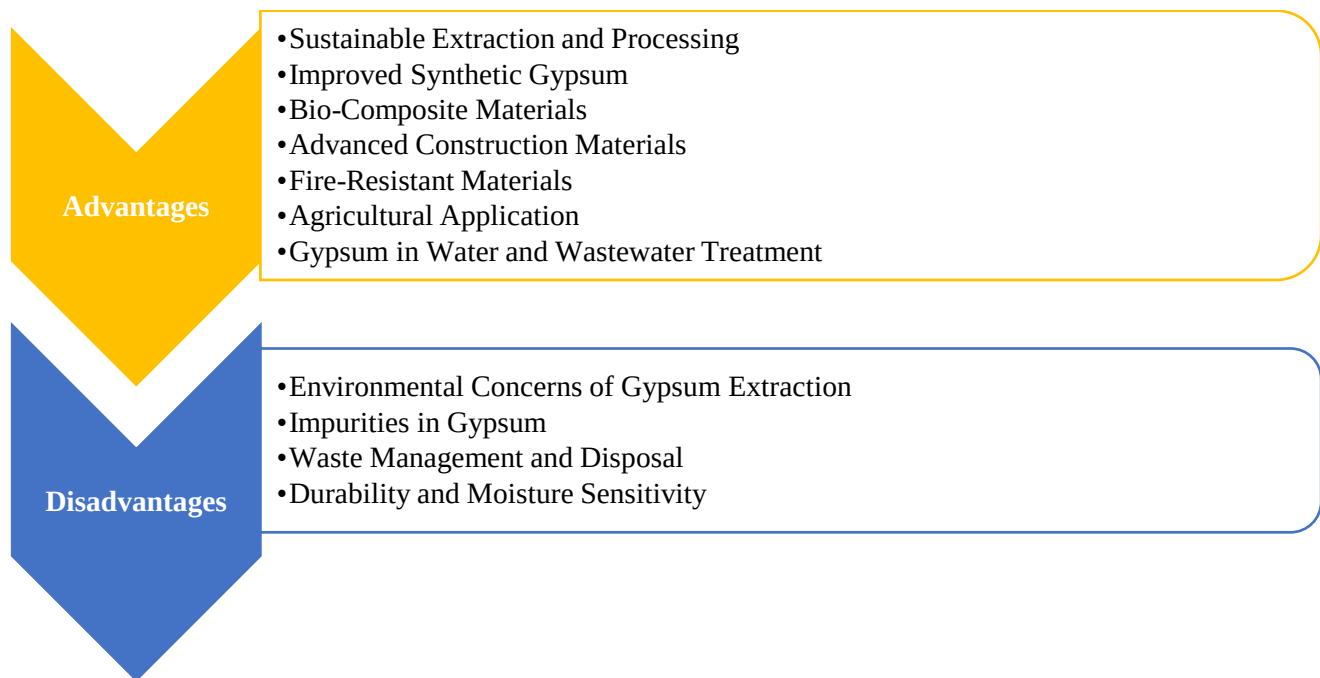


**Figure II.15. Advantages and Disadvantages of Using Gypsum in Bio-Composite Materials.**

The overview of the advantages and disadvantages of using gypsum in bio-composite materials is presented in the following aspects:

### II.12.1. Advantages of Using Gypsum in Bio-composite Materials

Though difficult, the future of gypsum seems bright, particularly as businesses emphasize eco-friendly solutions and sustainability. Among the possible advantages for the growth and more widespread application of gypsum in bio-composite materials are:

**1. Sustainable Extraction and Processing:** It will be as follows:

- a. **Green Mining Technologies:** Sustainable mining methods hold the key to gypsum manufacturing in the future. Addressing environmental issues will depend on technologies that reduce carbon emissions in mining and processing, land degradation, and water consumption. This covers energy-efficient calcination methods, water treatment strategies, and closed-loop systems [236];
- b. **Utilizing Industrial By-products:** Improvements in using synthetic gypsum from industrial by-products, such as coal-fired power plants (Flue-Gas Desulfurization (FGD) Gypsum) and phosphoric acid manufacture, can help lower the environmental impact of gypsum manufacturing while offering a valuable resource for several applications [19, 20];
- c. **Enhanced Recycling Method:** Advances in gypsum recycling technologies must enhance the efficiency and cost-effectiveness of gathering, processing, and reusing gypsum waste. A better classification of gypsum-rich waste from building and demolition will also enable more effective and broad recycling initiatives [19, 237].

**2. Improved Synthetic Gypsum:** It will be as follows:

- a. **Purity Enhancements:** Developing fresh methods to raise the purity of synthetic gypsum generated from industrial waste, such as Flue-Gas Desulfurization (FGD) and phosphor-gypsum, can create new markets for its utilization, particularly in sensitive food processing or agriculture sectors [238, 239];
- b. **Radioactive Gypsum Disposal:** Solutions for controlling the radioactive elements in phosphor-gypsum depend on safe disposal and alternate usage. Continual study could reduce the environmental concerns connected to radioactive elements, allowing phosphor-gypsum to be used [240].

**3. Bio-Composite Materials:** The gypsum-based bio-composites, a combination of gypsum and natural fibers, are being investigated for building and other applications as demand for sustainable and biodegradable materials rises [17, 241];**4. Advanced Construction Materials:** It will be as follows:

- a. **3D Printing with Gypsum:** 3D printing methods for construction and architecture can apply gypsum, producing intricate, bespoke buildings with shorter construction timeframes and lower material prices [242, 243];

- b. Smart Gypsum Products:** Developing innovative gypsum materials with enhanced thermal and acoustic insulation properties, as well as self-healing capacity, for smart buildings or integrated sensors is attracting increasing interest. The gypsum-based panels that may respond to temperature or humidity changes, for example, represent the future of flexible, durable structural materials [17, 244-246].
- 5. Fire-Resistant Materials:** As fire safety rules become increasingly strict, innovations in gypsum-based fire-resistant materials will probably follow. These might be gypsum board products with improved fire resistance, insulating qualities, and high-heat structural integrity [234].
- 6. Agricultural Application:** It will be as follows:
  - a. Soil Remediation:** New complicated applications for soil health improvement could be developed even if gypsum is currently used as a soil conditioner, given problems like saline soils, erosion, and damaged land [209, 217];
  - b. Nutrient Delivery:** In areas with low sulfur soils or high salt concentrations, gypsum-based fertilizers with regulated calcium and sulfate release might be more important for sustainable agriculture methods [184].
- 7. Gypsum in Water and Wastewater Treatment:** It will be as follows:
  - a. Phosphate Removal:** However, as the need for clean water grows, progress will be made in using gypsum to clean up dirty water. Gypsum-assisted filtration and other technologies may become more important for cleaning up wastewater and stopping lakes and rivers from becoming too rich in nutrients [227, 247].

Ultimately, through the aforementioned aspects, these advantages will provide a promising future for using gypsum in bio-composite materials in their exploitation, environmental conservation, and application in various fields.

### II.12.2. Disadvantages of Using Gypsum in Bio-composite Materials

Though they have many advantages, employing gypsum in different contexts presents specific disadvantages. Maximizing the potential of this material in contemporary sectors depends on overcoming these disadvantages. Among the main disadvantages are:

- 1. Environmental Concerns of Gypsum Extraction:** It will be as follows:
  - a. Mining Impact:** Taking natural gypsum from the ground or the air can cause a lot of damage to the environment, such as destroying habitats, polluting water, and spreading dust [19, 248];
  - b. Energy Consumption:** The process of calcining gypsum involves heating it to produce gypsum for use in Paris, which consumes energy and generates carbon emissions. Mining and processing can leave many carbon footprints [249];
  - c. Water Application:** The mining and processing of gypsum require a significant amount of water, which could lead to water shortages in some places [250].
- 2. Impurities in Gypsum:** It will be as follows:
  - a. Contaminants:** Natural gypsum deposits can include contaminants such as iron, silica, and magnesium. These contaminants might compromise the performance and quality of the gypsum in bio-composites, gypsum, and other goods [251];
  - b. Synthetic Gypsum:** Although synthetic gypsum from industrial methods like Flue-Gas Desulfurization (FGD) provides environmental advantages, it can occasionally include minute contaminants (e.g., Heavy Metals and Radioactive Elements from Phospho-Gypsum), which could restrict its applications, particularly in agriculture or building [204].
- 3. Waste Management and Disposal:** It will be as follows:
  - a. Phospho-Gypsum Waste:** Phospho-gypsum, a by-product of phosphate fertilizer manufacture, contains tiny quantities of radioactive elements, including radon. This raises environmental issues with its disposal and usage in particular applications [252];
  - b. Gypsum Recycling:** Although gypsum products like drywall may be recycled, effectively gathering, processing, and utilizing gypsum waste is difficult. Not all gypsum-recycling initiatives are financially feasible, and not everyone recycles waste gypsum [19, 253].
- 4. Durability and Moisture Sensitivity:** It will be as follows:
  - a. Water Absorption:** The gypsum can attract moisture from the environment, especially in its natural state. This can decrease the number of users who are exposed to high humidity or rain conditions. When the water is exposed, a standard gypsum board can be bought, or worse. Thus, some places, such as bathrooms or kitchens, require moisture-resistant versions [254];
  - b. Fire Resistance Limitations:** Although gypsum is fire-resistant, especially in building applications, continuous exposure to fire or extremely high temperatures can compromise its integrity [234, 255].

Ultimately, the disadvantages confronting the application of gypsum in bio-composites are many yet surmountable. The bio-composites also enable the realization of their objectives to save the environment and repurpose resources for practical applications in daily life.

To conclude, while important difficulties include gypsum mining, impurities, waste management, and moisture sensitivity, the benefits of gypsum include stress sustainability, inventiveness, and better performance. The gypsum will change as a primary material in industrial, agricultural, and building applications with continuous research on recycling, synthetic gypsum, bio-composite materials, and fire-resistant technologies [180]. These advances will maximize the sustainability and efficiency of gypsum-based goods and reduce environmental effects.

### **II.13. Literature Investigations of Bio-composite Materials Composed of Palm Fibers and Gypsum**

The sustainability of bio-composite materials makes them more favored as renewables [256]. The bio-composite materials that utilize gypsum as a matrix and palm fiber as reinforcement underscore their ecological significance and applications in the subsequent studies:

- 1. Mohamed BOUMHAOUT and his colleagues (2025)** [257], assert that their primary objective, developing bio-composite materials composed of mortar reinforced with plant waste, is garnering more attention as a construction resource. The thermos-physical assessment of mortar reinforced with Moroccan Date Palm Fibers (DPFs) was conducted using the hot wire and hot plane methods (FP2C), which assess thermal conductivity and thermal effusivity, respectively. The study characterized mortar reinforced with varying volume percentages of Moroccan Date Palm Fibers (DPFs), ranging from 0-44%.

The findings indicate favorable thermal performance of the Mortar Date Palm Fibers (MDPFs). Date Palm Fibers (DPFs) in the mortar reduces thermal conductivity by 53%, enhancing its insulating properties. The thermal effusivity of this material has decreased by as much as 48.5%. Date Palm Fibers (DPFs) reduce heat diffusivity by up to 44%, alter the mortar's density by as much as 33%, and diminish its volumetric capacity by up to 51%. The self-consistent model predicts the measured thermal conductivity with an average relative error above 11%. This means that the analyzed bio-composite materials are heterogeneous materials, including three components: a solid matrix, Date Palm Fibers (DPFs), and air, interspersed throughout [257].



- 2. Houssam Eddine BENCHOUIA and his colleagues (2024)** [9], assert that their primary objective in conducting research is safeguard the environment by repurposing two prevalent wastes in Algeria, Date Palm Petiole (DPP) fibers and Expanded Polystyrene Waste (EPS), to create a gypsum Plaster hybrid bio-composite material that is equivalent to or superior to Neat Gypsum Plaster (NGP). They employed hand-made specimens with varying Date Palm Petiole (DPP) mass loadings (0, 5, 10, and 15%), Expanded Polystyrene Waste (EPS) mass ratios (0.3%), or both. They conducted extensive testing on the hybrid composites we developed. These tests looked at its shape, strength, and heat transfer.

The Fourier Transform Infrared Spectroscopy (FT-IR) displays the peaks that are unique to gypsum, polystyrene, and fibers. X-Ray Diffraction (XRD) shows that Neat Gypsum Plaster (NGP), Expanded Polystyrene Waste (EPS), and Date Palm Petiole (DPP) have crystallinity levels of 71.58, 29.91, and 52.93%, respectively. The compression strengths, flexural strengths, and Young's modulus demonstrate that Expanded Polystyrene Waste (EPS), Date Palm Petiole (DPP), or both substantially diminish the mechanical parameters of the produced specimens. The bio-composites showed lower thermal conductivity ( $\lambda$ ) (0.265-0.414 W/m.K at 24°C) and bulk density ( $\rho$ ) (852-925 kg/m<sup>3</sup>) than reference Neat Gypsum Plaster (NGP) specimens, which had thermal conductivity of 0.425 W/m.K and bulk density of 977 kg/m<sup>3</sup>. A comparative study shows that gypsum Plaster reinforced with Date Palm Petiole (DPP), Expanded Polystyrene Waste (EPS), or both could be a good substitute for insulating materials, making it a long-lasting option for building.

- 3. Youssef KHRISSI and his colleagues (2024)** [258], assert that their primary objective in conducting research is to enhance the thermal properties of gypsum by using Date Palm Fibers (DPFs) and creating an outside wall covering. They conducted several experimental measurements of thermos-physical parameters to evaluate the thermal properties of the resulting gypsum-DPF bio-composite material. This research employed a steady-state hot plate method to ascertain thermal conductivity and bulk density.

Experimental methods assessed gypsum specimens' bulk density and thermal conductivity with varying mass fractions of Date Palm Fibers (DPFs) (0, 1, 2, 3, 4, and 5%). The findings indicate that around 26.24% of the composite material's 5% Date Palm Fiber (DPF) significantly reduces thermal conductivity. Furthermore, Date Palm Fiber (DPF) results in a 17.16% reduction in the

density of the construction material, hence decreasing its weight. These findings emphasize the potential and significance of utilizing this bio-composite material in buildings to enhance thermal comfort and reduce greenhouse gas emissions, including CO<sub>2</sub> emissions [258].

- 4. Asma SOUIDI and his colleagues (2024)** [259], assert that their primary objective proposed to investigate the viability of generating gypsum-based bio-composite materials utilizing plant fibers obtained from date palm waste (Date Palm Fibers (DPFs)) to exploit a local resource and application synthetic polyester fibers (Polyester Fibers (PF)) for comparison. Four varieties of mixes of gypsum-based bio-composite materials have been developed: the first is pure gypsum, the second consists of gypsum with (0.5% Date Palm Fibers (DPFs)), the third includes (0.5% Polyester Fibers (PF)) and the last comprises both types of fiber (0.25% Date Palm Fibers (DPFs) + 0.25% Polyester Fibers (PF)).

In the tests conducted on the bio-composite materials based on the physical characteristics and morphology of the two types of fiber, they found that Date Palm Fibers (DPFs) have a simple porous structure; this porosity helps to lower heat conductivity but, at the same time, weakens the composite. The bio-composite material microstructure indicates poor adhesion between the gypsum composite reinforced with Date Palm Fibers (DPFs), as shown by the holes and cavities developed between the fiber's surface and the matrix. Conversely, the synthetic fibers adhere pretty well to the gypsum matrix. While the percentages added to the gypsum matrix were modest, the thermal conductivity values varied somewhat; the combination comprising both Polyester Fibers (PF) and Date Palm Fibers (DPFs) had the lowest value reported; the thermal insulating performance was enhanced by around 13.09%. Adding Date Palm Fibers (DPFs) to gypsum had a detrimental effect on compressive strength: a 24.32% loss for the case of 0.5% Date Palm Fibers (DPFs) and a 12.56% reduction for the bio-composite material, including 0.25% Date Palm Fibers (DPFs) + 0.25% Polyester Fibers (PF). Still, the bio-composite material with 0.5% Polyester Fibers (PF) had a value equivalent to the control. One can understand this decline by considering voids inside the volume. From an increase of 29.25% for the gypsum composite with 0.5% FPD to an increase of 45.07% for the gypsum composite with (0.25% Date Palm Fibers (DPF) + 0.25% Polyester Fibers (PF)), reinforced gypsum showed observed increases in flexural strength values. The orientation of the fibers in a direction opposite the applied force explains these gains. Date Palm Fibers (DPFs) added to the gypsum matrix produce high thermal

performance, so these bio-composite materials can be employed as thermal insulation to save energy consumption [259].

- 5. Tahar Ali-BOUCETTA and his colleagues (2021)** [260], assert that the effect of different treatments of Date Palm Fibers (DPFs) on the properties of mortar in both fresh and hardened states. Three Date Palm Fiber (DPF) treatments were examined: boiling water, sodium hydroxide (NaOH), and a polymer surface treatment with linseed oil. Initially, single-Date Palm Fiber (DPF) tests, including water absorption, setting time, and direct tensile strength, were conducted to identify the dominant parameters associated with each treatment. Subsequently, mortars reinforced with raw and treated Date Palm Fibers (DPFs) were prepared and evaluated using flow table tests and measurements of water-accessible porosity, flexural strength (Three-Point Bending), compressive strength, and capillary water absorption.

The results showed that Date Palm Fiber (DPF) treatments consisting of three hours of boiling, a 3% NaOH solution, and a 1.5% linseed oil-to-Date Palm Fiber (DPF) ratio provided the lowest water absorption rates, shorter setting times, and improved tensile behaviour at the Date Palm Fiber (DPF) level. For mortar composites, Date Palm Fibers (DPFs) treated with linseed oil improved workability compared with raw Date Palm Fibers (DPFs). In the hardened state, boiling and NaOH treatments increased both flexural and compressive strengths, whereas linseed oil treatment increased porosity and did not improve mechanical strength. Additionally, the incorporation of Date Palm Fibers (DPFs) reduced the capillary absorption rate of mortars compared with unreinforced mortar, with the lowest absorption coefficients observed for mortars containing linseed-oil-treated fibres [260].

- 6. Wissem GALLALA and his colleagues (2020)** [261], assert that their primary objective is to create a bio-composite material utilizing locally sourced materials: gypsum and natural fibers. The natural fibers are sourced from date palm waste in Gabes Oasis, Tunisia. Various parameters were examined to characterize these materials, including sizes, thermal characteristics, and microstructural attributes. The bio-composite materials specimens exhibited higher compressive and flexural strength tests and enhanced water absorption with the inclusion of fiber wastes. Chemical treatments employing sodium hypochlorite and matrix coating were conducted to improve composite durability by reducing surface tension and reinforcing adhesion with the gypsum matrix. The bio-composite material demonstrated its suitability as a thermal

insulation construction material by exhibiting adequate mechanical, thermal, and physical properties.

The optimal fiber percentage was 17% for a length of 20 mm. The Date Palm Fibers Waste (DPFW) composites enhance mechanical strength as the fiber proportion and length increase, unlike control gypsum without fibers. Decreased workability and inadequate fiber orientation resulted in a small reduction in a specific percentage; the same effects were seen for compressive strength. The NaOH treatment diminished the retardation effect and improved the rheological properties flexion and compression by eliminating contaminants and specific soluble components from the bio-composite material. Moreover, the epoxy coating significantly decreased water absorption. The physical attributes exhibit more hydrophilic, porous fibers with enhanced absorption capacity. The drying test on the reinforced fiber gypsum showed a gradual enhancement in flexural and compressive strength over time at varying temperatures. The enhanced adhesion between the matrix and fiber and the progression of gypsum solidification may elucidate this phenomenon. According to experimental results, the amalgamation of 17% Date Palm Fibers Waste (DPFW) yielded a composite with a density of  $\rho = 1415 \text{ kg m}^{-3}$  and a thermal conductivity of  $k = 0.52 \text{ W/m.k}$ . The findings indicate that the examined bio-composite material is appropriate as a sustainable insulating material for construction [261].

- 7. Fatma NAIIRI and his colleagues (2019)** [262], assert that the primary objective of their work is to evaluate whether gypsum mortar reinforced with doum palm fibers may be a thermal insulator in building materials. They developed many bio-composite material configurations for the mechanical and thermo-physical study involving three sizes and five weight ratios (0.5-2.5%) of doum palm fiber. They treated doum palm fibers in a 1% NaOH solution to increase their chemical degradation resistance and remove some hemicellulose and lignin to produce more homogenous fiber for enhanced fiber-matrix interfacial properties. After seven days, twenty-eight days, and one year of typical curing, the mechanical properties of the specimens were assessed.

Their findings indicate an improvement in the mechanical properties of bio-composite materials reinforced with treated fibers. They tested in his work the impact of chemical treatments on the mechanical behavior of the gypsum composite, which was assessed using three-point bending tests. They found that the NaOH treatment enhances the surface smoothness of the fiber, leading to improved fiber-matrix adhesion and a bio-composite material with superior characteristics. In addition, the experimental assessment of fiber sizes and weight ratios concerning mechanical

characteristics was conducted. In addition, using a high fiber dose (1.5%) and long fiber (Reinforcement Sieve 2-RS2) diminished flexural and compressive strength [262].

Their results also found that the doum palm fiber enhances flexural toughness, with this improvement influenced by both the fiber dose and length concurrently. The reference gypsum mortar exhibits brittle behavior when subjected to flexural strength testing. Following one year of natural aging, the composite improved performance, with flexural strength increasing from an average of 1.53-3.25 MPa. In addition, this research examines the impact of fiber quantity and length on the thermal characteristics of gypsum. The findings demonstrate a correlation between thermal conductivity and fiber concentration. The thermal conductivity diminishes with an increase in fiber concentration. In conclusion, their findings indicate that incorporating Reinforcement Sieve 2 (RS2) with a 0.2 mm mesh and a 1% weight ratio of doum palm fiber is promising for manufacturing a bio-composite gypsum mortar [262].

- 8. Aymen BRAIEK and his colleagues (2017)** [263], assert that their primary objective is to create a novel composite material that will make gypsum plaster manufactured with Date Palm Fibers (DPFs) lighter and better at holding heat, which would lower the amount of energy used by buildings. Instead of gypsum boards, Date Palm Fibers (DPFs) and gypsum boards could be utilized as thermal insulators in structures.

An experimental examination of the thermo-physical properties of gypsum reinforced with Date Palm Fibers (DPFs) was conducted by altering the mass fraction of Date Palm Fibers (DPFs) (0-20%). They used periodic methods and flash methods, which were integrated with a reverse function. Experimental findings about thermal conductivity demonstrated sufficient agreement with the theoretical models provided by Woodside and Messmer to calculate equivalent thermal conductivity [263].

- 9. Mustapha BOUMHAOUT and his colleagues (2017)** [264], assert that their primary objective, the thermomechanical characterization of bio-composite materials made of mortar and Date Palm Fiber Mesh (DPFM), is investigated in this work. The objective is to evaluate this material's mechanical performance and thermal insulating qualities for construction. The test specimens' volume percentage of Date Palm Fiber Mesh (DPFM) ranged from 0-51%. Experimental methods enabled one to ascertain the thermal characteristics of these materials:

conductivity, diffusivity, capacity, and effusivity. Moreover, carefully evaluated the compressive and flexural strengths of the specimens.

This bio-composite material shows good thermomechanical performance according to the results. By lowering their heat conductivity by up to 70%, Date Palm Fiber Mesh (DPFM) inclusion in mortar boosts its insulating ability. Its heat-damping characteristics also improve as its thermal effusivity is lowered by up to 56% and its thermal diffusivity by up to 52%. Moreover, Date Palm Fiber Mesh (DPFM) reduces the density of the mortar by up to 39%, therefore lightening it. Conversely, the mechanical strength of the bio-composite material suffers in the increasing Date Palm Fiber Mesh (DPFM) concentration. Still, the compressive strength values are within a reasonable range even with high Date Palm Fiber Mesh (DPFM) concentration. Furthermore, fiber addition helps the Mortar-Date Palm Fiber Mesh (DPFM) specimens to be more ductile. A thermomechanical diagram developed with appropriate testing findings defined the examined Mortar-Date Palm Fiber Mesh (DPFM) bio-composite materials. Based on the International Union of Laboratories and Experts in Construction Materials (RILEM) functional classification of lightweight concrete, this graphic illustrates that the bio-composites might be essentially regarded as structural and insulating material [264].

**10.Ali Mohamed ALGARNY and his colleagues (2016)** [265], assert that their primary objective in this study is to investigate the feasibility of manufacturing indoor bricks using a mixture of palm tree sawdust and gypsum powder. They used different weights to amalgamate the proportions of the admixtures. They are finding that incorporating palm tree sawdust with gypsum powder significantly improves the characteristics of the resultant admixture in several aspects. In addition, their results showed that the specimens' increased proportions of sawdust demonstrated elevated compressive strength values, with the maximum compressive strength achieved at a 6% sawdust-mixing ratio, surpassing the control value by 150%. Their results indicate the capacity to enhance the quality of gypsum cubes for application in interior construction. In addition, the 6% palm tree sawdust-mixing ratio demonstrated the most significant enhancement in all tests, making it the optimal ratio among the evaluated specimens. As a conclusion, their finding that the enhancement of gypsum's properties as a construction material by the incorporation of palm tree waste, with the affordability of both gypsum and these byproducts, makes the manufacturing of cost-effective building materials, primarily consisting of gypsum and palm tree residues, more viable.

**11. Mourad CHIKHI and his colleagues (2013)** [266], assert that their primary objective for this project was to create a fresh bio-composite material fit for usage as building thermal insulation. The aim was to experimentally explore the effects on thermal conductivity, water absorption, and mechanical characteristics of gypsum-based materials of Date Palm Fibers (DPFs) (*Phoenix Dactylifera* L. from Biskra oasis in Algeria). Two distinct Date Palm Fiber (DPF) sizes and many Date Palm Fiber (DPF) filler contents were developed in bio-composite materials.

As a result, the compressive and flexural strengths of the completely bio-composite material specimens, which rely on the gypsum matrix, diminish with an increase in fiber content. The DPF3-based gypsum composites exhibited superior compressive and flexural strength compared to those containing larger particles (DPF6). Additionally, the Gypsum/Date Palm Fiber (DPF) composites have superior mechanical properties (Compressive and Flexural Strength) relative to other materials documented in the literature. Composites of HG/Date Palm Fiber (DPF) exhibit enhanced compressive strength with 5% DPF3 and 1.2% DPF6. Conversely, 3% Date Palm Fiber (DPF) produces superior flexural strength for both fibers. Furthermore, the investigation of thermos-physical properties indicates that an increase in Date Palm Fiber (DPF) concentration within the gypsum matrix diminishes the heat conductivity and density of the composites. The thermal properties are more significantly affected by Date Palm Fiber (DPF) concentration than by Date Palm Fiber (DPF) size. Experimental results showed that including 10% Date Palm Fiber (DPF) produced a composite with a density of  $\rho = 753 \text{ kg m}^{-3}$  and thermal conductivity of  $k = 0.15 \text{ W m}^{-1} \text{ K}^{-1}$ . These values include the thermal conductivity and density ranges of many insulating materials. At 10% Date Palm Fiber (DPF) loading, the minimum values of thermal conductivity and density of HG/Date Palm Fiber (DPF) are achieved. Consequently, it is essential to recognize that a gypsum matrix containing 5% Date Palm Fiber (DPF) yields a composite with superior mechanical and thermal properties. They contend that the low cost and ample availability of noncommercial Date Palm Fiber (DPF) waste provide a substantial opportunity for a large initiative in sustainable materials for thermal insulation [266].

Ultimately, the studies [9, 257-266], described above highlight the potential of using palm fiber-reinforced gypsum matrices to produce bio-composite materials. Despite their varying mechanical and physical properties, these bio-composite materials improve the thermal insulation properties of buildings. This is related to the size-to-weight ratio of palm fibers used in the reinforcement. Furthermore, these

bio-composite materials are generally high-performance and environmentally friendly thermal insulation.

## **Conclusion**

In conclusion, this second chapter has provided a comprehensive introduction to the utilization of Date Palm Fibers (DPFs) and gypsum in the creation of bio-composite materials. The properties, types, and extraction methods of both Date Palm Fibers (DPFs) and gypsum have been explored in detail, highlighting their relevance as sustainable and locally sourced materials for bio-composites. Date Palm Fibers (DPFs), with their abundance and favorable mechanical properties, present a strong case for use as a reinforcement material in composite formulations. Similarly, gypsum, with its versatility and widespread applications, proves to be an effective matrix material in bio-composite systems.

The discussion in this second chapter has also addressed the advantages and disadvantages of using Date Palm Fibers (DPFs) and gypsum in bio-composites. While Date Palm Fibers (DPFs) offer benefits such as biodegradability, cost-effectiveness, and environmental sustainability, challenges related to their processing and durability must be carefully considered. Similarly, gypsum's ease of extraction and suitability for construction applications make it an attractive option. However, its limitations in terms of mechanical properties and performance under specific conditions require further investigation.

The review in this second chapter of existing literature on bio-composite materials composed of Date Palm Fibers (DPFs) and gypsum has provided valuable insights into previous research efforts and identified gaps in knowledge. This sets the stage for future research that will focus on improving the performance, processing techniques, and applications of these bio-composite materials. Ultimately, this second chapter lays the groundwork for understanding the potential of Date Palm Fibers (DPFs) and gypsum as key components in sustainable bio-composites, offering a solid foundation for the subsequent chapters that will delve into their specific applications, manufacturing techniques, and performance optimization. The integration of these materials in bio-composite formulations holds significant promise for advancing eco-friendly solutions in various industries, particularly in construction and manufacturing.



## Conclusion

In conclusion, the first part of this dissertation has provided a comprehensive exploration of composite materials, with a particular focus on bio-composites, Date Palm Fibers (DPFs), and gypsum.

The first chapter introduced the concept of composite materials, delving into their classification, properties, and the role of natural fibers, especially bio-composites, in advancing sustainable material science. It emphasized the environmental benefits, biodegradability, and mechanical properties of natural fibers as reinforcement agents in bio-composites, while also addressing the challenges that still need to be overcome, such as cost and durability. The manufacturing methods of bio-composites were thoroughly discussed, providing a foundation for understanding their fabrication processes and potential applications across industries.

The second chapter shifted the focus to the specific materials of Date Palm Fibers (DPFs) and gypsum, exploring their properties, extraction methods, and relevance in the context of bio-composites. Date Palm Fibers (DPFs), with their abundance and favorable mechanical properties, emerged as a promising reinforcement material, while gypsum proved to be an effective matrix material. However, both materials presented unique challenges, including processing issues and performance limitations, which were critically examined. The second chapter also highlighted the importance of further research to optimize these materials' properties and broaden their applications in the construction and manufacturing sectors.

Ultimately, the first part provides a solid foundation for the dissertation's subsequent exploration of bio-composites, paving the way for future research aimed at overcoming the existing challenges and expanding the practical use of sustainable materials in engineering. The integration of Date Palm Fibers (DPFs) and gypsum in bio-composites offers promising eco-friendly solutions for industries seeking to reduce their environmental footprint, making it a valuable area for continued study and innovation.

# **Part 2**

## **Experimental Study**

## Introduction

In this second part of the thesis, we present a comprehensive examination of the bio-composite materials, focusing on their material properties and experimental results.

The third chapter introduces the materials and methods employed to evaluate the bio-composite under study. It provides an in-depth description of the raw materials, specifically Date Palm Fiber (DPFs) as the reinforcement and gypsum as the matrix, along with the methodology of manufacturing bio-composite materials used for testing their mechanical, physical, thermal, chemical, and morphological properties. These tests are critical to understanding the bio-composite material's performance under different conditions and its potential for practical applications.

The fourth chapter builds upon the methodologies outlined in the previous section by presenting and analyzing the experimental results. These findings include detailed measurements of the mechanical, physical, and thermal properties, along with the chemical composition and morphological characteristics of the bio-composite materials. The results offer a clear picture of the bio-composite's capabilities and limitations, contributing valuable insights into its potential use in various industrial and sustainable applications.

Ultimately, the second part lays the foundation for evaluating the bio-composite material's performance and its future applicability in diverse fields.

The second part can be summarized by the hierarchy, as shown in [Figure 03](#).

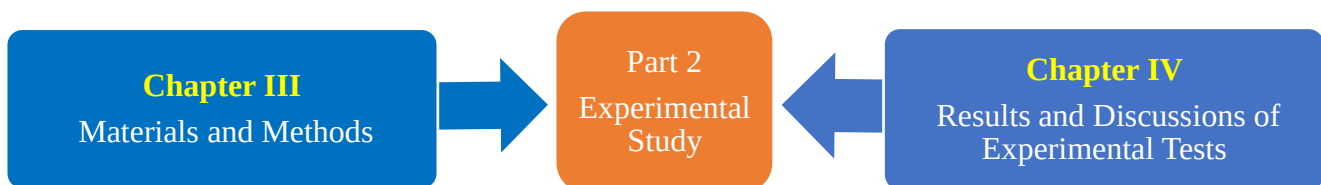


**Figure 03. Graphical Abstract of the Second Part of the Thesis.**

# Chapter III

## Materials and Methods

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## Introduction

In this third chapter, the materials and methods utilized to evaluate the properties of the bio-composite materials under study are presented in detail. The first section provides an overview of the raw materials, focusing on the specific components that contribute to the formation of the bio-composite, including Date Palm Fiber (DPF) as reinforcement and gypsum as matrix. Each material is chosen for its unique properties and its potential synergistic effect when combined into a bio-composite structure.

Following the Date Palm Fiber (DPF) and gypsum description and the manufacturing of the bio-composite, the methodology section outlines a comprehensive set of tests designed to assess the mechanical, physical, thermal, chemical, and morphological properties of the bio-composite materials. These tests are integral to understand the bio-composite materials' behavior under different conditions and contribute to evaluating their potential applications in various fields.

The mechanical tests, which include flexural (Three-Points) testing, compressive strength testing, Brinell hardness testing, and Charpy impact strength testing, offer information regarding the bio-composite material's ability to withstand external forces and its durability. Additionally, a series of physical tests, such as bulk density, water absorption, thickness swelling, and moisture content, is conducted to determine the bio-composite material's stability and resistance to environmental factors. Thermal testing, specifically the thermal conductivity test, helps assess the material's ability to resist heat transfer, an essential characteristic for potential insulation applications. Chemical tests, including Energy Dispersive X-Ray Spectroscopy (EDS) and Fourier Transform Infrared Spectroscopy (FT-IR), offer detailed insights into the elemental composition and molecular structure of the bio-composite materials, ensuring their suitability for various industrial uses. Finally, the morphological analysis through Scanning Electron Microscopy (SEM) provides a visual representation of the bio-composite material's surface structure, enabling a more profound understanding of the interaction between the fibers and the matrix.

Ultimately, this third chapter offers a comprehensive assessment of the bio-composite material, ensuring that it meets the requirements for practical and sustainable applications.

### III.1. Materials

Preparing materials requires adherence to the following points in the specified order:

### III.1.1. Date Palm Fiber-Reinforcement

As shown in [Figure III.01.a](#), the palm oases located in the Tolga-Biskra region of southeastern Algeria, which contain numerous palm trees (As Shown in [Figure III.01.b](#)), produce vast quantities of date palm waste (As Shown in [Figure III.01.c](#)). These wastes will be recycled to extract Date Palm Fibers (DPFs) of various sizes, which will be used as reinforcement for bio-composite materials.



**Figure III.01. Date Palm: a) Oasis, b) Tree, and c) Waste.**

The Date Palm Fiber (DPFs) preparation steps are as follows:

- 1. Cutting the Rachis Stalks:** The palm leaves are brought, and then the leaflets are removed to reveal the palm petioles connected to the Rachis stalk. According to a study conducted by Tarek Djoudi et al [65], the Rachis stalk of the palm tree is located after the palm petioles, which are between 30-150 cm from the beginning of the palm leaves, as shown in [Figure III.02](#).

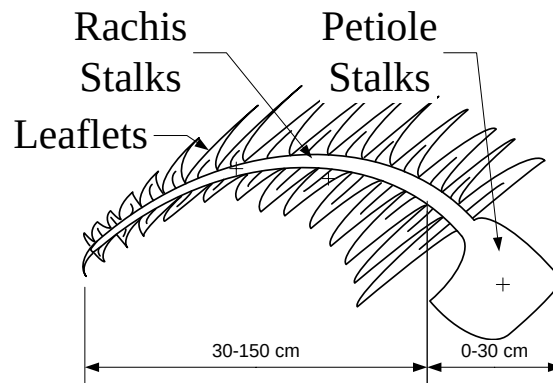


**Figure III.02. Date Palm Leaves Sections.**

After separating the components of the palm leaves, the palm leaflets is collected as shown in [Figure III.03.a](#), the palm petioles as shown in [Figure III.03.b](#), and the Rachis stalks as shown in [Figure III.03.c](#), each separately.



**Figure III.03. Date Palm Leaves Components:**  
**a) Leaflets, b) Petiole, and c) Rachis Stalks.**

**2. Grinding the Rachis Stalks:** To ensure the palm fibers are not damaged, the Rachis stalks are ground according to the following two steps:

- a. **Step 1:** Using a Zakandra mill as shown in [Figure III.04.a](#) [267], located at the Technical Institute for the Development of Saharan Agronomy in the Ain Ben Naoui, El Hadjeb Municipality, Biskra Province, we obtain large fibers with 12 cm, as shown in [Figure III.04.b](#);
- b. **Step 2:** Using a small-sized mill as shown in [Figure III.04.c](#), located in the technological hall of the Faculty of Science and Technology, Mohamed Khider University of Biskra, Algeria. The grinder is characterized by its handcrafted construction. The fibers obtained from the first



step are placed in the second grinder, which grinds them to produce fibers smaller than those obtained from the first grinder, as shown in [Figure III.04.d](#).



**Figure III.04. Steps of Grinding Date Palm Rachis Stalks.**

- 3. Washing the Fibers:** According to research conducted by Tahar Ali-Boucetta et al [260], to wash the Date Palm Rachis Fibers (DPFRs) as shown in [Figure III.05](#), they must be soaked in boiling water for a full three hours. After the boiling process is complete, the fibers are filtered and rinsed with water to remove organic matter that would hinder their adhesion to the matrix when used as a reinforcement for bio-composites.





**Figure III.05. Washing Date Palm Rachis Fibers.**

- 4. Drying of the Rachis Fibers:** Also, according to the research conducted by Tahar Ali-Boucetta et al. [260], to dry the Date Palm Rachis Fibers (DPFRs), they should be placed at a temperature of 110°C for 24 hours, in order to reduce the water absorption of the fibers, completely remove moisture from them, and increase their Young's modulus. Therefore, the washed Date Palm Rachis Fibers (DPFRs) were placed in a Nabertherm drying oven located in the Department of Mechanical Engineering, University of Mohamed Khider, Biskra [268], as shown in [Figure III.06.a and b](#), at a temperature of 110°C for 24 hours, as shown in [Figure III.06.c](#).



**Figure III.06. Drying Date Palm Rachis Fibers.**

- 5. Sieving of the Rachis Fibers:** Using a Retsch AS 200 [269], as shown in [Figure III.07](#) vibrating screen and in accordance with international standards AFNOR D253, D255, D256, D259, D261, D262, and D265 [270], the dried Date Palm Rachis Fibers (DPFRs) were screened to obtain different sizes (0.080, 0.125, 0.160, 0.315, 0.500, 0.630, and 1.250 mm) of reinforcement Date Palm Rachis Fibers (DPFRs).



**Figure III.07. Sieving Date Palm Rachis Fibers.**

**6. Final fibers:** After sieving, Date Palm Rachis Fibers (DPFRs) of sizes 0.080, 0.315, and 0.500 mm were retained and named smaller, medium, and larger Date Palm Rachis Fibers (DPFRs), with symbolizations of s: I, II, and III, respectively as shown in [Figure III.08](#).



**Figure III.08. Sizes of Date Palm Rachis Fibers:**

**a) Smaller Fiber (s: I)-Size: 0.080 mm, b) Medium Fiber (s: II)-Size: 0.315 mm, and c) Larger Fiber (s: III)-Size: 0.500 mm.**

Ultimately, Date Palm Rachis Fibers (DPFRs) preparation is a fundamental step in the production of bio-composites, as essential steps are followed to ensure high-quality fibers, thus ensuring superior bio-composite properties.

### III.1.2. Gypsum-Matrix

Industrial gypsum for the molding process, produced by Cosider, Lafarge Plasters Algeria (COLPA) [271], was used as the matrix for the bio-composite materials, as shown in [Figure III.09](#). The gypsum used as a matrix is extracted from the quarries of Bouira, Algeria, located in northeastern Algeria.

The Cosider, Lafarge Plasters Algeria (COLPA) Manhargyps casting gypsum is a quick-setting gypsum designed explicitly for creating gypsum parts, such as tiles, cornices, rosettes, and quick moldings. The work is broken up into phases: mixing takes 1 to 2 minutes, pouring takes 3 to 4 minutes, and demolding happens 15 minutes after pouring.

The curing can be done by hand or with a small quantity of mechanical agitation. The time it takes to stir, the purity of the water, and the mixing ratio all affect how long it takes to pour, how strong it is, and how porous it is [271].

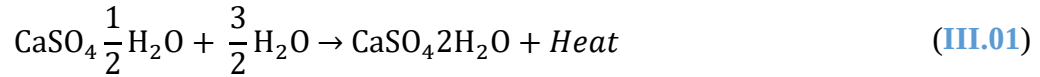


**Figure III.09. Matrix-Gypsum COLPA [271].**

This gypsum can be easily molded by pouring it into a mold when mixed with a specific amount of water. The rapport of water to gypsum used was 0.6. In accordance with the international standard ASTM C59/C59M-00 (2020) [272], the gypsum powder was produced, which went through several stages, starting with crushing and grinding extracts from underground mines and quarries of natural rocks. At the end of the production stage, it was treated at 150°C to obtain gypsum powder.

This specification ASTM C59/C59M-00 (2020) [272], pertains to casting and molding gypsum materials primarily composed of calcined gypsum. The chemical composition of any gypsum material must satisfy the specified purity standard. Equal quantities of material should be extracted from both the exterior and

the core of each packet. Specimens will be evaluated for setting time, compressive strength, and fineness. According to Equation (III.01), gypsum is dehydrated to form calcium sulfate, which contains dehydrated calcium sulfate hemihydrate, upon addition of water.



Ultimately, gypsum is a highly efficient thermal insulation matrix and possesses diverse properties that make it a preferred choice for the manufacture of bio-composites. Therefore, it is the ideal choice for the formulation of bio-composites.

### III.1.3. Bio-composite Material

The twelve different types of bio-composite materials were manufactured for comparison with the gypsum specimen. The bio-composites consist of a gypsum matrix reinforced with Date Palm Fibers (DPFs). Each bio-composite consists of a specific size and mass ratio of Date Palm Fibers (DPFs). Each bio-composite is reinforced with Date Palm Fibers (DPFs) at 05, 10, 15, and 20% for each size of Date Palm Fibers (DPFs), 0.080, 0.315, and 0.500 mm, yielding twelve bio-composite specimens.

The bio-composites were coded GF.s.m, where each symbol represents:

- **G:** Gypsum;
- **F:** Date Palm Fiber (DPF);
- **s:** The Palm Fiber Size is Symbolized by I, II, and III for the Smallest, Medium, and Largest Fibers, which are for the Sizes 0.080, 0.315, and 0.500 mm, Respectively;
- **m:** The Notation Represents the Mass Ratio of Date Palm Fiber (DPF) added to Gypsum relative to the Total Mass of the Bio-Composite Material, and is 05, 10, 15, and 20% each Time.

When manufacturing gypsum or bio-composite specimens, the water-to-gypsum mixing ratio was maintained at 0.6 to ensure consistency of the mixture, regardless of changes in the size or mass ratio of the palm fibers.

The bio-composite or gypsum specimens were manufactured by casting them into prefabricated wooden molds, which allowed us to extract laboratory specimens according to the dimensions specified in international standards and in accordance with the desired test.

Laboratory tests revealed that the preparation of a gypsum specimen measuring 40 × 40 × 160 mm required 340 grams of gypsum and 204 mL of water. Accordingly, specimens of bio-composites and gypsum were manufactured in accordance with the test to be performed. Before preparing bio-composite

specimens, it is essential to weigh the gypsum and palm fibers. Equation (III.02) must be employed to guarantee compliance with designated Date Palm Fibers (DPFs) mass ratios in the bio-composite.

$$W_f = \frac{m_f}{m_{b-c-m}} \times 100 \quad (\text{III.02})$$

Where:

- **W<sub>f</sub>**: The Mass Ratio of Date Palm Fiber (DPF) [%];
- **m<sub>f</sub>**: The Mass of Date Palm Fiber (DPF) [g];
- **m<sub>b-c-m</sub>**: The Mass of Bio-Composite Material [g].

As shown in Figure III.10, after weighing the gypsum and Date Palm Fibers (DPFs) for the bio-composite material, water must initially be added to the pot.

Subsequently, pour the gypsum over the water and allow it to sit for 1 minute to enable the water to permeate the gypsum powder. The Date Palm Fibers (DPFs) are subsequently positioned on the gypsum powder.

The ingredients are thereafter combined using an electric mixer for 90 seconds. The mold is then lubricated with synthetic or food-grade oil to assist the extraction of the specimens from the mold. The amalgamation is thereafter positioned in the mold allocated for the test specimens.

Thereafter, the components are correctly aligned, as the mixture may occasionally be semi-dry due to the rapid absorption of water by the palm fibers. The specimens are allowed to rest for four hours prior to removal from the mold to avert collapsing or fracturing. The specimens are subsequently maintained at room temperature for 28 days prior to testing.



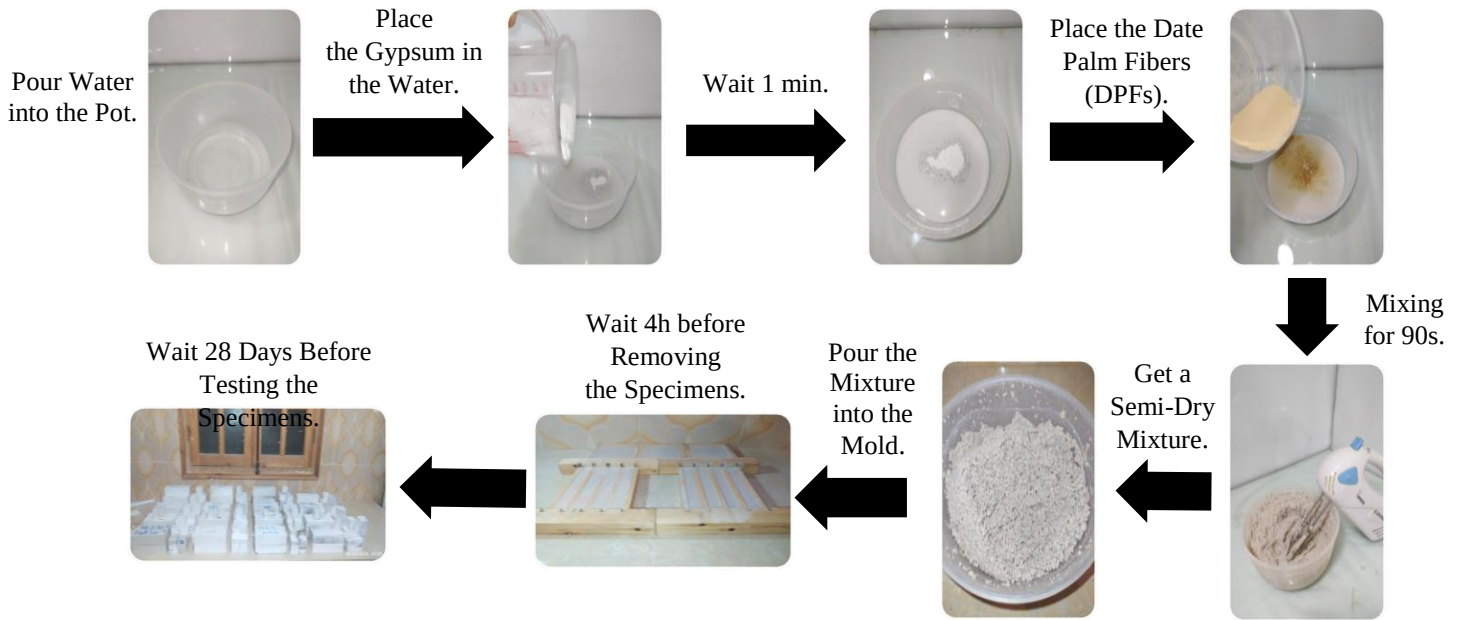


**Figure III.10. Experimental Protocol for Making Bio-composite Material Specimens.**

Ultimately, preparing bio-composites requires several important steps, from preparing the Date Palm Fibers (DPFs) used as reinforcement to identifying the basic properties of the gypsum used as the matrix. This ensures the production of highly efficient composite materials with diverse properties, making them versatile.

To conclude, the materials preparation section, starting with palm fibers as reinforcement and gypsum as matrix, and leading to the manufacture of bio-composite materials, is considered a crucial step to ensure the diversity of the latter's applications, based on the hope of combining the properties of reinforcement and matrix in a single material.

## III.2. Method

The experimental method for characterizing bio-composites and gypsum depends on two main aspects:

### III.2.1. Experimental Tests

Several experimental tests will be performed on bio-composite materials (GF.s.m) made of gypsum and Date Palm Fibers (DPFs), and these will be compared to a typical gypsum specimen. The made of these bio-composites (GF.s.m) by changing the sizes and mass ratios of the Date Palm Fibers (DPFs) in the mix for each size. [Figure III.11](#). Shows that the experimental testing was split into four primary groups:

mechanical tests, physical tests, thermal tests, chemical tests and morphological tests. Each group has its tests that were done according to international standards to measure distinct material attributes.

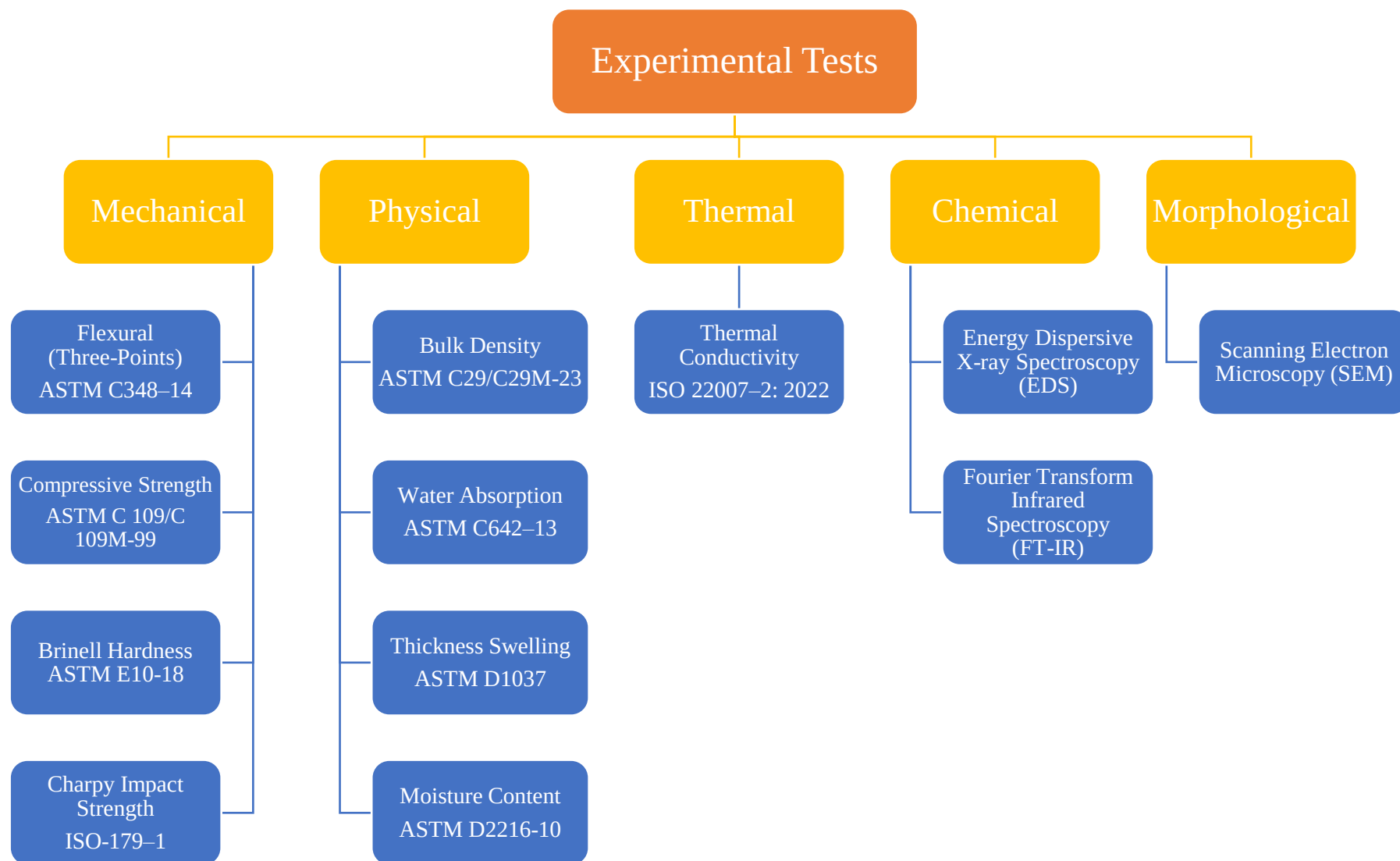


**Figure III.11. Experimental Tests.**



Below is a full explanation of these test groups:

**1. Mechanical Tests:** Mechanical tests assess the strength and structural integrity of materials.

Moreover, it is as follows:

- a. **Flexural (3-Points) Test (According to ASTM C348–14):** This test evaluates the material's capacity to withstand flexural under stress [273];
- b. **Compressive Strength Test (According to ASTM C109/C 109M-20):** This test checks to see how well the material can hold up under pressure without breaking [274];
- c. **Brinell Hardness Test (According to ASTM E10-18):** To test the hardness of a material, a hard sphere is pressed into its surface, and the indentation is measured [275];
- d. **Charpy Impact Strength Test (According to ISO-179-1:2023):** This test checks how well the material can handle rapid shocks or impacts [276].

**2. Physical Tests:** Physical tests show how the material reacts to different environmental conditions. Moreover, it is as follows:

- a. **Bulk Density Test (According to ASTM C29/C29M-23):** This test checks how dense the material is, which is vital for determining its strength and durability [277];
- b. **Water Absorption Test (According to ASTM C642-13):** This test measures how much water the material can hold, which affects how long it lasts and how well it works [278];
- c. **Thickness Swelling Test (According to ASTM D1037):** This test checks how much the material expands or swells when it gets wet, which is essential for figuring out how stable it is in terms of size [279];
- d. **Moisture Content Test (According to ASTM D2216-10):** This test checks how much moisture is in the material, which can change how it behaves physically [280].

**3. Thermal Tests:** Thermal testing looks at how the material reacts when the temperature varies.

Moreover, it is as follows:

- a. **Thermal Conductivity Test (According to ISO 22007–2: 2022):** This test checks how well the material can carry heat. It is a key feature for users where thermal insulation is needed [281].

#### 4. **Chemical Tests:** Chemical tests look at the material's composition and chemical properties.

Moreover, it is as follows:

- a. **Energy Dispersive X-ray Spectroscopy (EDS) Test:** This test examines the material's elemental makeup by analyzing X-rays emitted from the specimen when it is bombarded with an intense electron beam [282];
- b. **Fourier Transform Infrared Spectroscopy (FT-IR) Test:** This test analyzes how the material absorbs infrared radiation at different wavelengths, which helps determine the molecules that make it up [283].

#### 5. **Morphological Test:** Microstructural tests look at the material's morphological properties.

Moreover, it is as follows:

- a. **Scanning Electron Microscopy (SEM) Test:** This test studies the structure of a material at the microscopic level. This test will be used on selected specimens that have the minimum, average, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ , Respectively) among bio-composite materials (GF.s.m) to learn more about the microstructure of the material and to correlate it with the results of various tests [284].

Ultimately, as the above tests have shown, any change in Date Palm Fibers (DPFs) size and mass ratio is likely to impact the mechanical, physical, thermal, chemical, and morphological properties of bio-composite materials (GF.s.m). The comparisons that will be made during the tests will help us understand how Date Palm Fibers (DPFs) size and mass ratio affect bio-composite materials (GF.s.m) compared to pure gypsum. These tests can also inform the design and application of bio-composite materials (GF.s.m) in a variety of applications.

### III.2.2. Mechanical Tests

The mechanical tests that will be conducted on the bio-composites (GF.s.m) and will be compared with gypsum are highlighted in the following tests:

#### III.2.2.1. Flexural (3-Point) Test

Three-point flexural testing was performed on bio-composite materials (GF.s.m) and gypsum to obtain results, according to ASTM C348-14 [273]. It is essential to follow a precise and systematic protocol. The required five prismatic specimens measuring  $40 \times 40 \times 160 \text{ mm}^3$  in size, as shown in [Figure III.12.a](#).

The Instron 5969 mechanical universal testing machine was utilized to apply a three-point flexural strain to the specimens, as shown in [Figure III.12.b](#) [285]. Furthermore, to ensure even load distribution throughout the test, the strain rate was set at 1 mm/min, as specified in ASTM C348-14 [273].

Furthermore, the specimens are placed on the machine's support span so that they align with the midpoint of two spots. The ASTM C348-14 rules specify that these supports should be spaced a certain distance apart, typically 100 mm (3.94 in), as shown in [Figure III.12.c](#) [273]. A loading support is used, and the load is placed at the center of the top of the specimen. The load should be added in small increments until the specimen breaks, which typically occurs when it exhibits visible cracks or breaks under the load.

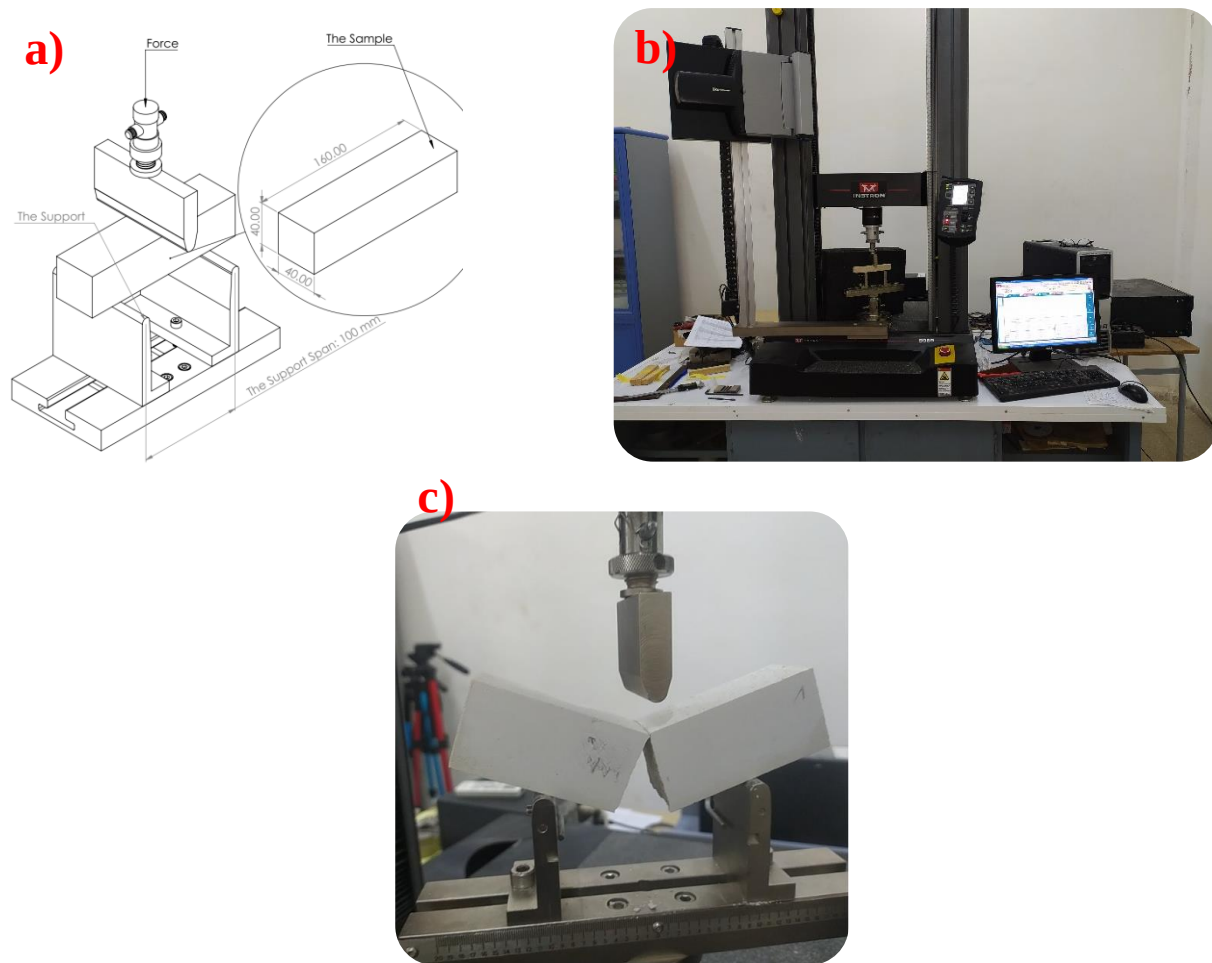


**Figure III.12. Flexural Test: a) Size-Position Specimen in Support Span, b) The Prismatic Specimen In Instron 5969, and c) The Specimen after The Break.**

The following Equations ([III.03](#), [04](#), and [05](#)) are usually used to figure out the flexural properties (Flexural Modulus, Flexural Strength, and Strain at Break) from the test results:

$$\sigma_{3P} = \frac{3 \times F \times L}{2 \times b \times h^2} \quad (\text{III.03})$$

$$E_{3P} = \frac{F}{\delta} \times \frac{L^3}{4 \times b \times h^3} \quad (\text{III.04})$$

$$\epsilon_{3P} = \frac{6 \times \delta \times h}{L^2} \quad (\text{III.05})$$

Where:

- **$\sigma_{3P}$** : Flexural Strength [MPa];
- **$E_{3P}$** : Flexural Modulus [MPa];
- **$\epsilon_{3P}$** : Strain at Break [mm/mm];
- **F**: The Maximum Load [N];
- **$\delta$** : Maximum Deflection/Displacement at the Center [mm];
- **L**: The Distance Between the Two Supports Span [mm];
- **h**: Height of the Specimen [mm];
- **b**: Width of the Specimen [mm].

In addition, the Instron 5969 machine will measure specimen load and deflection during the test [285]. The data is needed to generate the load-displacement curve. After that, the specimen failure happens when it breaks or cracks significantly. The Instron 5969 machine measures the maximum load (F) and deflection ( $\delta$ ) at the specimen center at the point of failure [285].

Furthermore, the Instron 5969 calculates the values of flexural strength, modulus, and strain at break ( $\sigma_{3P}$ ,  $E_{3P}$ , and  $\epsilon_{3P}$  Respectively), based on the maximum load (F), maximum deflection/displacement ( $\delta$ ), span length (L), and specimen dimensions (h, and b) [285]. These values are determined using the Equations (III.03, 04, and 05) for each specimen. Furthermore, all specimens (Five per Group of Bio-composite Material (GF.s.m) and Gypsum) were tested, and the mean and standard deviations of flexural strength, flexural modulus, and strain at break were calculated.

Ultimately, after determining the flexural properties of bio-composites (GF.s.m) and gypsum, the findings will be compared to assess how bio-composites compare to gypsum in terms of flexural strength,

modulus of elasticity, and strain at break. By following these steps, we will obtain precise data that will enable us to compare the flexural properties of different materials and their reactions to flexural forces.

#### III.2.2.2. Compressive Strength Test

Compressive strength testing was performed on bio-composite materials (GF.s.m) and gypsum to obtain results, in accordance with ASTM C109/C109M-20 [274]. Cubic specimens measuring 50 mm x 50 mm x 50 mm were prepared, as shown in [Figure III.13.a](#). The specimens were properly shaped by casting them into wooden molds, and after removal, they were dried for the recommended period (Approximately 28 Days for Cementitious Materials) before testing. The proper amounts of each component were mixed for each type of specimen (Bio-composite Materials (GF.s.m) and Gypsum). The combination remained the same throughout the test, with a water-to-gypsum ratio of 0.6, to ensure a smooth process.

Furthermore, to test the specimens, an Instron 5969 universal mechanical testing machine was used to apply a compressive load to each specimen, as shown in [Figure III.13.b](#) [285]. The strain rate was set to 1 mm/min, as recommended in ASTM C109/C109M-20 [274]. This loading was performed slowly and evenly, facilitating the determination of the material's failure threshold. During testing, each cubic specimen was placed in the testing machine so that the pressure was applied to the top surface of the specimen, as shown in [Figure III.13.c](#). During testing, proper alignment of the specimens was ensured to prevent eccentric loading.

Additionally, the Instron 5969 gently squeezes the material until it breaks, and the specimen fractures when loaded, revealing its maximum compressive load [285]. The device continually monitors the Newton-measured failure load. This allows failure point deformation verification.

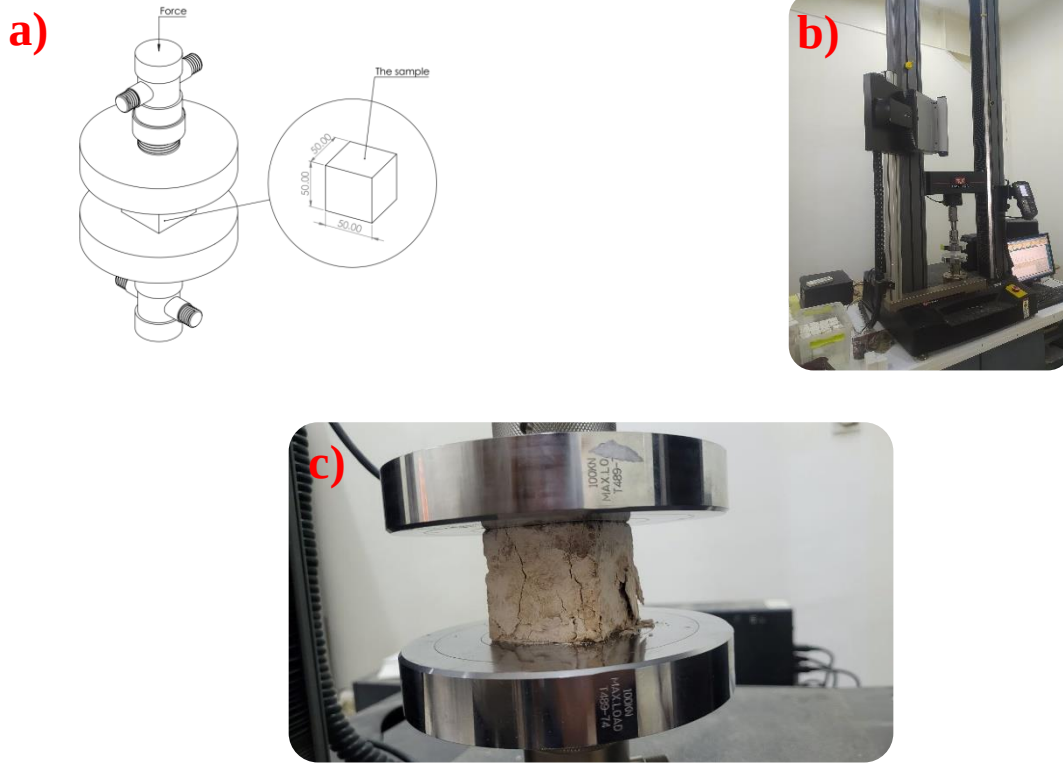


**Figure III.13. Compressive Strength Test:**  
**a) Size-Position Specimen in Support Pressure,**  
**b) The Cubic Specimen in Instron 5969, and c) The Specimen after the Break.**

The following Equations (III.06, and III.07) is usually used to figure out the compressive strength and compressive modulus from the test results:

$$f_m = \frac{P_{TML}}{A} \quad (\text{III.06})$$

$$E_m = \frac{f_m}{\epsilon_m} \quad (\text{III.07})$$

Where:

- $f_m$ : Compressive Strength [MPa or N/mm<sup>2</sup>];
- $P_{TML}$ : Maximum Load/Force at Failure [N];
- $A$ : Cross-Sectional Area of the Specimen [mm<sup>2</sup>];
- $E_m$ : Compressive Modulus [MPa];
- $\epsilon_m$ : Compressive Strain at Break [mm/mm].

Furthermore, to find the compressive strength ( $f_m$ ) of each specimen, divide the maximum load ( $P_{TML}$ ) by the cross-sectional area ( $A$ ), as shown in Equation (III.06). The compressive modulus ( $E_m$ ) of each specimen is determined by dividing compressive strength ( $f_m$ ) by the strain at break at compressive ( $\epsilon_m$ ), as shown in Equation (III.07). The five specimens of each material (Bio-composite Materials (GF.s.m) and Gypsum) should be tested for compressive strength. To find the average compressive strength, they add up the compressive strengths of all five specimens and divide by five. In addition, the standard deviation indicates the degree of variation in the compressive strength data for each material.

Ultimately, after getting the average compressive strengths and standard deviations for both the bio-composite material (GF.s.m) and Gypsum, they can compare how well they hold up under compression. Higher compressive strength values indicate that a material can better withstand applied stresses, which is crucial for structural applications.

### III.2.2.3. Brinell Hardness Test

Brinell hardness was tested on bio-composite materials (GF.s.m) and gypsum to determine the surface hardness of these materials, in accordance with ASTM E10-18 [286]. The created specimens measure 20 mm × 40 mm × 40 mm, as shown in Figure III.14.a. Before testing, the specimens were carefully cut, polished, and cleaned to ensure accurate measurements and a high-quality surface. The Brinell hardness test was done with an INNOVATEST VERZUS 750CCD hardness tester, as shown in Figure III.14.b [287]. The tester uses a 2.5 mm diameter steel ball as an indenter, as shown in Figure III.14.c. A load of 6.2 kgf or 60.80123 N is applied to the specimen. The load is applied for 30 seconds, which is sufficient for the ball to penetrate the material and leave a visible deformation. This is accomplished by placing the specimen on the device's platform with the drill pressing down from above.

Furthermore, after removing the steel ball, the indentation it had made was checked. A Brinell microscope with a resolution of 2.5/0.05 (Steel ball diameter/Zooming), type 160/0, is used to accurately measure the indentation's diameter in two perpendicular directions, typically the horizontal and vertical directions ( $d_1$ , and  $d_2$ ), as shown in Figures III.14.a, and d. The indentation measurements are made in two directions perpendicular to each other, with the largest diameter being taken in both directions ( $d_1$ , and  $d_2$ ), as shown in Figure III.14.a.

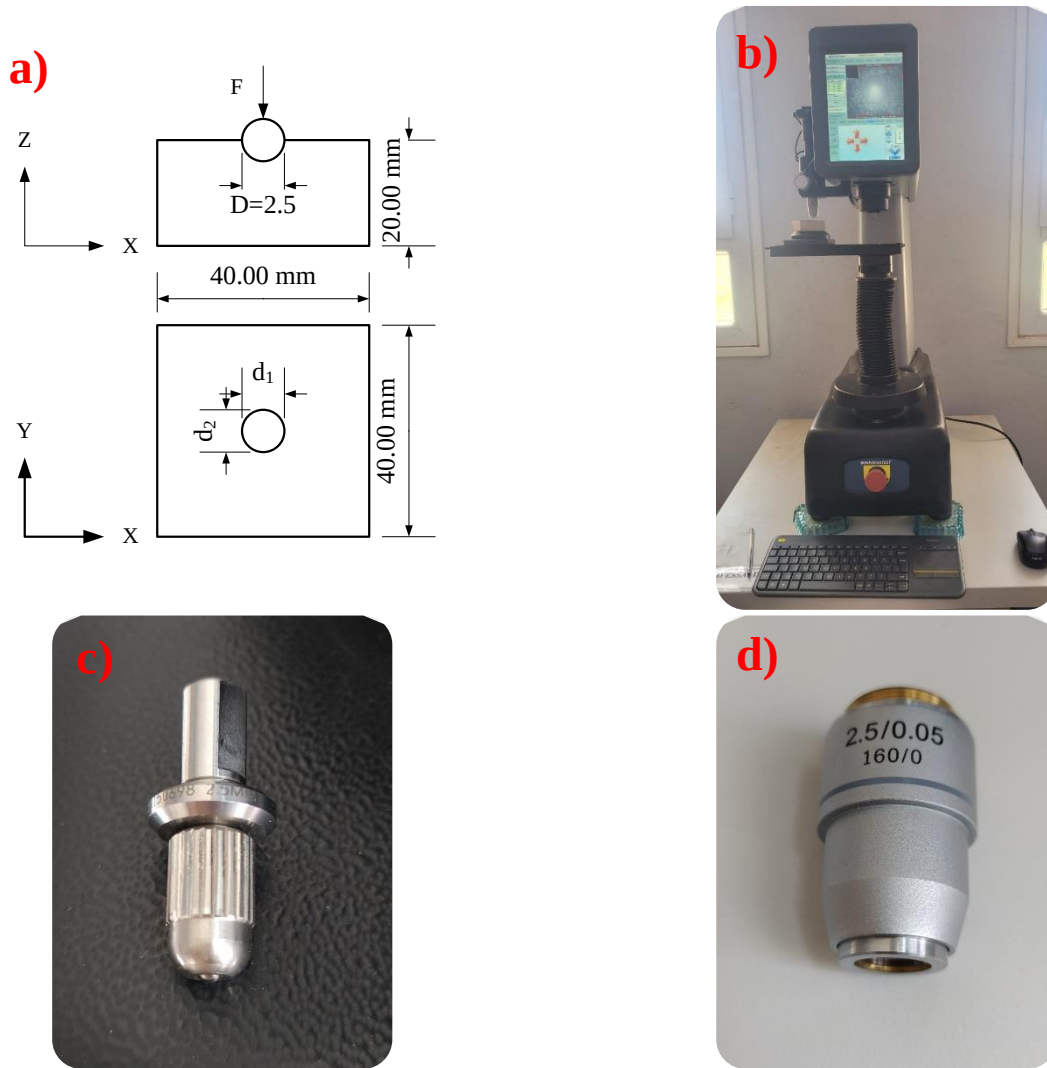


**Figure III.14. Brinell Hardness Test: a) Indentation of the Steel Ball in the Specimen, b) The Specimen in the INNOVATEST VERZUS 750CCD Device, c) The Steel Ball as an Indenter, and d) The Brinell Microscope.**

The following Equation (III.08) is used to get the Brinell Hardness Number from the test results:

$$HB = \frac{2 \times P}{\pi \times D \times (D - \sqrt{D^2 - d^2})} \quad (\text{III.08})$$

Where:

- **HB:** Number of Brinell Hardness [MPa];
- **P:** Load applied [In Kilograms-Force, Kgf], With 1 Kgf = 9.80665 N;
- **D:** Diameter of the Steel Ball Indenter [mm], In this Case, 2.5 mm;
- **d:** Average Diameter of the Indentation Measured in Two Directions  $d_1$  and  $d_2$  [mm];
- $\pi$ : Approximately 3.1416.



Furthermore, the INNOVATEST VERZUS 750CCD calculates the values of Brinell Hardness (HB) number based on the applied load (P) (6.2 kgf or 60.80123 N), the diameter of the steel ball (D) ( $\varnothing$  2.5), and the average diameter (d) of the indentation measured in two directions,  $d_1$  and  $d_2$  [287]. This value is determined using Equation (III.08) for each specimen. The five specimens were taken from each group, regardless of whether they were made of gypsum or bio-composite materials (GF.s.m). This makes the results statistically significant by accounting for material effects. All tests must use the same load, timing, and measurement methods. After performing the Brinell test on the five specimens, the average and standard deviation Brinell Hardness (HB) number is determined for each type of specimen (Bio-composite Materials (GF.s.m), and Gypsum).

Ultimately, by monitoring the performance of the bio-composite materials (GF.s.m) under pressure, their hardness values will be compared to those of gypsum. This process provides accurate and reliable Brinell hardness measurements of the materials, facilitating detailed comparisons of their surface hardness properties.

#### III.2.2.4. Charpy Impact Strength Test

Charpy impact strength tests were performed on bio-composites (GF.s.m) and gypsum to determine the impact resistance of these materials. Test specimens were manufactured according to ISO-179-1:2023 and measured 10 mm  $\times$  10 mm  $\times$  55 mm, as shown in Figure III.15.a [276]. A V-shaped notch was cut at a 45° angle in the center of each specimen, 2 mm deep and 0.25 mm radius, as shown in Figure III.15.a. A Charpy impact testing apparatus from KARL FRANK GMBH WEINHEIM-BIRKENAU was used for the experiments, as shown in Figure III.15.b [288-290]. The apparatus contains a pendulum that swings freely around a circular base, as shown in Figure III.15.a. Two joule of energy is required to move the pendulum, as shown in Figures III.15.a, and c. The pendulum was set so that it strikes the specimen at the notch, as shown in Figure III.15.d. This allows the material to be subjected to a controlled force to assess its ability to withstand impact or fracture.

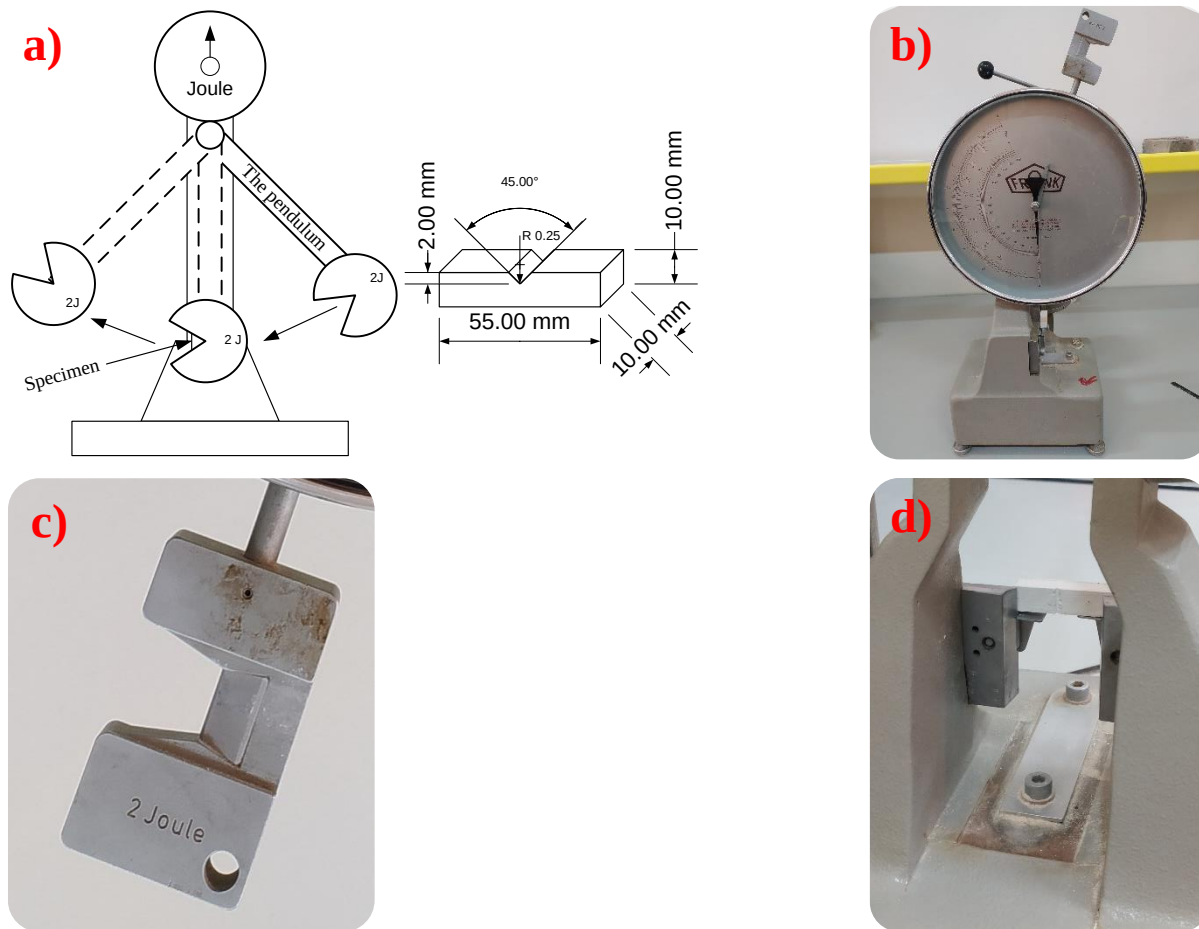


**Figure III.15. Charpy Impact Strength Test:**  
**a) The Specimen Size and the Pendulum Swing, b) The KARL FRANK GMBH WEINHEIM-BIRKENAU Device, c) The Pendulum of Two Joules, and d) The Specimen at Device Support.**

Furthermore, each specimen is attached to the gadget so that the notch points toward where the pendulum will hit. The pendulum goes and let it swing back and forth over the round base. The pendulum will hit the specimen at the V-shaped notch, which will cause a fracture depending on how strong the material is against impact. The instrument measures the amount of energy the specimen absorbs as it breaks. The energy that is absorbed is related to how well the material can resist impacts, which is also called Charpy impact strength.

The following Equation (III.09) is used to get the Charpy impact strength from the test results:

$$G = \frac{U}{a \times s} \quad (\text{III.09})$$

Where:

- **G:** Charpy Impact Strength [ $\text{KJ/m}^2$ ];
- **U:** Energy Absorbed During the Impact, Typically Measured by the Apparatus as The Difference Between the Potential Energy of the Pendulum Before and After Impact [ $\text{KJ}$ ];
- **a:** Width of Specimen [ $\text{m}$ ];
- **s:** Thickness of Specimen [ $\text{m}$ ].

Furthermore, the KARL FRANK GMBH WEINHEIM-BIRKENAU device calculates the values of Charpy impact strength (G) based on (U), which is energy absorbed during the impact in joules provided by the testing machine, and (a) is the width and (s) is the thickness of the specimens, respectively [288-290]. This value is determined using Equation (III.09) for each specimen. To obtain valid data, five specimens of each type of material (Bio-composite Materials (GF.s.m), and Gypsum) were tested. This ensured that each test was performed in the same way, with the pendulum swinging at the same height and with the same amount of energy. After evaluating all five specimens for each type of material (Bio-composites (GF.s.m) and Gypsum), the mean and standard deviation of Charpy impact strength of each kind were determined.

Ultimately, after obtaining the average Charpy impact strength values for bio-composites (GF.s.m) and gypsum, they are compared to determine which one is better at resisting impact fractures. This approach also enables us to assess their effectiveness after exposure to impacts.

### III.2.3. Physical Tests

The physical tests that will be conducted on the bio-composites (GF.s.m) and will be compared with gypsum are highlighted in the following tests:

#### III.2.3.1. Bulk Density Test

Bulk density tests were done on bio-composites (GF.s.m) and gypsum to see how light they are when employed in different applications, according to ASTM C29/C29M-23 [277]. Before testing, the size of each specimen was verified to be  $20 \text{ mm} \times 90 \text{ mm} \times 90 \text{ mm}$ , ensuring that the measurements were accurate during testing, as shown in Figure III.16.a. The specimens were placed at  $20^\circ\text{C}$  and 65% relative humidity for 24 hours before the wholesale density test. It ensures material stability and coherent consequences in specimens.

Furthermore, a KERN PLS 420-3F electronic balance that was accurate to 0.001 grams was used to weigh each specimen, as shown in [Figure III.16.b](#) [291]. To be sure that nothing outside the balance affected the measurement, the specimens were carefully put on it. Five times, the weight of each specimen in grams was written down. Equation ([III.10](#)) was used to get the average of the five specimen weight measurements:

$$m = \frac{\sum_{i=1}^5 m_i}{5} \times 10^{-3} \quad (\text{III.10})$$

Where:

- **m**: Average Weight of Specimen [Kg];
- **m<sub>i</sub>**: Weight of Specimen [g].

In addition, a Berent BT 4151 electronic caliper with an accuracy of 0.01 mm was used to measure the specimen dimensions (Length, Width, and Height) five times and at different positions (i) for each dimension, as shown in [Figure III.16.c](#) [292]. The average of each five measurements for each specimen dimension was then calculated using Equations ([III.11](#), [12](#), and [13](#)):

$$L_{\text{Specimen}} = \frac{\sum_{i=1}^5 L_i}{5} \quad (\text{III.11})$$

$$W_{\text{Specimen}} = \frac{\sum_{i=1}^5 W_i}{5} \quad (\text{III.12})$$

$$H_{\text{Specimen}} = \frac{\sum_{i=1}^5 H_i}{5} \quad (\text{III.13})$$

Where:

- **L<sub>Specimen</sub>**: Average Length of Specimen [mm];
- **W<sub>Specimen</sub>**: Average Width of Specimen [mm];
- **H<sub>Specimen</sub>**: Average Height of Specimen [mm];
- **L<sub>i</sub>**: Length of Specimen at Position (i) [mm];
- **W<sub>i</sub>**: Width of Specimen at Position (i) [mm];
- **H<sub>i</sub>**: Height of Specimen at Position (i) [mm].

The specimen volume is calculated based on Equation ([III.14](#)):

$$v = (L_{\text{Specimen}} \times W_{\text{Specimen}} \times H_{\text{Specimen}}) \times 10^{-9} \quad (\text{III.14})$$

Where:

- **v:** The Volume of Specimen [ $\text{m}^3$ ];
- **L<sub>Specimen</sub>:** Average Length of Specimen [mm];
- **W<sub>Specimen</sub>:** Average Width of Specimen [mm];
- **H<sub>Specimen</sub>:** Average Height of Specimen [mm].



**Figure III.16. Bulk Density Test: a) The Specimen Size, b) Weigh the Specimen, and c) The Specimen Measurements.**

The following Equation (III.15) is used to get the bulk density from the test results:

$$\rho = \frac{m}{v} \quad (\text{III.15})$$

Where:

- **$\rho$ :** Bulk Density of Specimen [ $\text{Kg}/\text{m}^3$ ];
- **m:** The Weight of Specimen [Kg];
- **v:** The Volume of Specimen [ $\text{m}^3$ ].

Furthermore, this test calculates the value of bulk density ( $\rho$ ) of the specimen based on (m), which represents the weight of the specimen, while (v) denotes the volume of the specimen. This value is determined using Equation (III.15) for each specimen. The bulk density testing was performed on five specimens for each type of material (Bio-composite Materials (GF.s.m) and Gypsum) to ensure the reliability and accuracy of the results. Each test was performed under equal conditions to reduce measurement errors. After testing five specimens for each type of material, the average and standard deviations were calculated to measure the variation in the bulk density values.

Ultimately, after obtaining the average bulk-density values, the results will be compared to assess the harmony and mass distribution of the material. Following these steps will provide reliable and accurate

results for wholesale density for specimens. The wholesale values will help to understand the harmony and structural properties of the bio-composite materials (GF.s.m) compared to gypsum.

### III.2.3.2. Water Absorption Test

Water absorption tests were done on bio-composites (GF.s.m) and gypsum to see how much water a specimen absorbs after being submerged for a set amount of time when employed in different applications, according to ASTM C642–13 [278]. The specimens are sized 20 mm × 90 mm × 90 mm, as shown in [Figure III.16.a](#). The specimens are put in water at 21°C for 48 hours, as shown in [Figure III.17.a](#). During this time, the specimens will be fully submerged in water at a constant temperature.

Furthermore, the specimens are taken out of the water after 48 hours. A dry cloth is used to wipe away any extra water droplets from the specimen surfaces. It does not take much effort to remove water from the surface of the specimen so as not to strip away the water absorbed. Then, each specimen is weighed separately using a KERN PLS 420-3F electronic balance that is accurate to 0.001 grams, as shown in [Figure III.17.b](#) [291]. The specimen must be weighed immediately upon removal and wiped with a cloth to obtain precise measurements.

In addition, the specimens were placed in a Nabertherm 30-3000°C dryer, set at 110°C, and allowed to dry for three days, as shown in [Figure III.17.c](#) [268]. This ensured that all the water absorbed by the specimens evaporated. The specimens were cooled after being removed from the oven. They were then placed in a Nabertherm 30-3000°C dryer set at 25°C for an additional two hours [268]. After the final drying stage, each specimen was weighed again using a KERN PLS 420-3F balance with an accuracy of 0.001 grams, as shown in [Figure III.17.d](#) [291]. This weight can be considered the dry weight of the specimen.

The following Equation ([III.16](#)) is used to get the water absorption ratio from the test results:

$$\text{WAR} = \frac{W_W - W_D}{W_D} \times 100 \quad (\text{III.16})$$

Where:

- **WAR:** Water Absorption Ratio of Specimen [%];
- **W<sub>w</sub>:** Weight of the Specimen after Immersion and Surface Cleaning [g];
- **W<sub>D</sub>:** Weight of the Specimen after Drying in the Oven [g].



**Figure III.17. Water Absorption Test: a) The Immersion of the Specimen, b) Weighing the Specimen after Immersion, c) Drying the Specimen, and d) Weighing the Specimen after Drying in the Dryer.**

Furthermore, this test calculates the ratio of water absorption (WAR) of the specimen based on ( $W_w$ ), which represents the weight of the specimen after immersion and surface cleaning. At the same time, ( $W_D$ ) denotes the weight of the specimen after drying in the oven. This value is determined using Equation (III.16) for each specimen. Water absorption tests were conducted on five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum) to verify the reliability and precision of the results. All tests were conducted under uniform settings to minimize measurement mistakes. Following the examination of five specimens for each material type, the average and standard deviations were computed to assess the variability in water absorption values.

Ultimately, this test indicates that the material can absorb more water, potentially leading to increased water intrusion and compromised durability and performance.

#### III.2.3.3. Thickness Swelling Test

Thickness swelling tests were conducted on bio-composites (GF.s.m) and gypsum to determine the thickness of the specimens when they absorb water, which occurs when the materials are used in ceiling and wall panel applications, according to ASTM D1037 [279].



The specimens measuring 20 mm × 90 mm × 90 mm were prepared for testing, as shown in [Figure III.16.a](#). These specimens were made and arranged like water absorption test specimens. The thickness of each specimen was measured five times at predetermined positions (i), with 15 mm between each position (i), as shown in [Figure III.18.a](#). They used an electronic caliper, Berent BT 4151, to take measurements [292]. Equation (III.17) was used to determine the average thickness from the five pre-immersion measurements:

$$h_1 = \frac{\sum_{i=1}^5 h_{1,i}}{5} \quad (\text{III.17})$$

Where:

- **h<sub>1</sub>**: Average of the Specimen Thickness Measurements before Immersion [mm].
- **h<sub>1,i</sub>**: Measurement of the Specimen Thickness before Immersion at Position (i) [mm].

Furthermore, after measuring the thickness of the specimens before immersion, they were placed in water at 21°C for 24 hours, as shown in [Figure III.18.b](#). Ensure that they are completely submerged at the same time, and maintain the water temperature at 21°C. The specimens were then removed from the water after 24 hours. In addition, thickness measurements were taken for each specimen at the same positions (i) as before immersion, as shown in [Figure III.18.c](#). Equation (III.18) was used to determine the average thickness from the five after-immersion measurements:

$$h_2 = \frac{\sum_{i=1}^5 h_{2,i}}{5} \quad (\text{III.18})$$

Where:

- **h<sub>2</sub>**: Average of the Specimen Thickness Measurements after Immersion [mm].
- **h<sub>2,i</sub>**: Measurement of the Specimen Thickness after Immersion at Position (i) [mm].





**Figure III.18. Thickness Swelling Test:**

**a) Specimen Thickness Measurements before Immersion, b) Immersion of Specimens, and c) Specimen Thickness Measurements after Immersion.**

The following Equation (III.19) is used to get the thickness swelling ratio from the test results:

$$T_s = \frac{h_2 - h_1}{h_1} \times 100 \quad (\text{III.19})$$

Where:

- **T<sub>s</sub>**: Thickness Swelling Ratio of Specimen [%];
- **h<sub>1</sub>**: Average of the Specimen Thickness Measurements before Immersion [mm];
- **h<sub>2</sub>**: Average of the Specimen Thickness Measurements after Immersion [mm].

Furthermore, this test calculates the ratio of thickness swelling ( $T_s$ ) of the specimen based on ( $h_1$ ), which represents the average of the specimen thickness measurements before immersion. At the same time, ( $h_2$ ) denotes the average of the specimen thickness measurements after immersion. This value is determined using Equation (III.19) for each specimen. Thickness swelling tests were conducted on five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum) to verify the reliability and precision of the results. All tests were conducted under uniform settings to minimize measurement mistakes. After examining five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum), the average was calculated, along with standard deviations, to assess the variability in thickness swelling values.

Ultimately, by obtaining accurate and reliable information about the amount of water absorbed by bio-composite materials (GF.s.m) and gypsum, and how they swell when wet, we can compare the performance of each material when exposed to water.

### III.2.3.4. Moisture Content Test

Moisture content tests were conducted on bio-composites (GF.s.m) and gypsum, according to ASTM D2216-10 [280]. The goal of this test is to find out how much moisture is in bio-composite materials (GF.s.m) as compared to gypsum so that too much moisture does not cause mold to grow or materials to break down or deformations, which would make them less effective as insulation for walls and roofs. Specimens with dimensions of 20 mm × 90 mm × 90 mm were prepared, as shown in [Figure III.16.a](#). Then each specimen is weighed individually to determine its initial weight before drying. To record the initial mass of each specimen, use a KERN PLS 420-3F electronic balance with an accuracy of 0.001 g to ensure that the measurements are as accurate as possible, as shown in [Figure III.19.a](#) [291].

Furthermore, the specimens will then be put in a Nabertherm 30-3000°C dryer set to 60°C for 24 hours, as shown in [Figure III.19.b](#) [268]. This method makes sure that all of the free moisture in the specimens evaporates, leaving only the dry material. The drying procedure must maintain a temperature of 60°C. After 24 hours of drying, the specimens are taken out of the oven and left to cool to room temperature. Each dried specimen is then weighed using a KERN PLS 420-3F electronic balance, with an accuracy of 0.001 grams, as shown in [Figure III.19.c](#) [291].



**Figure III.19. Moisture Content Test: a) Specimen Weight before Drying, b) Drying Specimens in a Dryer, and c) Specimen Weight after Drying.**

The following Equation ([III.20](#)) is used to get the moisture content ratio from the test results:

$$MC = \frac{m_{MC.W} - m_{MC.D}}{m_{MC.D}} \times 100 \quad (\text{III.20})$$

Where:

- **MC:** Moisture Content Ratio of Specimen [%];
- **$m_{MC.W}$ :** The Specimen Mass before Entering the Oven for Drying [g];
- **$m_{MC.D}$ :** The Specimen Mass after Entering the oven for Drying [g].

Furthermore, this test calculates the ratio of Moisture Content (MC) of the specimen based on ( $m_{MC.W}$ ), which represents the specimen mass before entering the oven for drying. At the same time, ( $m_{MC.D}$ ) denotes the specimen mass after entering the oven for drying. This value is determined using Equation (III.20) for each specimen. Moisture content tests were conducted on five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum) to verify the reliability and precision of the results. All tests were conducted under uniform settings to minimize measurement mistakes. After examining five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum), the average was calculated, along with standard deviations, to assess the variability in moisture content values.

Ultimately, the primary purpose of this test is to find materials that are too wet, which can cause mold to grow and damage. Materials that contain a high amount of moisture may not be suitable for use as insulation in roofs and walls, as they can cause structural issues over time. By performing these tests, accurate and reliable results will be obtained regarding the moisture content of materials, enabling their performance to be evaluated in areas where moisture is a concern.

### III.2.4. Thermal Tests

The thermal test that will be conducted on the bio-composites (GF.s.m) and will be compared with gypsum is highlighted in the following test:

#### III.2.4.1. Thermal Conductivity Test

Thermal conductivity tests were conducted on bio-composites (GF.s.m) and gypsum to determine the thermal properties of the specimens when they are used in ceiling and wall panel applications, according to ISO 22007-2:2022 [281]. For the test, specimens with sizes of 20 mm × 90 mm × 90 mm were prepared, as shown in Figure III.16.a. Test conditions were 16.9 °C and 27% relative humidity, as shown in Figure III.20.a. A HOT Disk TPS500, which was a thermal conductivity tester, was used to determine how well the specimens conducted heat, as shown in Figure III.20.b [293]. The thermal properties calculated by the device are thermal conductivity ( $\lambda$ ), which represents the rate of heat transfer through

the medium. Thermal deficiency ( $\alpha$ ) measures the speed of the heat flow in a substance. In addition, specific heat capacity ( $C$ ) shows how much heat is required to increase the temperature of the material by one degree Celsius.

Furthermore, the Hot Disc TPS500 features a thermal sensor that can be used to heat specimens and measure their temperature, as shown in [Figure III.20.c](#) [293]. The heat sensor is on the top of the specimen surface, as shown in [Figure III.20.d](#). After that, the higher specimen is put on top of the lower specimen, with the thermal sensor in between, as shown in [Figure III.20.e](#). When testing, it is essential to ensure that the two specimens are aligned precisely so that their thermal properties can be measured accurately. The two specimens must be of the same type, either bio-composite material (GF.s.m) or gypsum, and have a smooth surface. The two specimens must then be secured with a mounting screw that is in the device, as shown in [Figure III.20.e](#). Thermal properties are measured by heating the specimen by a specified amount and then monitoring the temperature change. The Hot Disc TPS500 also tracks the temperature response over time [293]. This information is then used to determine thermal conductivity ( $\lambda$ ), thermal diffusivity ( $\alpha$ ), and specific heat capacity ( $C$ ).

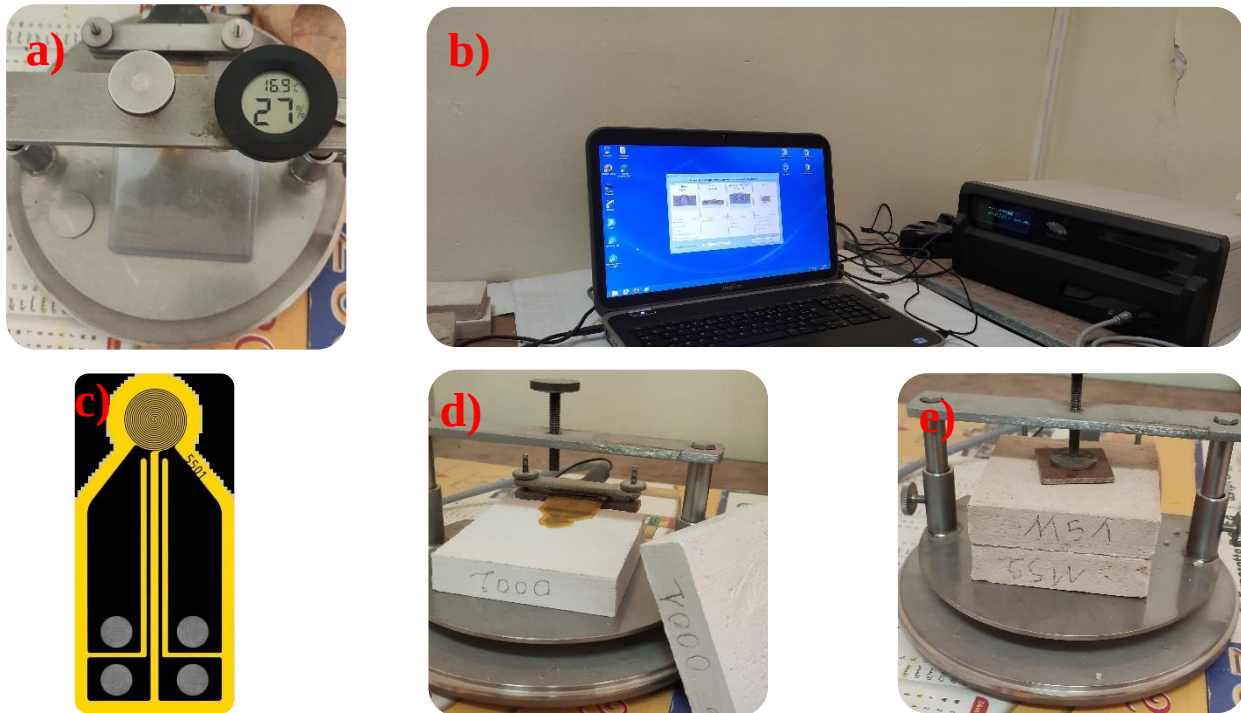


**Figure III.20. Thermal Conductivity Test: a) The Test conditions, b) The Hot Disc TPS500 Device, c) The Thermal Sensor, d) The Thermal Sensor in Top Specimen Surface, and e) The Fixation of Two Specimens with a Mounting Screw.**

The HOT Disk system's built-in analysis calculates thermal conductivity ( $\lambda$ ) [293]; however, the basic calculation is usually based on Fourier's Equation of heat conduction, as shown in [Figure III.21](#), and Equation ([III.21](#)) [294]:

$$\lambda = \frac{Q \times \Delta x}{A \times \Delta T} \quad (\text{III.21})$$

Where:

- **$\lambda$** : The Thermal Conductivity of Specimen [W/m·k];
- **$Q$** : The Amount of Heat Supplied [W];
- **$\Delta x$** : The Difference in Specimen Thickness [m];
- **$A$** : The Cross-Sectional Area of the Specimen [m<sup>2</sup>];
- **$\Delta T$** : The Temperature Difference across the Specimen [K].

With:

$$\Delta x = (x_2 - x_1) \times 10^{-3} \quad (\text{III.22})$$

Where:

- **$\Delta x$** : The Difference in Specimen Thickness [m];
- **$x_1$** : The Specimen Thickness at the Start [mm];
- **$x_2$** : The Specimen Thickness at the End [mm].

With:

$$\Delta T = (T_1 - T_2) + 273,15 \quad (\text{III.23})$$

Where:

- **$\Delta T$** : The Temperature Difference across the Specimen [K];
- **$T_1$** : The Specimen Temperature at the Start [°C];
- **$T_2$** : The Specimen Temperature at the End [°C].

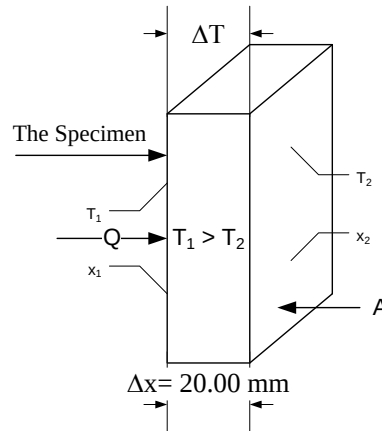


**Figure III.21. The Heat Transfer in the Specimen.**

In addition, thermal conductivity ( $\lambda$ ) is typically measured directly using a device such as the HOT Disk TPS500 System, which uses transient heat flux to determine the speed at which heat passes through the material [293]. The HOT Disk TPS500 system also automatically measures thermal diffusivity ( $\alpha$ ) based on the temperature response and heat diffusion through the material [293]. In addition, the bulk density ( $\rho$ ) is pre-measured for the bio-composite material (GF.s.m) or gypsum. The specific heat ( $C$ ) is calculated using Equation (III.24) along with the values of thermal conductivity, thermal diffusivity, and bulk density.

$$C = \frac{\lambda}{\alpha \times \rho} \quad (\text{III.24})$$

Where:

- **C:** The Specific Heat of Specimen [J/kg·k];
- **$\lambda$ :** The Thermal Conductivity of Specimen [W/m·k];
- **$\alpha$ :** The Thermal Diffusivity of Specimen [m<sup>2</sup>/s];
- **$\rho$ :** The Bulk Density of Specimen [Kg/m<sup>3</sup>].

Furthermore, this test calculates the value of the thermal conductivity ( $\lambda$ ) of the specimen based on ( $C$ ), which represents the specific heat. At the same time, ( $\alpha$ , and  $\rho$ ) denote the thermal diffusivity and bulk density, respectively. This value is determined using Equation (III.24) for each specimen. Thermal conductivity tests were conducted on five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum) to verify the reliability and precision of the results. All tests were conducted

under uniform settings to minimize measurement mistakes. After examining five specimens of each material type (Bio-composite Materials (GF.s.m) and Gypsum), the average was calculated, along with standard deviations, to assess the variability in thermal properties values.

Ultimately, comparing the thermal conductivity of gypsum and the composite materials (GF.s.m) is crucial. High thermal conductivity means that a material can quickly move heat, while low thermal conductivity is usually what you want for insulating substances.

### **Note:**

The average and standard deviation mentioned in the description of the tests of flexural (3-points), compressive strength, Brinell hardness, Charpy impact strength, bulk density, water absorption, thickness swelling, moisture content, and thermal conductivity they calculated by the Equations (III.25, and 26).

$$\text{Average} = \frac{\sum_{j=1}^5 \text{The Value of Specimen } j}{5} \quad (\text{III.25})$$

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{j=1}^5 (\text{Specimen } j - \text{Average})^2}{5 - 1}} \quad (\text{III.26})$$

Where:

- **j:** The Specimen Number [-].

Ultimately, the average and standard deviation mentioned in the description of each test are important statistical results for obtaining reliable results.

### **III.2.5. Chemical Tests**

The chemical tests that will be conducted on the bio-composites (GF.s.m) and will be compared with gypsum are highlighted in the following tests:

#### **III.2.5.1. Energy Dispersive X-Ray Spectroscopy (EDS) Test**

A Prisma E Scientific Scanning Electron Microscope (SEM) equipped with Energy-Dispersive X-Ray Spectroscopy (EDS) was used on specimens with the minimum, mean, and maximum ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ , Respectively) flexural modulus values among bio-composite materials (GF.s.m), as shown



in **Figure III.22.a** [295]. The electron beam voltage was set at 10 kV for the Scanning Electron Microscope (SEM) test. This setting is advantageous for high-resolution imaging of materials such as gypsum-based bio-composites and palm fibers, which require excellent surface sensitivity. The specimens were placed on aluminum stubs, as shown in **Figure III.22.b**. These stubs are used to stabilize the specimen in the Scanning Electron Microscope (SEM) chamber to prevent it from moving during imaging. This setting is important for bio-composite materials (GF.s.m).

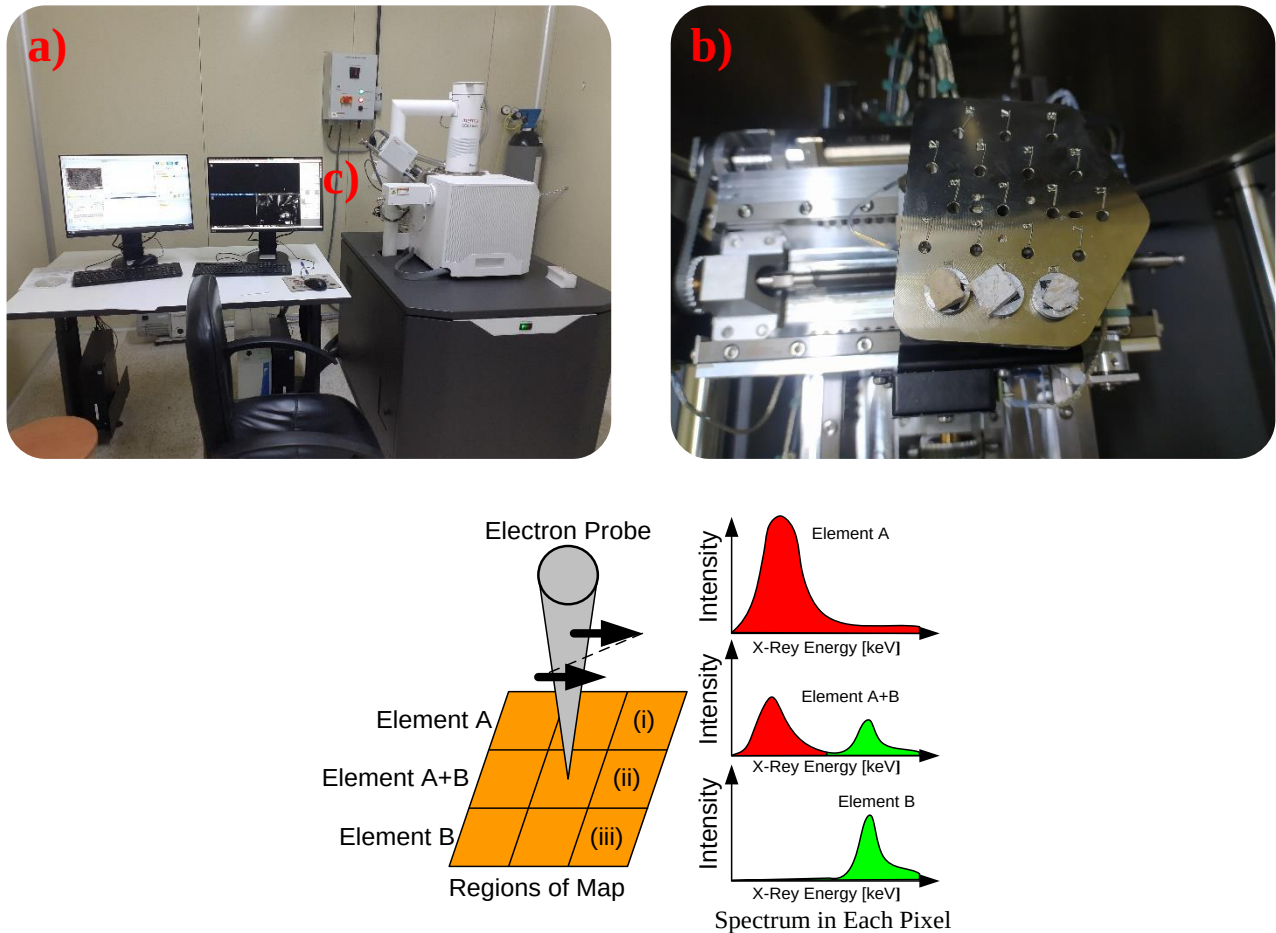


**Figure III.22. The Energy Dispersive X-Ray Spectroscopy (EDS) Test:**  
**a) The Prisma E Scientific Scanning Electron Microscope (SEM),**  
**b) The Specimens in an Aluminum Stub, and c) The EDS Elemental Map.**

Furthermore, the Scanning Electron Microscope (SEM) scans the surface, and the X-Ray Spectroscopy (EDS) collects information about the elements that make it up. The X-Ray Spectroscopy (EDS) works by finding X-rays that are generated when the electron beam hits the material. The X-Ray Spectroscopy (EDS) system can discover and measure the elements in a material because each element gives out X-rays at different energies. The X-Ray Spectroscopy (EDS) system will make an elemental map of the



area that was scanned, as shown in [Figure III.22.c](#) [296]. This can help determine if certain parts of the materials are linked to the minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ , Respectively) in bio-composite materials (GF.s.m).

Ultimately, to determine any differences in the bio-composite materials (GF.s.m), these results will be compared to see if there are any differences between the specimens in terms of flexural modulus, whether the effect is due to the size or mass ratio of the fibers added as reinforcement to the gypsum to make the bio-composite materials (GF.s.m).

### III.2.5.2. Fourier Transform Infrared Spectroscopy (FT-IR) Test

The goal of this Fourier Transform Infrared Spectroscopy (FT-IR) test is to look at the chemical bonds in Date Palm Fiber (DPF) and bio-composite materials (GF.s.m) specimens. The specimens demonstrate the minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ , Respectively) inside bio-composite materials (GF.s.m). The test seeks to ascertain whether gypsum and Date Palm Fiber (DPF) establish new chemical bonds in bio-composite materials (GF.s.m). It also aims to interpret the results of the flexural modulus test and understand how they relate to the chemical bonds observed in Fourier Transform Infrared Spectroscopy (FT-IR).

Furthermore, an Agilent Cary 630 Fourier Transform Infrared Spectroscopy (FT-IR) with Attenuated Total Reflectance (ATR) sampling was used, as shown in [Figure III.23.a](#) [297]. Attenuated Total Reflectance (ATR) is often used to study surfaces, as it can obtain high-quality spectra from the surface of a material without the need for special specimen preparation. To achieve high resolution in the Fourier Transform Infrared Spectroscopy (FT-IR) spectrum, the spectrometer was set to  $4 \text{ cm}^{-1}$ . The test includes 140 scans per minute. This enhances spectrum interpretation data averaging. The range was increased to  $4000\text{--}400 \text{ cm}^{-1}$ . This encompasses the normal infrared absorption range of organic and inorganic materials for the detection of functional groups and chemical bonds. In addition, the transmission (%) mode recorded light flowing through the specimen. This gives an idea of the material's absorption at specific wavelengths.

In addition, each specimen, whether bio-composite (GF.s.m) or Date Palm Fiber (DPF), is placed on the Attenuated Total Reflectance (ATR) crystal, as shown in [Figure III.23.b](#). This ensures that the specimen is in good contact with the crystal to absorb as much infrared light as possible. Gently but firmly pressed to ensure the specimen remains in contact with the Attenuated Total Reflectance (ATR) crystal. This

facilitates the acquisition of a high-quality Fourier Transform Infrared Spectroscopy (FT-IR). During the Fourier Transform Infrared Spectroscopy (FT-IR) scan to obtain the specimen's infrared spectra, the Fourier Transform Infrared Spectroscopy (FT-IR) spectrometer sends infrared light through the specimen, as shown in [Figure III.23.c](#). At the same time, the Attenuated Total Reflectance (ATR) crystal analyzes how much light the specimen absorbs at different wavelengths.

Furthermore, the Agilent Cary 630 Fourier Transform Infrared Spectroscopy (FT-IR) device will measure how much light is absorbed at different wavelengths ( $\text{cm}^{-1}$ ) in the infrared spectrum [297]. Fourier Transform Infrared Spectroscopy (FT-IR) peaks are the results of chemical vibrations. The peaks of the material show that it has chemical functional groups. Look at the Fourier Transform Infrared Spectroscopy (FT-IR) spectra to find functional groups and chemical bonds. When analyzing Fourier Transform Infrared Spectroscopy (FT-IR) spectra, we will compare the visible chemical bond to flexural modulus values obtained from mechanical testing. The bio-composite materials (GF.s.m) with better chemical bonding, like crosslinked or dry composites, may possess improved structural properties, such as a maximum flexural modulus.



**Figure III.23. Fourier Transform Infrared Spectroscopy (FT-IR) Test:**  
**a) Agilent Cary 630 Fourier-Transform Infrared (FT-IR) Device, b) The Specimen in an ATR crystal, and c) The FTIR Spectroscopy for Specimen Analysis.**

Ultimately, by performing this experiment, you will obtain high-quality Fourier Transform Infrared Spectroscopy (FT-IR) spectra that reveal the chemical bonds and functional groups in palm fibers and bio-composite materials (GF.s.m). The differences in chemical bonds observed in the Fourier Transform Infrared Spectroscopy (FT-IR) data can be correlated with flexural modulus measurements, which reveal how the material functions.

### III.2.6. Morphological Test

The morphological test that will be conducted on the bio-composites (GF.s.m) and will be compared with gypsum is highlighted in the following test:

#### III.2.6.1. Scanning Electron Microscopy (SEM) Test

The Scanning Electron Microscopy (SEM) test analysis focuses exclusively on the specimens demonstrating the minimum, mean, and maximum flexural modulus values ( $E_{3P\text{ Min}}$ ,  $E_{3P\text{ Mean}}$ , and  $E_{3P\text{ Max}}$ , Respectively) inside bio-composite materials (GF.s.m), as shown in [Figure III.22.a](#) [295]. The primary aim is to analyze the surface morphology of bio-composite materials (GF.s.m) by Scanning Electron Microscopy (SEM) [295]. The specimens are put on aluminum stubs, as shown in [Figure III.22.b](#). These stubs help keep the specimens steady in the Scanning Electron Microscopy (SEM) chamber, so they do not move or change position while imaging. This is especially important for things like bio-composite materials (GF.s.m).

Furthermore, the Scanning Electron Microscopy (SEM) was set to 10 kV for the electron beam. This is optimal for high-resolution imaging and is suitable for bio-composite materials (GF.s.m) such as palm fiber-reinforced composites and gypsum, which require high surface sensitivity [295]. Images were captured at 500× magnification to gain a general idea of the surface morphology and structure. The magnification was then switched to 85× for a closer look at some areas of interest. The Scanning Electron Microscopy (SEM) will produce high-resolution images of particle arrangement, porosity, fracture behavior, and other parameters that affect flexural modulus and structural integrity. This will enable us to compare the measured flexural modulus with the structural characteristics.

Ultimately, the structural integrity of the bio-composite material (GF.s.m) is likely closely related to both the flexural modulus and the size-to-mass ratio of the palm fibers used as reinforcement with gypsum in its production.

## Conclusion

This third chapter, focuses on the manufacturing and evaluation of the bio-composite materials by reinforcing gypsum with Date Palm Fibers (DPFs). This chapter discusses the process of preparing palm fibers, which involves several stages, from collecting palm waste from palm groves to obtaining different

sizes of palm fibers that will be used as reinforcement for bio-composite materials (GF.s.m). The gypsum matrix was also discussed to highlight its importance. In addition, an entirely new and straightforward approach was adopted to manufacture bio-composite materials (GF.s.m) with different fiber mass ratios (m: 05-20%) added as reinforcement for each size (s: I-III) of extracted palm fiber. In addition, the various methods used to test bio-composite materials (GF.s.m) and gypsum, including three-point flexural tests, compressive strength tests, Brinell hardness tests, Charpy impact strength tests, bulk density tests, water absorption and thickness swelling tests, moisture content tests, thermal conductivity tests, Energy Dispersive X-ray Spectroscopy (EDS), Fourier Transform Infrared Spectroscopy (FT-IR), and Scanning Electron Microscopy (SEM) to interpret and support the results obtained, demonstrate that the process is thorough and systematic. These tests provide a comprehensive understanding of how well the materials perform in various environments and their potential applications for building and insulation. The main objective of selecting each test is highlighted by:

1. **In mechanical tests**, the three-point flexural test will measure the flexural strength, flexural modulus, and strain at break. These values, which may show structural integrity, can be affected by composition, porosity, mass ratio, and fiber size. The results can also be used to test gypsum and bio-composite materials (GF.s.m) used for insulating walls and ceilings, which require flexuring and load-bearing capacity. Tests for compressive strength and Brinell hardness can indicate how well the materials resist buckling and localized deformation. Compression testing can also reveal the strength and durability of the materials, as well as their suitability for building purposes. In addition, Charpy impact strength tests measure how well materials can handle sudden blows and how much energy they can absorb. This test is important for figuring out how these materials act when shocks or other short-lived forces hit them;
2. **In physical testing**, bulk density testing may indicate the density of the tested materials, which affects their strength, durability, and ability to conduct heat. Water absorption and thickness swelling expansion tests will indicate that a material's resistance to moisture directly affects its performance when wet, mainly when used for insulation. These tests may show that gypsum and bio-composite materials (GF.s.m) do not perform well in this situation because they absorb moisture and expand, accelerating their decomposition. In addition, moisture content testing may also focus on the water in the material to determine how moisture can lead to mold

growth, material deterioration, and reduced performance. For building materials to last a lifetime, they must be kept dry, especially in humid locations;

3. **In thermal tests,** the thermal conductivity test will reveal important information about the heat transfer of the tested materials and their suitability for use as thermal insulation for walls and roofs. In addition, when palm fibers are incorporated into gypsum, the thermal conductivity may decrease, demonstrating that the resulting bio-composite material (GF.s.m) offers high thermal insulation performance;
4. **In chemical testing,** Energy-Dispersive X-ray Spectroscopy (EDS) will look at the different parts of the examined material by using chosen bio-composite (The Minimum, Mean, and Maximum Flexural Modulus Values  $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ , Respectively) specimens. Lastly, Fourier transform infrared (FT-IR) spectroscopy was employed to investigate the bonding of the components in the bio-composite materials (GF.s.m). This test could show how adding palm fibers to gypsum as a bio-composite reinforcement (GF.s.m) modifies the chemical bonds and makes new ones. There will also be a connection made between the findings of tests, Energy-Dispersive X-ray Spectroscopy (EDS), and Fourier Transform Infrared Spectroscopy (FT-IR) for the specimens that were analyzed;
5. **In morphological testing,** the Scanning Electron Microscope (SEM) is used to demonstrate porosity, roughness, and surface architecture of the material. Morphological and compositional factors can help us understand how microstructural factors influence macroscopic behaviors, such as mechanical, physical, thermal, and chemical.

Furthermore, this method enables you to accurately evaluate materials by examining their mechanical, physical, thermal, chemical, and morphological properties. Adding palm fibers to gypsum to make bio-composites (GF.s.m), which will be compared to gypsum, will change several features. They will also help us identify building and insulation materials that meet standards for strength, durability, and efficiency.

Ultimately, this third chapter provides a comprehensive understanding of how palm fibers are prepared as reinforcement, a more detailed understanding of gypsum as a matrix in bio-composites (GF.s.m), and the preparation of various bio-composites (GF.s.m) with varying fiber mass ratios (m: 05-20%) relative to each selected palm fiber size (s: I-III). This third chapter provides equipment and devices used in

multiple tests, which are also discussed, leading to a consistent approach across tests to obtain multiple specifications for bio-composites (GF.s.m) compared to gypsum. In the building industry, the outcomes of these thorough evaluations will help create new materials or enhance existing ones, enabling better bio-composite material selection and more efficient design solutions.

# Chapter IV

## Results and Discussions of Experimental Tests

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## Introduction

In this fourth chapter, present and analyze the comprehensive results of the experimental investigations conducted on the bio-composite materials (GF.s.m) specimens as compared to gypsum. The findings are organized into several sections, beginning with a general overview of the mechanical, physical, and thermal properties. This fourth chapter explores the specific measurements related to flexural strength, flexural modulus, compressive strength, compressive modulus, Brinell hardness, and Charpy impact strength as mechanical properties. In addition, bulk density, water absorption, thickness swelling, and moisture content as physical measurement properties. Further, examine the material's thermal performance, including conductivity, diffusivity, and specific heat, followed by an analysis of thermo-physical interactions.

In addition, the fourth chapter also delves into the details of the bio-composite material's (GF.s.m) experimental chemical composition results using techniques such as Energy Dispersive X-ray Spectroscopy (EDS) and Fourier Transform Infrared Spectroscopy (FT-IR) and provides insights into the morphological characteristics through Scanning Electron Microscopy (SEM) as morphological properties. These results offer a holistic understanding of the bio-composite material's (GF.s.m) properties, contributing to the broader scope of the study's objectives. The systematic presentation of these results aims to enhance the interpretation of the data, ensuring clarity in how the bio-composite material's (GF.s.m) performance meets the set criteria for practical applications.

Ultimately, this fourth chapter sets the foundation for the ensuing discussions and interpretations, as the results provide the necessary context for assessing the bio-composite material's (GF.s.m) potential in various applications.

### IV.1. The Results Organigram

**Figures IV.01.** Shows a diagram that organizes the experimental results related to the characterization of bio-composites compared to gypsum. The results are classified into six main categories: mechanical results, physical results, thermal results, thermos-physical results, chemical results, and morphological results.

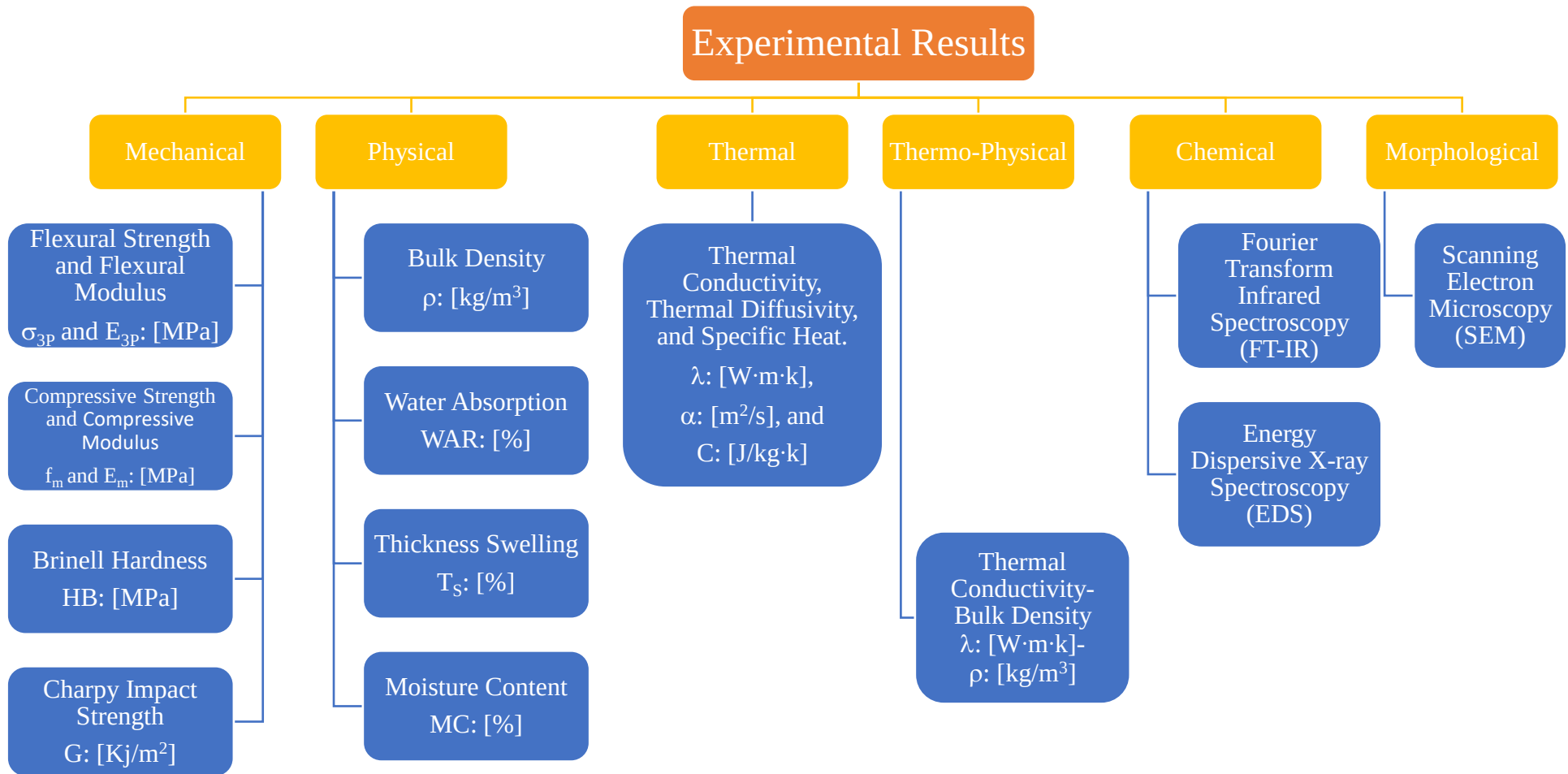


**Figure IV.01. The Experimental Results Diagram.**

**Figure IV.01.** Shows this organization enables a comprehensive assessment of bio-composite materials (GF.s.m) properties in various areas, which is crucial for understanding the material's performance in various applications. The main categories include the following:

- 1. Mechanical Results:** This category tests the mechanical properties of specimens of the bio-composite material (GF.s.m) by comparing them to a gypsum specimen. The specimens use different sizes (s: I-III) of Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) for each size to see if they are suitable for construction. The specimens were evaluated for flexural strength and modulus ( $\sigma_{3P}$ , and  $E_{3P}$ ), which indicated that the material can flex when it is under pressure. This is especially important for bio-composite materials (GF.s.m), which need to stay strong when they are under stress. In addition, compressive strength and compressive modulus ( $f_m$ , and  $E_m$ ) also helps us better understand how well a bio-composite material (GF.s.m) can resist crushing forces, which is an important attribute for materials utilized in load-bearing applications. Furthermore, the Brinell hardness (HB) tells how well a bio-composite material (GF.s.m) can resist local deformation, which gives you a direct idea of how long it will last. Furthermore, the Charpy impact strength (G) measures how strong a bio-composite material (GF.s.m) is and how well it can absorb energy when it is hit. This is especially true for places where the mechanical load changes;
- 2. Physical Results:** This category tests the physical properties of specimens of the bio-composite material (GF.s.m) by comparing them to a gypsum specimen. The specimens use different sizes (s: I-III) of Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) for each size to see if they are suitable for construction. The physical properties, including bulk density ( $\rho$ ), are essential for understanding the bio-composite material's (GF.s.m) cohesion and, indirectly, its strength and thermal characteristics. In addition, bio-composite materials (GF.s.m) are tested for their water absorption ratio (WAR), which tells how easily water can pass through them. Furthermore, the thickness swelling ratio ( $T_s$ ) is crucial since it tells how much the bio-composite materials (GF.s.m) grow when they get wet. This reveals how stable it is when it is humid. Furthermore, the moisture content ratio (MC) is crucial for bio-composite materials (GF.s.m) that will be used in places where the humidity changes a lot, since the moisture content can change the qualities of materials a lot;

3. **Thermal Results:** This category tests the thermal properties of specimens of the bio-composite material (GF.s.m) by comparing them to a gypsum specimen. The specimens use different sizes (s: I-III) of Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) for each size to see if they are suitable for construction. The thermal conductivity, thermal diffusivity, and specific heat capacity ( $\lambda$ ,  $\alpha$ , and  $C$ ) are important factors that affect how heat moves through bio-composite material (GF.s.m). In addition, this affects how well it works for thermal insulation, energy storage, or high-temperature applications. Furthermore, changing the composition and structure of the bio-composite materials (GF.s.m) can make it easier to control their thermal qualities;
4. **Thermo-Physical Results:** This category tests the thermo-physical properties of specimens of the bio-composite material (GF.s.m) by comparing them to a gypsum specimen. The specimens use different sizes (s: I-III) of Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) for each size to see if they are suitable for construction. This thermal conductivity-bulk density test shows bio-composite material (GF.s.m) behavior. Thermal conductivity and bulk density ( $\lambda$ - $\rho$ ) are also highly essential. It also displays how well it works, which is vital for applications that need much heat;
5. **Chemical Results:** The need to undertake a chemical analysis to find out what the bio-composite material (GF.s.m) is made of and how its molecules interact with each other. The used Energy-Dispersive X-ray Spectroscopy (EDS). Furthermore, this method also lets us find out what elements are in the material by looking at certain specimens of bio-composite material (GF.s.m) that have the minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ ), respectively. In addition, it is crucial to ascertain the influence of the ingredients in the bio-composite material (GF.s.m) on its chemical, mechanical, and reactive properties. In addition, the Fourier Transform Infrared Spectrometer (FT-IR) also uses infrared radiation to study how chemical bonds form in the bio-composite material (GF.s.m) between Date Palm Fibers (DPFs) and gypsum. The test uses Date Palm Fibers (DPFs) and bio-composite material (GF.s.m) specimens that exhibit minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ ), respectively. Furthermore, this is to find out how chemical bonding changes different qualities and whether new chemical bonds can be made;

- 6. Morphological Results:** To understand how bio-composite material (GF.s.m) works on a morphological level, it is important to know its morphological qualities. A Scanning Electron Microscope (SEM) was used to take close-up images of the specimen surface, which helped us determine fine details such as the fiber orientation, distribution, and how they adhere to the matrix. In addition, the study focused on specimens of bio-composite material (GF.s.m) that have the minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ ), respectively.

Ultimately, by the experimental results diagram, significant insights can be gleaned regarding the prospective benefits of bio-composites (GF.s.m) in contrast to conventional materials like gypsum, particularly in contexts where enhanced mechanical, thermal, and moisture resistance characteristics are essential.

## IV.2. Mechanical Results

The results obtained from [Table IV.01](#). are discussed as mechanical properties related to the bio-composite material specimens (GF.s.m), including different sizes (s: I-III) and mass ratios (m: 05-20%) of each size of Date Palm Fiber (DPF) compared to the gypsum specimen, as follows:

### IV.2.1. Flexural Strength and Modulus Results

[Figure IV.02](#). Shows the average flexural strength of five specimens as a function of strain at break for gypsum compared to bio-composite materials (GF.s.m), as values of mechanical properties. The findings are presented as a line graph as follows:

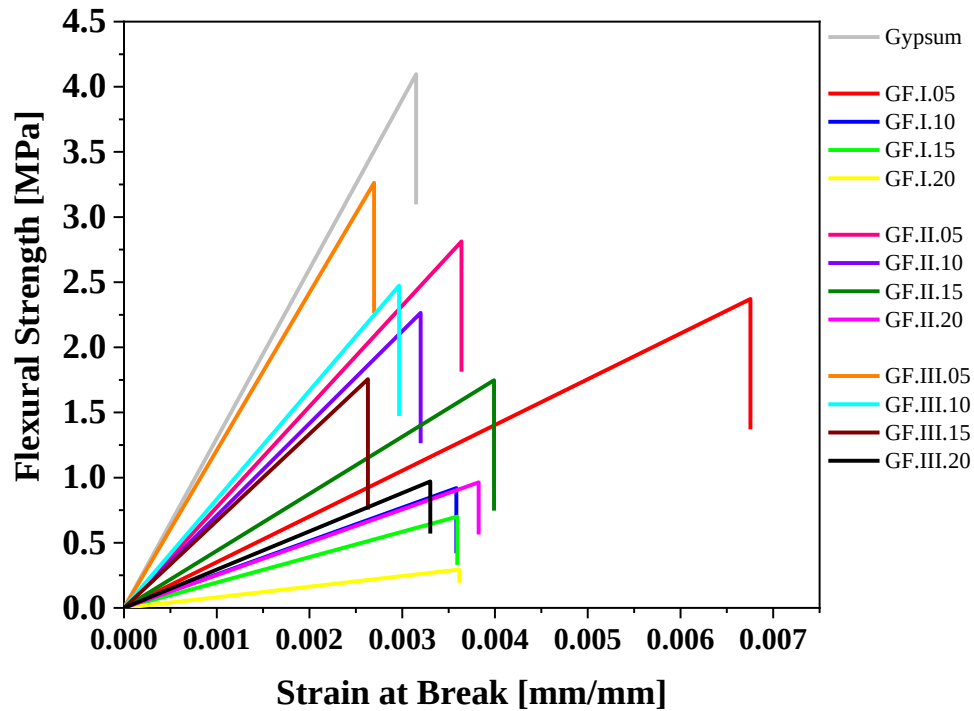


**Figure IV.02. Flexural Strength Results of Gypsum vs. Bio-Composite Materials (GF.s.m): Flexural Strength as a Function of Strain at Break.**

**Figure IV.02.** Show that pure gypsum, as a brittle material, exhibits consistent but relatively high flexural strength. In contrast, the bio-composite materials (GF.s.m) demonstrated weak performance as compared to gypsum, particularly when small-sized (s: I) Date Palm Fiber (DPF) and higher Date Palm Fiber (DPF) mass ratios (m: 15-20%) were used as compared to medium and larger sizes (s: II, and III) Date Palm Fibers (DPFs), respectively. Larger-sized (s: III) Date Palm Fibers (DPFs) increased strain at break tolerance, suggesting greater flexibility and toughness in bio-composite materials (GF.III.m). Bio-composite materials (GF.I.20, GF.II.20, GF.III.20) with high mass ratios (m: 20%) of Date Palm Fiber (DPF) showed minimum flexural strength across all data palm fiber sizes (s: I, II, and III). However, this improvement plateaued beyond certain mass ratios (m: 05-10%). The best performance was observed in bio-composites (GF.s.m) with larger-sized (s: III) Date Palm Fiber (DPF) and lower mass ratios (m: 05-10%). This indicates that both larger-sized (s: III) Date Palm Fiber (DPF) and low mass ratios (m: 05-10%) improve mechanical properties compared to small and medium sizes (s: I, and II) Date Palm Fiber (DPF), respectively.

Ultimately, these findings highlight the potential of date palm fiber-reinforced gypsum for applications requiring a balance of strength and flexibility. Further research is needed to optimize the fiber size and mass ratio for various uses.

**Figures IV.03.a, b, c, and d.** Shows the flexural strength and modulus results of gypsum compared to bio-composite materials (GF.s.m), as values of mechanical properties. The findings are presented as bar columns with corresponding number labels as follows:

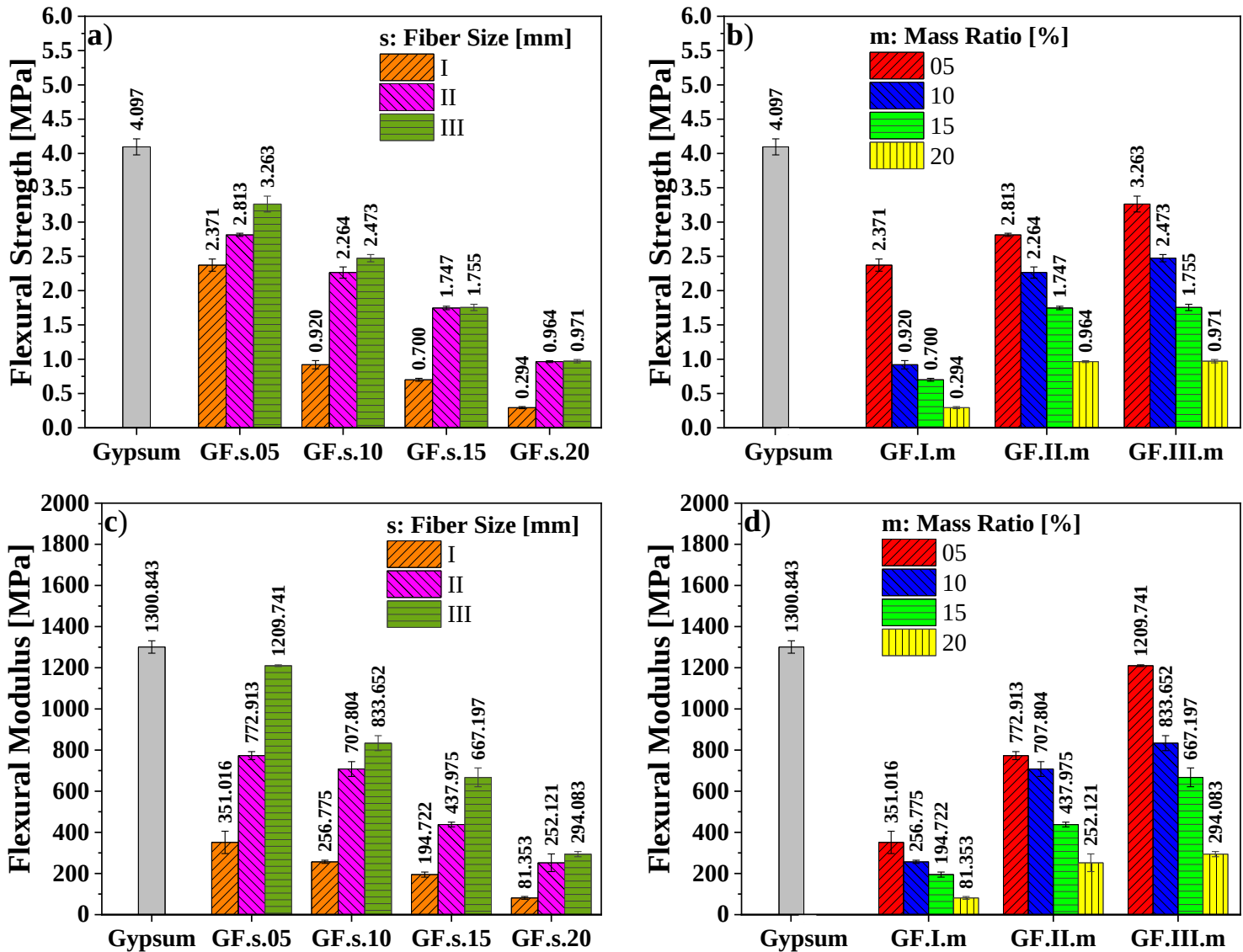


**Figure IV.03. Flexural Results of Gypsum vs. Bio-Composite Materials (GF.s.m): a, and b) Flexural Strength, c, and d) Flexural Modulus.**

**Figures IV.03.a, and c.** Shows that reinforcing gypsum with Date Palm Fibers (DPFs) to make bio-composite material (GF.s.m) reduces the flexural strength and modulus of the latter compared to

gypsum. The flexural strength and modulus values of gypsum were ( $4.097 \pm 0.117$  MPa) and ( $1300.843 \pm 30.256$  MPa), respectively, without Date Palm Fiber (DPF) reinforcement, as shown in **Table IV.01**. The flexural strength and modulus values of bio-composite material (GF.I.20) were the lowest, with values of ( $0.294 \pm 0.015$  MPa) and ( $81.353 \pm 6.685$  MPa), a decrease of 92.82 and 93.75%, respectively, compared to the gypsum specimen, as shown in **Table IV.01**. The bio-composite material (GF.III.05) also showed the highest flexural strength and modulus, with values of ( $3.263 \pm 0.115$  MPa) and ( $1209.741 \pm 4.131$  MPa), with a decrease of 20.36 and 7.00%, respectively, compared to the gypsum specimen, as shown in **Table IV.01**.

Furthermore, **Figures IV.03.a, and c**. Shows that when Date Palm Fiber (DPF) sizes (s: I-III) increase, the flexural strength and modulus of the bio-composite materials (GF.s.m) rise, concurrently with a drop in the mass ratio (m: 20-05%) of the reinforcing fibers. This is mainly because the Date Palm Fiber (DPF) of size (s: III) has a larger surface area that comes into contact with the gypsum matrix, which is confirmed by study [298]. In addition, this is confirmed by study [299], which is because the mass ratio has to be lower so that the structural integrity is not affected by the increased voids in the bio-composite materials (GF.s.m) that happen when fibers clump together. It was noted that the bio-composite material (GF.III.05) with larger (s: III) Date Palm Fibers (DPFs) exhibited flexural strength and modulus values of ( $3.263 \pm 0.115$  MPa) and ( $1209.741 \pm 4.131$  MPa) at a mass ratio of (m: 05%), which surpassed the values at a mass ratio of (m: 20%), recorded as ( $0.971 \pm 0.024$  MPa) and ( $294.083 \pm 12.583$  MPa), respectively, as shown in **Table IV.01**. A comparable tendency is noted for the bio-composite material (GF.s.m), which has smaller and medium (s: I, and II) Date Palm Fibers (DPFs), with a distinct preference for the latter.

Furthermore, **Figures IV.03.b, and d**. Shows that the lower mass ratio (m: 05%) of the bio-composite (GF.I.05) containing small Date Palm Fiber (DPF) size (s: I) exhibits higher flexural strength and modulus compared to the bio-composite (GF.I.m) containing higher mass ratios (m: 10-20%) of Date Palm Fibers (DPFs) added as gypsum reinforcement. The same applies to medium and larger (s: II and III) Date Palm Fibers (DPFs), with a clear advantage for the latter at a comparable mass ratio. As the mass ratio (m: 05-20%) of Date Palm Fibers (DPFs) added as gypsum reinforcement for the bio-composite (GF.s.m) increases, regardless of the Date Palm Fiber (DPF) size (s: I-III), the flexural strength and modulus reduce. This is confirmed by studies [300, 301], which indicate that increasing the mass ratios of reinforcing fibers weakens the integrity of the gypsum matrix, thus reducing the flexural strength and modulus of bio-composites. Furthermore, the flexural strength and modulus of fiber-



reinforced bio-composites (GF.s.m) are chiefly affected by fiber orientation, dispersion, and the quality of the fiber-matrix interface, which is confirmed by studies [300, 302].

Furthermore, **Figures IV.03.b, and d.** Shows that at a lower mass ratio (m: 05%) the bio-composite material (GF.I.05) with smaller-sized (s: I) Date Palm Fibers (DPFs) showed flexural strength and modulus of ( $2.371 \pm 0.089$  MPa) and ( $351.016 \pm 54.260$  MPa), respectively, which were higher than those of the bio-composite (GF.I.20) with a higher mass ratio (m: 20%), which were ( $0.294 \pm 0.015$  MPa) and ( $81.353 \pm 6.685$  MPa), respectively, as shown in **Table IV.01**. The decrease in flexural strength and modulus with increasing mass ratio (m: 05-20%) of Date Palm Fibers (DPFs) added as reinforcement can be attributed to insufficient bonding of the reinforcement Date Palm Fibers (DPFs) to the matrix due to the predominance of the former over the latter, leading to increased stress concentration points that are catalysts for failure, which is confirmed by studies [303, 304]. These results may also be consistent with studies [305-308], which indicate that fiber dispersion, matrix compatibility, and mass ratio of reinforcement are essential in influencing the mechanical performance of bio-composites, especially flexural strength and modulus.

Ultimately, these results show that larger size (s: III) of Date Palm Fibers (DPFs) can form eco-friendly panels made of bio-composite materials (GF.s.m) with lower mass ratios (m: 05-10%). Furthermore, date palm fiber-reinforced bio-composite materials (GF.s.m) with smaller (s: I) sizes and a higher mass ratio (m: 15-20%) should be avoided unless it is necessary to increase the flexural strength and modulus and save the amount of reinforcement Date Palm Fibers (DPFs).

#### IV.2.2. Compressive Strength Results

**Figure IV.04.** Shows the average compressive strength of five specimens as a function of compressive strain at break for gypsum compared to bio-composite materials (GF.s.m), as values of mechanical properties. The findings are presented as a line graph as follows:

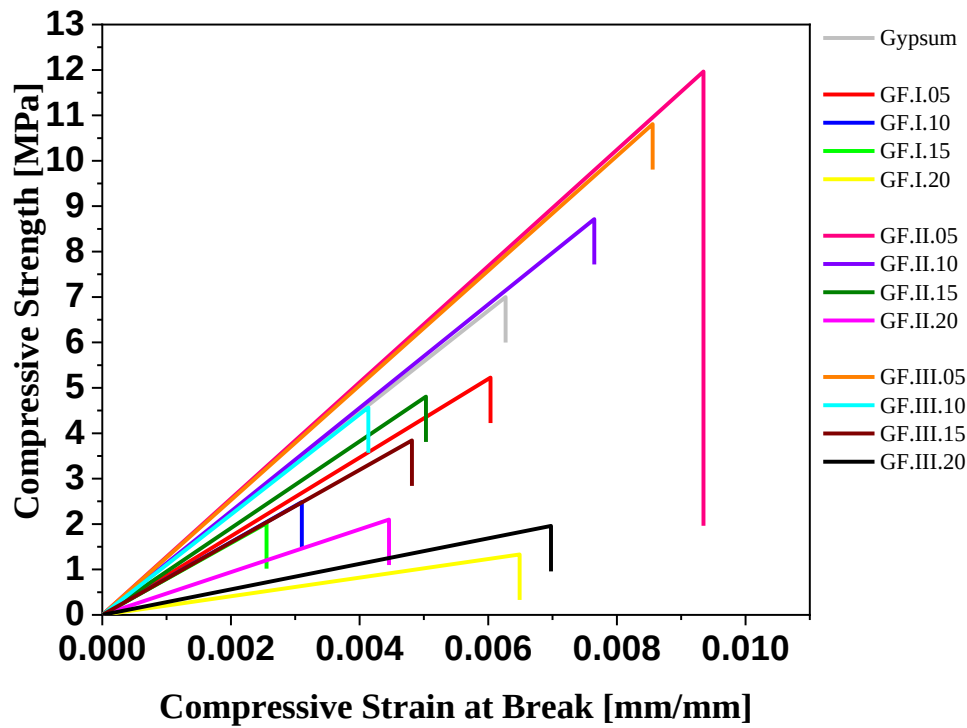


**Figure IV.04. Compressive Strength Results of Gypsum vs. Bio-Composite Materials (GF.s.m): Compressive Strength as a Function of Compressive Strain at Break.**

**Figure IV.04.** Show that the results of gypsum alone show typical brittle failure behavior, with compressive strength increasing up to a certain strain point before sharply dropping at failure. In contrast, bio-composite materials (GF.s.m) exhibit significant improvements in compressive strength as the Date Palm Fiber (DPF) mass ratio (m: 20-05%) decreases at Date Palm Fiber (DPF) sizes medium, larger, and smaller (s: II, III, and I), respectively. Bio-composite materials (GF.s.m) with smaller-sized (s: I) Date Palm Fibers (DPFs) and increasing mass ratios (m: 05-20%) show progressive failure in compressive strength, while those with medium and larger sizes (s: II, and III) Date Palm Fibers (DPFs), respectively, show even more pronounced improvements, particularly in specimens with low mass ratios (m: 05-10%).

Ultimately, the findings suggest that the inclusion of Date Palm Fibers (DPFs), especially at low mass ratios (m: 20-05%) and medium-sized (s: II) Date Palm Fiber (DPF), enhances the compressive strength of gypsum, making it a promising sustainable alternative for bio-composite materials. Optimized Date Palm Fiber (DPF) mass ratios and sizes can significantly improve the performance of these bio-composite materials (GF.s.m), offering potential applications in structural engineering.

Figures IV.05.a, b, c, and d. Shows the compressive strength and compressive modulus results of gypsum compared to bio-composite materials (GF.s.m), as values of mechanical properties. The findings are presented as bar columns with corresponding number labels as follows:

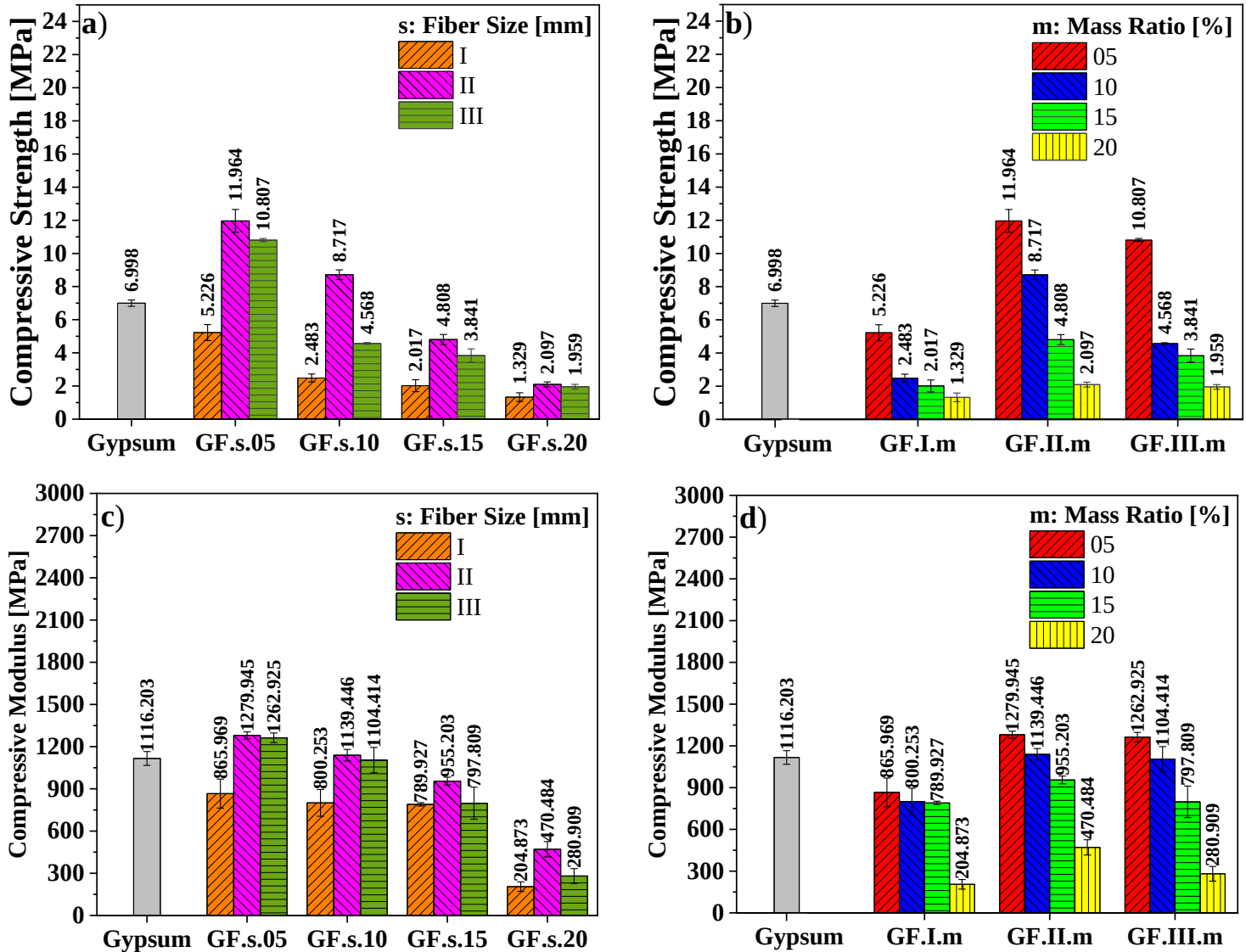


**Figure IV.05. Compressive Strength Results of Gypsum vs. Bio-Composite Materials (GF.s.m): a, and b) Compressive Strength, c, and d) Compressive Modulus.**

Figures IV.05.a, and c. Shows that the reinforcement of gypsum with Date Palm Fibers (DPFs) to create bio-composite materials (GF.s.m) may either diminish or enhance the compressive strength and compressive modulus of the latter compared to gypsum. The compressive strength and compressive modulus of gypsum without Date Palm Fiber (DPF) reinforcement were  $(6.998 \pm 0.191 \text{ MPa})$  and  $(1116.203 \pm 49.089 \text{ MPa})$ , respectively, as shown in Table IV.01. The bio-composite (GF.I.20) had the lowest compressive strength and compressive modulus at  $(1.329 \pm 0.261 \text{ MPa})$  and

(204.873  $\pm$  34.447 MPa), which is 81.01 and 81.65% lower than the gypsum specimen, respectively, as shown in **Table IV.01**. The bio-composite material (GF.II.05) had the highest compressive strength and compressive modulus, which was (11.964  $\pm$  0.687 MPa) and (1279.945  $\pm$  025.613 MPa), a 70.96 and 14.67% increase from the gypsum specimen, respectively, as shown in **Table IV.01**.

Furthermore, **Figures IV.05.a, and b**. Show that the bio-composite material (GF.s.m) reinforced with medium-sized (s: II) Date Palm Fibers (DPFs) exhibited superior compressive strength compared to those reinforced with small and large (s: I, and III) Date Palm Fibers (DPFs), demonstrating a distinct advantage for larger fibers over smaller fibers, at an equivalent mass ratio of added reinforcement fibers. The bio-composite materials (GF.II.05, GF.III.05, and GF.I.05) were reinforced with a mass ratio of (m: 05%) and included medium, large, and small (s: II, III, and I) sizes Date Palm Fiber (DPF), respectively. The resulting compressive strength values were (11.964  $\pm$  0.687, 10.807  $\pm$  0.097, and 5.226  $\pm$  0.480 MPa), respectively, as shown in **Table IV.01**. In addition, the bio-composite materials (GF.s.m) follow the same process when they are strengthened with mass ratios of (m: 10-20%). The clear advantage of medium (s: II) Date Palm Fibers (DPFs) over larger and smaller (s: III, and I) Date Palm Fibers (DPFs) is mainly because they can keep the fibers well dispersed and find the best balance with the gypsum matrix to get the best value of compressive strength, which is confirmed by study [309]. It also has the best aspect ratio, which lets this size fit perfectly into the gypsum matrix and gives it a higher compressive strength, which is confirmed by study [310]. In addition, these results may also be consistent with studies [311, 312], which confirmed that the larger fibers may cause gaps and holes in the gypsum matrix, which provides it with medium compressive strength. Simultaneously, the smaller fibers may exhibit poor adhesion to the gypsum matrix due to their limited surface area, achieving the lowest compressive strength, which is confirmed by studies [313, 314].

Furthermore, **Figures IV.05.c, and d**. Show that for the bio-composite materials (GF.s.m) with smaller-sized (s: I) Date Palm Fibers (DPFs), the compressive modulus decreases as the mass ratio (m: 05-20%) increases, with the most notable decline at a mass ratio (m: 20%), where the compressive modulus drops to (204.873  $\pm$  034.447 MPa), as shown in **Table IV.01**. The primary reason is that stress concentrations within the bio-composite materials (GF.s.m) are enhanced by matrix failure and discontinuity, resulting in a diminished compressive modulus, as confirmed by studies [315, 316]. Conversely, bio-composite materials (GF.s.m) with medium and larger sizes (s: II and III) made from Date Palm Fibers (DPFs) exhibit relatively better performance. The bio-composite material (GF.II.05) sample, for instance, maintains a compressive modulus of (1279.945  $\pm$  25.613 MPa), closer to that of pure gypsum, as shown

in **Table IV.01**. In addition, these results may be consistent with studies [259, 317], that confirmed that greater interaction between the gypsum matrix and Date Palm Fibers (DPFs) can be attributed to the increased surface area of the Date Palm Fibers (DPFs) in contact with the gypsum matrix to get higher compressive modulus values. Furthermore, studies [317, 318], confirm that the aggregation of Date Palm Fibers (DPFs) inside the gypsum matrix results in a deficiency in stress transmission. Consequently, it significantly increases the compressive modulus of bio-composite materials (GF.s.m). However, as the Date Palm Fiber (DPF) mass ratio (m: 05-20%) increases, the compressive modulus still declines, albeit more gradually compared to smaller-sized (s: I) Date Palm Fibers (DPFs).

Ultimately, the results suggest that enhancing bio-composite materials (GF.s.m) with medium and large (s: II, and III) Date Palm Fibers (DPFs) at mass ratios of (m: 05-10%) and avoiding mass ratios of (m: 15-20%) is beneficial for producing eco-friendly panels, especially those with high compressive strength and compressive modulus. It is advisable to refrain from using panels constructed from bio-composite materials (GF.s.m) reinforced with smaller (s: I) Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) unless the optimal compressive strength and compressive modulus of eco-friendly panels is of utmost importance.

### IV.2.3. Brinell Hardness Results

**Figures IV.06.a, and b.** Shows the Brinell hardness results of gypsum compared to bio-composite materials (GF.s.m), as values of mechanical properties. The findings are presented as bar columns with corresponding number labels as follows:

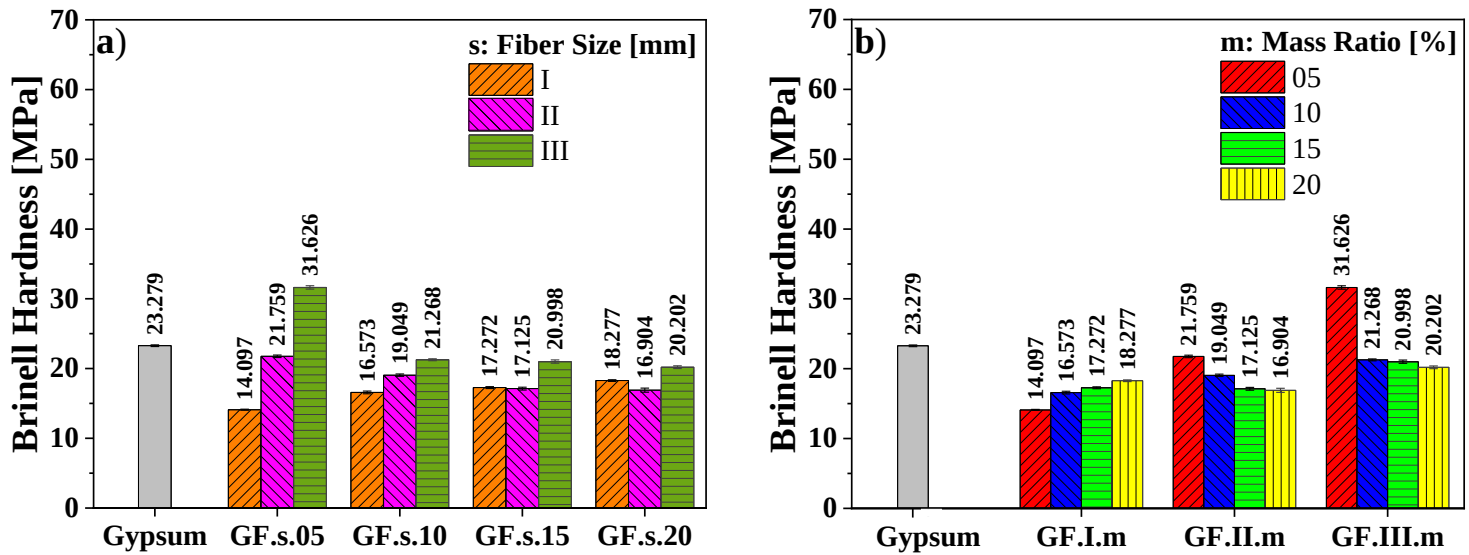


**Figure IV.06. Brinell Hardness Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.06.a, and b.** Show that the reinforcement of gypsum with Date Palm Fibers (DPFs) to create bio-composite materials (GF.s.m) diminishes the surface hardness of the latter compared to gypsum. The surface hardness value of gypsum devoid of Date Palm Fiber (DPFs) reinforcement was  $(23.279 \pm 0.126 \text{ MPa})$ , as shown in [Table IV.01](#). The bio-composite material (GF.I.05) had the lowest surface hardness value at  $(14.097 \pm 0.074 \text{ MPa})$ , which is 39.44% lower than the gypsum specimen, as shown in [Table IV.01](#). The bio-composite material (GF.III.05) had the hardest surface, measuring  $(31.626 \pm 0.245 \text{ MPa})$ , which is a 35.86% increase from the gypsum specimen, as shown in [Table IV.01](#).

Furthermore, [Figure IV.06.a](#), Show the influence of Date Palm Fiber (DPFs) size on bio-composite materials (GF.s.m), demonstrating that an increase in the size (s: I-III) of Date Palm Fibers (DPFs) is associated with improved surface hardness when reinforced with mass ratios ranging from (m: 05-10%). This is mainly because bio-composite materials (GF.s.m) are best at withstanding buckling stresses when their surface hardness is tested, which is confirmed by study [319]. Moreover, an increase in the size of (s: I-III) Date Palm Fibers (DPFs) correlates with an enhanced capacity of bio-composite materials (GF.s.m) to withstand local deformation and an increase in surface hardness, which is confirmed by studies [320, 321]. Nevertheless, when using mass ratios (m: 15-20%), however, the bio-composite materials (GF.s.m) that are reinforced with medium (s: II) size of Date Palm Fibers (DPFs) have a lower surface hardness than those that are reinforced with smaller and larger (s: I, and III) Date Palm Fibers (DPFs). In addition, these results may also be consistent with study [322], which confirmed that the ideal fiber size has a substantial impact on surface hardness at high mass ratios of the reinforcing fibers.

Furthermore, **Figure IV.06.b**. Show that when the mass ratio (m: 05-20%) of the reinforcing Date Palm Fibers (DPFs) increases, the surface hardness of the bio-composite materials (GF.s.m) reinforced with smaller (s: I) Date Palm Fibers (DPFs) also rises. This is primarily attributable to the favourable compatibility between the aspect ratio of the small (s: I) date palm fibre size, which enhances surface hardness as the mass ratio (m: 05-20%) increases, which is confirmed by studies [323, 324]. Conversely, the bio-composite materials (GF.s.m) reinforced with medium and large (s: II and III) Date Palm Fibers (DPFs) exhibited behaviour contrary to that of the smaller (s: I) Date Palm Fibers (DPFs). This is mostly attributable to the dispersion of Date Palm Fibers (DPFs) and their inadequate adhesion to the gypsum matrix, resulting from the elevated mass ratio (m: 05-20%) of the reinforcement fibers to get low Brinell surface hardness, which is confirmed by studies [309, 325]. In addition, the mass ratios (m: 05-20%) of Date Palm Fibers (DPFs) correlates with a rise in the number of voids in the bio-composite materials (GF.s.m), and thus obtaining low Brinell surface hardness. The primary cause may stem from the disturbance of the load transfer mechanism inside the gypsum matrix, and thus obtaining low Brinell surface hardness is attributed to the increased number of voids in the bio-composite material (GF.s.m) when the increase in the mass ratios (m: 05-20%) with medium and large (s: II and III) Date Palm Fibers (DPFs), which is confirmed by studies [326, 327].

Ultimately, it can be concluded that the best bio-composite materials (GF.s.m) with a superior surface hardness that can be used in the production of eco-friendly panels for structural constructions should be reinforced with larger (s: III) Date Palm Fibers (DPFs) with a lower mass ratio (m: 05%). Meanwhile, other bio-composite materials (GF.s.m) should be avoided unless the surface hardness of eco-friendly panels is given great importance.

#### IV.2.4. Charpy Impact Strength Results

**Figures IV.07.a, and b**. Shows the Charpy impact strength results of gypsum compared to bio-composite materials (GF.s.m), as values of mechanical properties. The findings are presented as bar columns with corresponding number labels as follows:



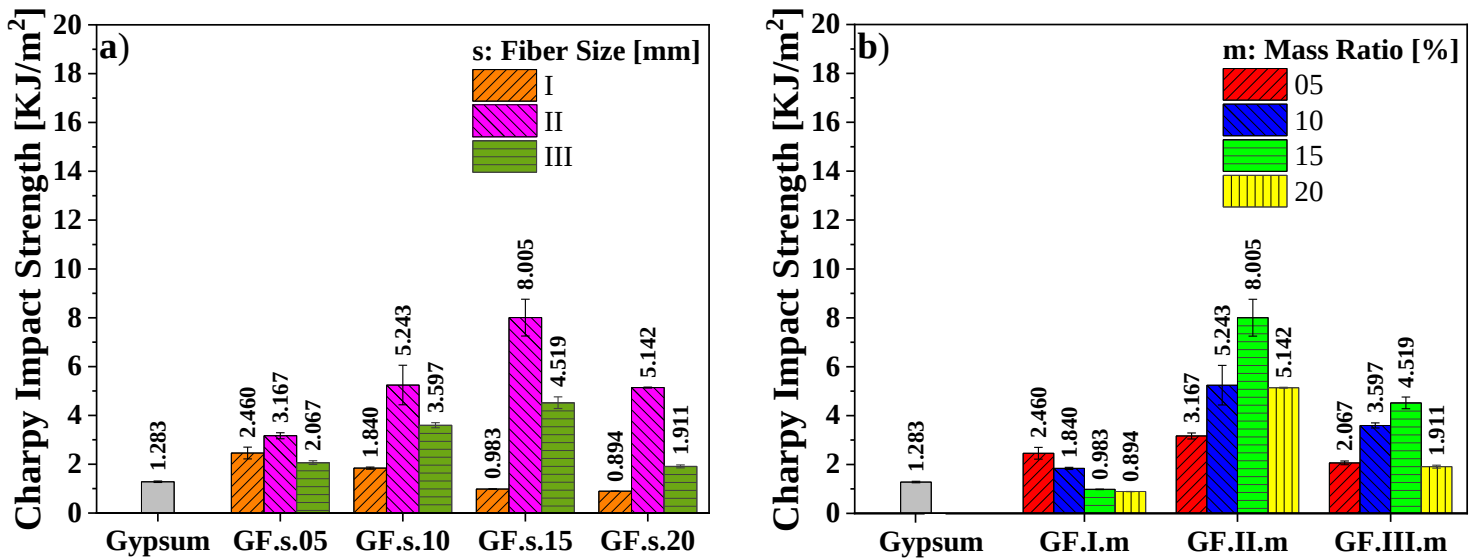


**Figure IV.07. Charpy Impact Strength Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.07.a, and b.** Shows that reinforcing gypsum with Date Palm Fibers (DPFs) to create bio-composite materials (GF.s.m) enhances the impact strength capacity of the materials compared to gypsum. The impact strength value of gypsum devoid of Date Palm Fiber (DPF) reinforcement was  $(1.283 \pm 0.029 \text{ KJ/m}^2)$ , as shown in **Table IV.01**. This is mostly attributable to the fragile characteristics of gypsum, which is confirmed by studies [328-330]. The bio-composite material (GF.I.20) had the lowest impact strength value, which was  $(0.894 \pm 0.002 \text{ KJ/m}^2)$ , a drop of 30.32% from the gypsum specimen, as shown in **Table IV.01**. The bio-composite material (GF.II.15) had the highest impact strength at  $(8.005 \pm 0.756 \text{ KJ/m}^2)$ , which is 523.93% more than the gypsum specimen, as shown in **Table IV.01**.

Furthermore, **Figure IV.07.a.** Show the influence of Date Palm Fiber (DPF) size on bio-composite materials (GF.s.m), indicating that with a reinforcement medium size (s: II) of Date Palm Fibers (DPFs), the impact strength is maximized for materials reinforced with increased mass ratios (m: 05-20%). Nevertheless, bio-composites (GF.s.m) show optimum impact strength values when reinforced with medium, large, and smaller (s: II, III and I) Date Palm Fibers (DPFs), respectively, with a mass fraction between (m: 10-15%). Nevertheless, the medium (s: II) Date Palm Fiber (DPF) size is optimal for dispersing impact strength due to its effective aggregation in bio-composite materials (GF.s.m), which is confirmed by studies [331, 332]. It also made the fracture deflection mechanism better since it spread out well, which is confirmed by studies [333, 334]. In addition, these results may also be consistent with study [335], which confirmed that the smaller fibers clumped together in a way that was opposite to what



was expected, which made the impact strength the weakest for bio-composite materials. Nevertheless, the larger (s: III) Date Palm Fibers (DPFs) exhibited a practically negative agglomeration effect, resulting in higher voids within the bio-composite materials (GF.s.m) and thus yielding the lowest impact strength relative to the medium-sized (s: II) Date Palm Fibers (DPFs).

Furthermore, **Figure IV.07.b**. Show the influence of mass ratios (m: 05-20%) on bio-composite materials (GF.s.m), indicating that impact strength diminishes when these materials are reinforced with a smaller (s: I) size of Date Palm Fiber (DPF). This indicates that the material's impact strength is significantly correlated with the dimensions of the reinforcing fibers, which is confirmed by studies [332, 336]. Conversely, when reinforced with medium or larger (s: II, and III) Date Palm Fibers (DPFs), the impact strength escalates at mass ratios (m: 05-15%) and thereafter diminishes at mass ratios (m: 20%). Moreover, this is mostly due to the reinforcement with certain fiber sizes demonstrating their impact strength at particular mass ratios, which is confirmed by study [337]. In addition, the high mass ratio of reinforcing fibers adversely impacts the material's capacity to absorb impact strength. Nevertheless, an excessive mass ratio might lead to flaws or agglomerations in the bio-composite materials (GF.s.m), which is confirmed by study [338].

Ultimately, the analysis of the results indicates that to produce environmentally sustainable panels from bio-composite materials (GF.s.m), reinforcement should be applied at mass ratios of (m: 05-15%) and utilize the medium or large (s: II and III) Date Palm Fibers (DPFs), with a distinct preference for size (s: II), to achieve optimal shock and impact strength in the panels. Simultaneously, it is feasible to utilize bio-composite materials (GF.s.m) reinforced with mass ratios ranging from (m: 05-10%) and smaller (s: I) Date Palm Fibers (DPFs) to produce ecologically friendly panels that exhibit impact strength and cracking, but not optimally.

IV.3. General Mechanical Results

**Table IV.01.** Shows the mechanical results for gypsum-based bio-composite materials (GF.s.m) reinforced with Date Palm Fibers (DPFs), clarifying the enhancements and trade-offs in mechanical properties related to varying sizes (s: I-III) of Date Palm Fibers (DPFs) and mass ratios (m: 05-20%) for each size. The mechanical properties include flexural strength ( $\sigma_{3P}$ ), flexural modulus ( $E_{3P}$ ), compressive strength ( $f_m$ ), compressive modulus ( $E_m$ ) Brinell hardness (HB), and Charpy impact strength (G).

**Table IV.01. The Mechanical Results of Bio-composite vs. Gypsum.**

| Code      | Fiber Size | Mass Ratio | Flexural Strength | Ratio   | Flexural Modulus  | Ratio   | Compressive Strength | Ratio   | Compressive Modulus | Ratio   | Brinell Hardness | Ratio   | Charpy Impact Strength | Ratio    |
|-----------|------------|------------|-------------------|---------|-------------------|---------|----------------------|---------|---------------------|---------|------------------|---------|------------------------|----------|
| GF.s.m    | s          | m          | $\sigma_{3P}$     | -       | $E_{3P}$          | -       | $f_m$                | -       | $E_m$               | -       | HB               | -       | G                      | -        |
| Unit      | [mm]       | [%]        | [MPa]             | [%]     | [MPa]             | [%]     | [MPa]                | [%]     | [MPa]               | [%]     | [MPa]            | [%]     | [KJ/m²]                | [%]      |
| Gypsum    | -          | -          | 4.097 ± 0.117     | — 00.00 | 1300.843 ± 30.256 | — 00.00 | 06.998 ± 0.191       | — 00.00 | 1116.203 ± 049.089  | — 00.00 | 23.279 ± 0.126   | — 00.00 | 1.283 ± 0.029          | — 000.00 |
| GF.I.05   | I          | 05         | 2.371 ± 0.089     | ▼ 42.13 | 0351.016 ± 54.260 | ▼ 73.02 | 05.226 ± 0.480       | ▼ 25.32 | 0865.969 ± 103.048  | ▼ 22.42 | 14.097 ± 0.074   | ▼ 39.44 | 2.460 ± 0.242          | ▲ 091.74 |
| GF.I.10   |            | 10         | 0.920 ± 0.060     | ▼ 77.54 | 0256.775 ± 07.456 | ▼ 80.26 | 02.483 ± 0.244       | ▼ 64.52 | 0800.253 ± 095.284  | ▼ 28.31 | 16.573 ± 0.196   | ▼ 28.81 | 1.840 ± 0.043          | ▲ 043.41 |
| GF.I.15   |            | 15         | 0.700 ± 0.022     | ▼ 82.91 | 0194.772 ± 12.396 | ▼ 85.03 | 02.017 ± 0.362       | ▼ 71.18 | 0789.927 ± 012.107  | ▼ 29.23 | 17.272 ± 0.135   | ▼ 25.80 | 0.983 ± 0.008          | ▼ 023.38 |
| GF.I.20   |            | 20         | 0.294 ± 0.015     | ▼ 92.82 | 0081.353 ± 06.685 | ▼ 93.75 | 01.329 ± 0.261       | ▼ 81.01 | 0204.873 ± 034.447  | ▼ 81.65 | 18.277 ± 0.120   | ▼ 21.49 | 0.894 ± 0.002          | ▼ 030.32 |
| GF.II.05  | II         | 05         | 2.813 ± 0.024     | ▼ 31.34 | 0772.913 ± 19.397 | ▼ 40.58 | 11.964 ± 0.687       | ▲ 70.96 | 1279.945 ± 025.613  | ▲ 14.67 | 21.759 ± 0.184   | ▼ 06.53 | 3.167 ± 0.123          | ▲ 146.84 |
| GF.II.10  |            | 10         | 2.264 ± 0.082     | ▼ 44.74 | 0707.804 ± 36.019 | ▼ 45.59 | 08.717 ± 0.288       | ▲ 24.56 | 1139.446 ± 040.909  | ▲ 02.08 | 19.049 ± 0.172   | ▼ 18.17 | 5.243 ± 0.809          | ▲ 308.65 |
| GF.II.15  |            | 15         | 1.747 ± 0.025     | ▼ 57.36 | 0437.975 ± 11.588 | ▼ 66.33 | 04.808 ± 0.300       | ▼ 31.29 | 0955.203 ± 027.865  | ▼ 14.42 | 17.125 ± 0.208   | ▼ 26.44 | 8.005 ± 0.756          | ▲ 523.93 |
| GF.II.20  |            | 20         | 0.964 ± 0.014     | ▼ 76.47 | 0252.121 ± 42.973 | ▼ 80.62 | 02.097 ± 0.147       | ▼ 70.03 | 0470.484 ± 054.829  | ▼ 57.85 | 16.904 ± 0.297   | ▼ 27.39 | 5.142 ± 0.019          | ▲ 300.78 |
| GF.III.05 | III        | 05         | 3.263 ± 0.115     | ▼ 20.36 | 1209.741 ± 04.131 | ▼ 07.00 | 10.807 ± 0.097       | ▲ 54.43 | 1262.925 ± 033.946  | ▲ 13.14 | 31.626 ± 0.245   | ▲ 35.86 | 2.067 ± 0.077          | ▲ 061.11 |
| GF.III.10 |            | 10         | 2.473 ± 0.053     | ▼ 39.64 | 0833.652 ± 36.417 | ▼ 35.91 | 04.568 ± 0.051       | ▼ 34.72 | 1104.414 ± 090.396  | ▼ 01.06 | 21.268 ± 0.138   | ▼ 08.64 | 3.597 ± 0.106          | ▲ 180.36 |
| GF.III.15 |            | 15         | 1.755 ± 0.046     | ▼ 57.16 | 0667.197 ± 45.744 | ▼ 48.71 | 03.841 ± 0.397       | ▼ 45.11 | 0797.809 ± 113.863  | ▼ 28.52 | 20.998 ± 0.233   | ▼ 09.80 | 4.519 ± 0.239          | ▲ 252.22 |
| GF.III.20 |            | 20         | 0.971 ± 0.024     | ▼ 76.30 | 0294.083 ± 12.583 | ▼ 77.39 | 01.959 ± 0.142       | ▼ 72.01 | 0280.909 ± 052.760  | ▼ 74.83 | 20.202 ± 0.196   | ▼ 13.22 | 1.911 ± 0.062          | ▲ 048.95 |

Where:

- G

: Gypsum;
- F

: Date Palm Fiber (DPF);
- GF.s.m

: The Bio-composite Material (GF.s.m) Made From Gypsum (G) Reinforced with Different Sizes (s: I, II, and III) and Mass Ratios (m: 05, 10, 15, and 20%) for each Size of Date Palm Fibre (F);
- The Symbol ▲

: The Ratio of the Value Increase of Bio-composite vs. Gypsum [%];
- The Symbol ▼

: The Ratio of the Value Decrease of Bio-composite vs. Gypsum [%];
- The Symbol —

: The Ratio of the Value Increase and Decrease of Bio-composite vs. Gypsum [%];

The results of the **Table IV.01.** were discussed as follows:

1.

**Flexural Strength ( $\sigma_{3P}$ ):** The pure gypsum matrix has the maximum flexural strength (4.097 ± 0.117 MPa), which means that unreinforced gypsum stays strong when it flexes. The flexural strength drops a lot when the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) increases for all sizes (s: I-III). This tendency is stronger when the mass ratios are larger (m: 15-20%), which means that too high a Date Palm Fiber (DPF) mass ratio makes it harder for the matrix to transmit loads, which is confirmed by studies [338, 339]. In date palm size (s: I), as the fiber mass ratio (m: 05-20%)

increased, the flexural strength of the bio-composite materials (GF.I.m) decreased until it reached a value of ( $0.294 \pm 0.015$  MPa). The decrease in flexural strength at high Date Palm Fiber (DPF) mass ratios (m: 15-20%) may be due to poor dispersion and aggregation of fibers, leading to stress concentrations, as confirmed by studies [338, 340]. Furthermore, for medium-sized (s: II) Date Palm Fibers (DPFs), the flexural strength stays moderate to a mass ratio of (m: 05-10%), but drops off beyond that. In addition, for Date Palm Fiber (DPFs) size (s: III), the findings are in the middle, with bio-composite material (GF.III.05) getting ( $3.263 \pm 0.115$  MPa) higher than the same (GF.I.05) and (GF.II.05) specimens. This is in line with what prior studies have shown [96, 341], which indicate that the right contact between fibers and the matrix is necessary for flexural function. In addition, this is confirmed by studies [320, 342], which indicate that The increased flexural strength in larger size (s: III) fiber specimens may be a result of increased bonding caused by increased surface roughness or better alignment;

**2. Flexural Modulus ( $E_{3P}$ ):** The flexural modulus behaves similarly to flexural strength, with pure gypsum exhibiting the maximum value ( $1300.843 \pm 30.256$  MPa). As for the size (s: I), the decrease is the most severe, as the bio-composite materials (GF.I.m) recorded in mass ratios (m: 05-20%) with a decrease value that reached ( $81.353 \pm 0.685$  MPa). This reveals that the flexural modulus of bio-composite materials (GF.I.m) has dropped a lot. Furthermore, the bio-composite materials (GF.II.m) have a considerable flexural modulus up to a fiber mass ratio (m: 05–10%), but beyond that, it drops quickly, with values of ( $351.016 \pm 54.260$ - $256.775 \pm 07.456$  MPa), respectively. Furthermore, the bio-composite materials (GF.III.m) are better than (GF.I.m) and (GF.II.m) when the mass ratios are low (m: 05-10%). In addition, this means that larger-sized (s: III) Date Palm Fibers (DPFs) are better at holding up loads in these instances, as confirmed by studies [320, 343]. In addition, this is confirmed by studies [333, 344], which indicate that the preservation of modulus depends heavily on fiber dispersion and interfacial adhesion. Research indicates that coarse fibers might create micro-cracks or voids, hence diminishing flexural modulus, especially under elevated loads [345-347]:

**3. Compressive Strength and Compressive Modulus ( $f_m$ , and  $E_m$ ):** The addition of Date Palm Fibers (DPFs) increases compressive strength and compressive modulus moderately compared to pure gypsum ( $6.998 \pm 0.191$ - $1116.203 \pm 49.089$  MPa), respectively, with bio-composite materials (GF.II.05) and (GF.III.05) with values of ( $11.964 \pm 0.687$ - $1279.945 \pm 25.613$  and  $10.807 \pm 0.097$ - $1262.925 \pm 033.946$  MPa), respectively, showing notable enhancements. Furthermore, when the mass ratio of Date Palm Fiber (DPFs) is higher (m: 15-20%), the compressive strength and compressive modulus of all sizes (s: I–III) of Date Palm Fiber (DPF) go down. In addition, the fiber reinforcement improves compressive characteristics by bridging cracks, as shown by investigations on bio-composites [348, 349]. However, too much fiber loading creates gaps, which function as stress points, as confirmed by study [338];

**4. Brinell Hardness (HB):** The bio-composite material (GF.III.m) specimens are much harder than the other bio-composite materials (GF.I.m and GF.II.m), with (GF.III.05) getting ( $31.626 \pm 0.245$  MPa). Furthermore, the bio-composite material (GF.III.m) is very hard, which is in line with what previous studies have shown about larger fibers [37, 350]. In addition, these studies say that larger fibers may make the surface more resistant to localized deformation, but they may also make other mechanical properties worse [59, 351]. In all bio-composite materials (GF.s.m), increasing the fiber mass ratio (m: 05-20%) usually makes the hardness less. In addition, this is confirmed by studies [338, 352], which indicate that this is probably because the matrix is diluted and the tension is not evenly spread out;

**5. Charpy Impact Strength (G):** The impact strength increases with the Date Palm Fiber (DPF) mass ratio up to (m: 15%) for the bio-composite materials (GF.II.m), reaching a maximum of ( $8.005 \pm 0.756$  KJ/m<sup>2</sup>). This suggests that the bio-composite material (GF.II.15) can absorb more energy for optimal fiber aggregation in the matrix, as confirmed by studies [353, 354]. Furthermore, the decrease at larger mass ratios (e.g., The Bio-Composite Materials (GF.II.20)) may be due to fibers clustering together, which makes stress redistribution less efficient, as confirmed by studies [355, 356]. In addition, the trend for impact strength for the bio-composite materials (GF.III.m) is inconsistent, although it is always greater than for the bio-composite materials (GF.I.m) across all mass ratios (m: 05-20%). In addition, this is confirmed by studies [357, 358], which indicate that which showed that larger-sized fiber-reinforced composites dissipate energy better because of fiber pullout processes and fracture deflection.

Ultimately, the mechanical results of bio-composite materials (GF.s.m) made from gypsum and Date Palm Fibers (DPFs) depend a lot on the size (s: I-III) of the fibers, the mass ratio (m: 05-20%), and how the Date Palm Fibers (DPFs) and matrix interact with each other. When it comes to flexural strength, flexural modulus, compressive strength, Brinell hardness, and Charpy impact strength, smaller-sized (s: I) Date Palm Fibers (DPFs) work less effectively, but larger fiber sizes (s: II-III) work better. These properties are the same at (m: 05-10%) Date Palm Fiber (DPF) loading. In addition, the higher mass ratios (m: 15-20%) are less beneficial.

## IV.4. Physical Results

The results obtained from [Table IV.02](#). are discussed as physical properties related to the bio-composite material specimens (GF.s.m), including different sizes (s: I-III) and mass ratios (m: 05-20%) of each size of palm fiber compared to the gypsum specimen, as follows:

### IV.4.1. Bulk Density Results

[Figures IV.08.a, and b](#). Shows the bulk density results of gypsum compared to bio-composite materials (GF.s.m), as values of physical properties. The results are presented as bar columns with number labels as follows:

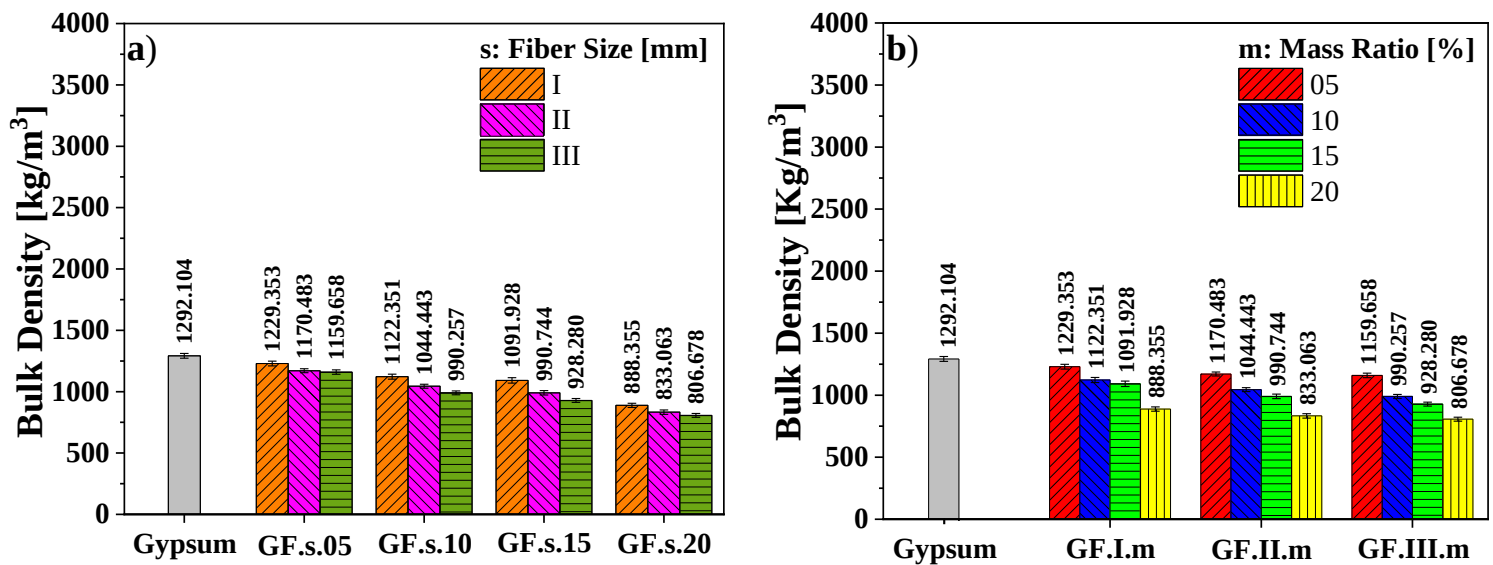


**Figure IV.08. Bulk Density Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

[Figures IV.08.a, and b](#). Shows that the specimens of bio-composite materials (GF.s.m), composed of a gypsum matrix and palm fiber as reinforcement, have a lower bulk density than the gypsum specimens. The gypsum specimen has a bulk density of  $(1292.104 \pm 19.413 \text{ Kg/m}^3)$ , the highest value documented among the analyzed specimens, as shown in [Table IV.02](#). The lowest and highest bulk density values were recorded for the two bio-composite materials (GF.III.20 and GF.I.05), which are reinforced with the larger and smaller (s: III and, I) Date Palm Fibers (DPFs), at mass ratios of 20 and 05%, yielding values of  $(806.678 \pm 16.085, \text{ and } 1229.353 \pm 19.154 \text{ Kg/m}^3)$ , respectively, in comparison to the gypsum specimen, as shown in [Table IV.02](#). The percentage reductions of bulk density in the two bio-composite

materials specimens, (GF.III.20 and GF.I.05) were 37.57 and 4.86%, respectively, compared to the gypsum specimen, as shown in [Table IV.02](#).

Furthermore, [Figure IV.08.a](#). Shows that the bio-composite materials (GF.s.m) with smaller-sized (s: I) Date Palm Fibers (DPFs) have greater bulk density than those that have medium and larger (s: II and III) sizes. The same trend was observed at all different mass ratios (m: 05-20%), indicating better packing and fewer voids with a better structural framework, allowing for a higher bulk density, which is confirmed by study [359]. The larger (s: III) sizes of Date Palm Fibers (DPFs) also tend to disperse less homogeneously, leading to increased porosity and lower bulk density, which is confirmed by studies [11, 360]. The bio-composite materials (GF.II.m), especially with medium (s: II) size Date Palm Fibers (DPFs), maintain a relatively medium bulk density due to better packing efficiency and optimal void distribution, which is confirmed by studies [3, 17, 361]. The bio-composites (GF.II.m) with medium (s: II) size of Date Palm Fiber (DPF) and fiber mass ratios ranging from (m: 05-10%) are ideal for applications requiring moderate density with improved strength and impact resistance, which is confirmed by study [3]. In addition, these results may also be consistent with studies [362, 363], which confirmed that the lightweight construction applications, bio-composite materials (GF.III.m) with larger (s: III) Date Palm Fiber (DPF) sizes, and higher fiber mass ratios (m: 15-20%) may be preferred due to their lower bulk density.

Furthermore, [Figure IV.08.b](#). Shows the effect of the mass ratios of Date Palm Fiber (DPF) added as reinforcement in bio-composite materials (GF.s.m). Increasing the Date Palm Fiber (DPFs) mass ratios (m: 05-20%) consistently reduces the bulk density for all Date Palm Fiber (DPF) sizes (s: I-III). The bulk density of the bio-composite (GF.I.05) reinforced with the smaller (s: I) Date Palm Fibers (DPFs) decreases from  $(1229.353 \pm 19.154 \text{ kg/m}^3)$  at a mass ratio of (m: 05%) to  $(888.355 \pm 17.492 \text{ kg/m}^3)$  at a mass ratio of (m: 20%). Similar trends were observed for the bio-composites (GF.II.m and GF.III.m). This is primarily due to a decrease in bulk density with fiber addition due to the lower density of the Date Palm Fibers (DPFs) compared to the gypsum matrix, which is confirmed by studies [258, 364]. Fibers also introduce porosity and voids, reducing the overall bulk density of the composite, which is confirmed by studies [327, 365]. At low Date Palm Fiber (DPF) mass ratios (m: 05-10%), the bulk density decrease is moderate because the matrix continuity is maintained, which is confirmed by studies [366, 367]. This breaks the integrity of the matrix, which leads to a big drop in bulk density, as shown by studies [366, 368]. Across all sizes of Date Palm Fiber (DPF) studied, bulk density drops sharply at (m: 20%) of the fiber mass ratio added as reinforcement to bio-composite materials (GF.s.m). In addition, these results

may also be consistent with studies [369, 370], which confirmed that this suggests that excessive fiber addition leads to significant porosity and potential aggregation, which reduces bulk density. Study also show that higher fiber mass fractions introduce structural discontinuities, increasing porosity and reducing density [338]. Research shows that fiber mass ratios (m: 05-15%) maximize density and mechanical performance, improving bulk density retention in (GF.II.05 and GF.III.05) bio-composite materials [371]. The fiber ratios over (m: 15%) reduce bulk density, which may affect mechanical performance and weight support, which is confirmed by studies [371, 372].

Ultimately, the findings for bulk density show how well Date Palm Fiber (DPF) reinforcement and matrix integrity work together in gypsum-based composites. The bio-composite materials (GF.III.m) with a mass ratio of (m: 05-10%) fiber content strike the best balance because they maintain a higher bulk density while still benefiting from Date Palm Fiber (DPF) reinforcement. Excessive Date Palm Fiber (DPF) mass ratios or larger (s: III) Date Palm Fibers (DPFs) result in a significant decrease in density, which points to the importance of optimized Date Palm Fiber (DPF) size and mass ratios for specific applications. It can be concluded that bio-composite materials (GF.s.m) specimens show low bulk density compared to gypsum specimens and are highly effective for environmentally sustainable construction.

#### IV.4.2. Water Absorption Results

**Figures IV.09.a, and b.** Shows the water absorption results of gypsum compared to bio-composite materials (GF.s.m), as values of physical properties. The results are presented as bar columns with number labels as follows:

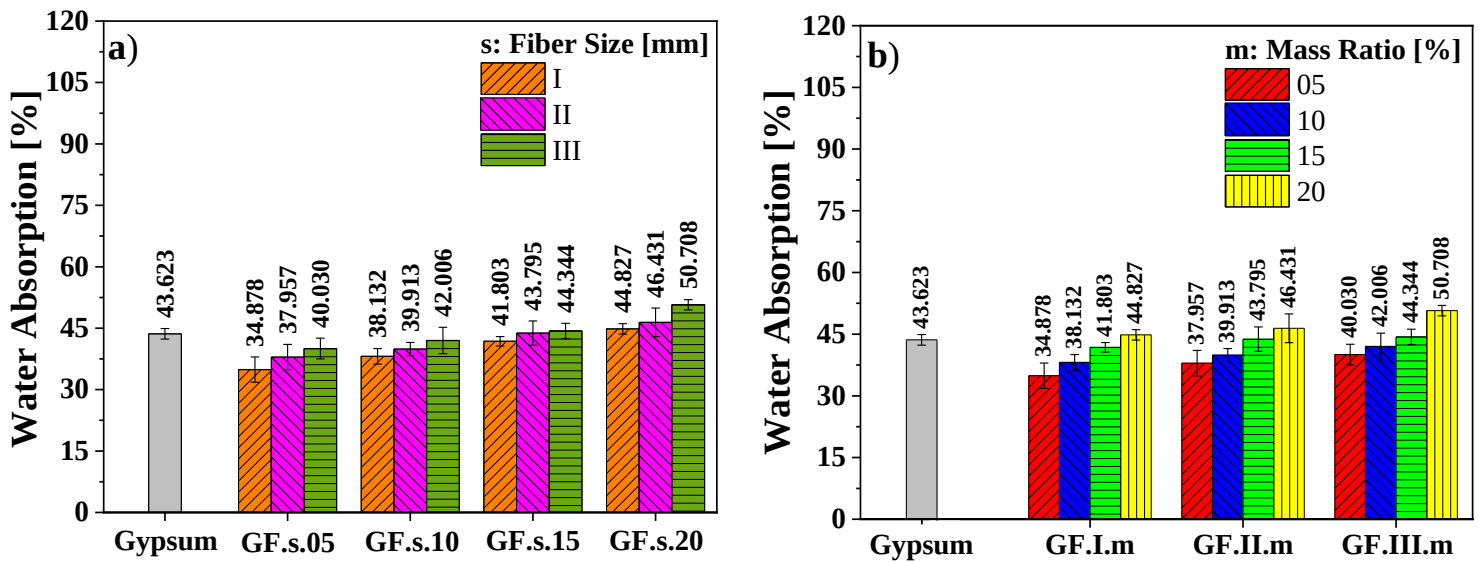


**Figure IV.09. Water Absorption Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.09.a, and b.** Shows that incorporating Date Palm Fibers (DPFs) into gypsum to produce bio-composite materials (GF.s.m) reduces the ratio of water absorption in some specimens compared to the standard gypsum specimen. The gypsum specimen attained a water absorption ratio of  $(43.623 \pm 1.295 \%)$ , as shown in **Table IV.02**. The lowest and highest water absorption ratio values achieved by the bio-composite materials (GF.s.m) specimens were observed in the bio-composite materials (GF.I.05 and GF.III.20), which incorporate the smaller and larger (s: I, III) Date Palm Fiber (DPF) with mass ratios of 05 and 20%, yielding values of  $(34.878 \pm 3.082, \text{ and } 50.708 \pm 1.267 \%)$ , respectively, in comparison to the gypsum specimen, as shown in **Table IV.02**. The two bio-composite materials (GF.I.05 and GF.III.20) exhibited a reduction of 20.05% and an increase of 16.24% in water absorption ratio, respectively, compared to the gypsum specimen, as shown in **Table IV.02**.

Furthermore, **Figure IV.09.a.** Shows that, for the same mass ratios, smaller and medium-sized (s: I, and II) Date Palm Fibers (DPFs) tend to absorb less water than larger-sized (s: III) Date Palm Fibers (DPFs). Research shows that natural fibers make composites absorb more water because they are hydrophilic [373-375]. When the mass ratios increase, larger (s: III) size Date Palm Fibers (DPFs) tend to create more voids in the gypsum matrix, leading to a greater water absorption ratio, as confirmed by studies [11, 365, 373]. For example, the bio-composite material (GF.III.20) had values of  $(50.708 \pm 1.267 \%)$ , a 16.24% increase compared to the gypsum specimen, as shown in **Table IV.02**. The smaller (s: I) size Date Palm Fibers (DPFs) also disperse more effectively within the gypsum matrix, reducing voids and improving water resistance at lower fiber mass ratios added as reinforcement. The



larger (s: III) size Date Palm Fibers (DPFs), on the other hand, tend to create localized defects and higher porosity, especially at higher mass ratios, leading to an increased water absorption ratio, as confirmed by studies [376, 377]. The bio-composites (GF.I.05 and GF.II.05) exhibit lower water absorption ( $34.878 \pm 3.082$  and  $37.957 \pm 3.092$  %, respectively) compared to pure gypsum, as shown in **Table IV.02**. This also indicates the beneficial effects of fiber reinforcement at lower mass ratios, as confirmed by studies [378, 379].

Furthermore, bio-composite materials (GF.II.m) that are reinforced with medium-sized (s: II) Date Palm Fibers (DPFs) show very even water absorption across fiber mass ratios. This is probably because the fibers are the right size for reinforcing without creating too many voids, as confirmed by studies [322, 337]. The bio-composites (GF.s.m) with smaller (s: I) or medium (s: II) size Date Palm Fibers (DPFs) and mass ratios of (m: 05-10%) are better for uses that do not need to absorb much water.

Furthermore, **Figure IV.09.b**. Shows that adding Date Palm Fiber (DPF) to gypsum-based bio-composites (GF.s.m) usually makes them absorb less water when the mass ratio of Date Palm Fiber (DPF) is low (m: 05-10%). Still, it makes them absorb more water when the mass ratio of Date Palm Fiber (DPF) is high (m: 15-20%). When the mass ratio of Date Palm Fiber (DPF) is low (m: 05-10%), the water absorption ratio is lower than with pure gypsum because the matrix fills better and the porosity is lower, as confirmed by study [15]. The amount of water that can be absorbed increases significantly when the mass ratio of Date Palm Fiber (DPF) is high (m: 15-20%), as confirmed by studies [174, 380]. This is probably because the Date Palm Fibers (DPFs) become more porous and the matrix becomes less continuous, as confirmed by studies [63, 381]. However, when the Date Palm Fiber (DPF) mass ratios are greater, too many fibers break apart the matrix, making it easier for water to get in. Because they are made of cellulose, Date Palm Fibers (DPFs) naturally attract water, as confirmed by studies [6, 174, 338, 380]. In addition, these results may also be consistent with studies [382, 383], which confirmed that the material may hold more water when the fiber mass ratio is larger, particularly if the fiber-matrix bonding is not strong enough. At the same time, the bio-composite materials (GF.s.m) with a Date Palm Fiber (DPF) mass ratio of (m: 15-20%) should be avoided in humid environments due to their high water absorption, as confirmed by studies [11, 174].

Ultimately, the findings of the water absorption ratios tests show that adding Date Palm Fiber (DPF) makes the bio-composite materials (GF.s.m) more resistant to water when there is not much of it, but more vulnerable when there is a lot of it, particularly for larger-sized (s: III) Date Palm Fibers (DPFs). Finding the right balance between mechanical performance and water absorption ratio in gypsum-based



bio-composites (GF.s.m) requires optimizing the size of Date Palm Fibers (DPFs), mass ratios, and manufacturing methods.

#### IV.4.3. Thickness Swelling Results

**Figures IV.10.a, and b.** Shows the thickness swelling results of gypsum compared to bio-composite materials (GF.s.m), as values of physical properties. The results are presented as bar columns with number labels as follows:

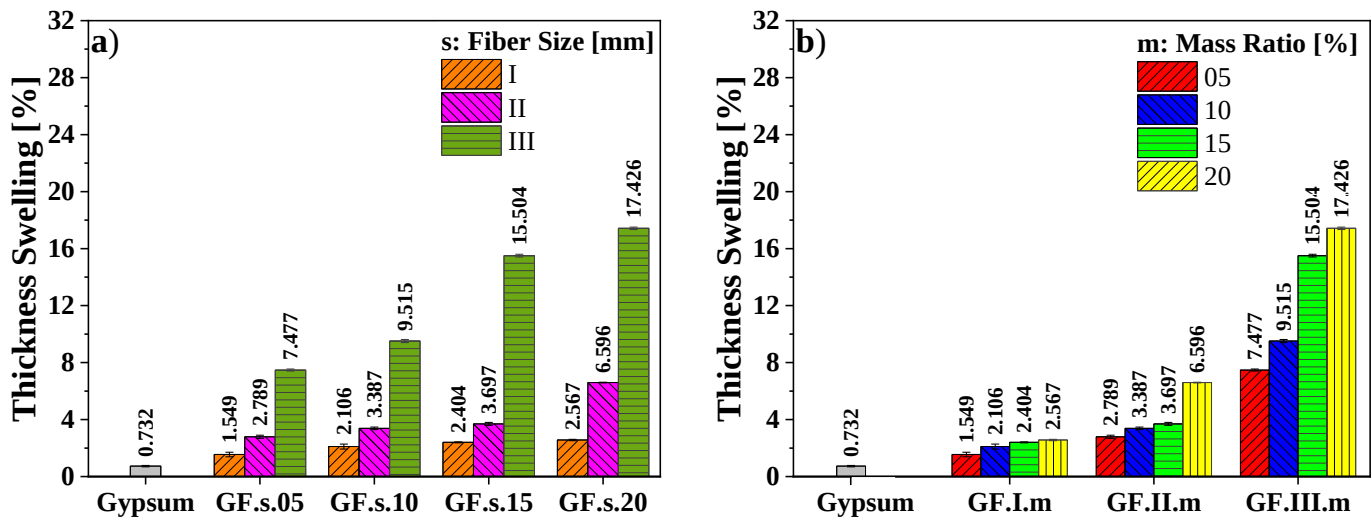


**Figure IV.10. Thickness Swelling Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.10.a, and b.** Shows that adding Date Palm Fibers (DPFs) to gypsum to make bio-composite materials (GF.s.m) increases the thickness swelling ratio in specimens compared to the normal gypsum specimen. The gypsum specimen had a thickness swelling ratio of  $(0.732 \pm 0.047 \%)$ , as shown in **Table IV.02**. The bio-composite materials (GF.I.05 and GF.III.20) specimens had the lowest and highest thickness swelling ratio values. The bio-composite materials (GF.I.05 and GF.III.20) specimens used the smaller and larger (s: I, and III) Date Palm Fiber (DPF) at mass ratios of 05 and 20%, respectively. When compared to the gypsum specimen, these thickness swelling ratio values of bio-composite materials (GF.I.05 and GF.III.20) were  $(1.549 \pm 0.158$  and  $17.426 \pm 0.083 \%)$  respectively, as shown in **Table IV.02**. Compared to the gypsum specimen, the two bio-composite material specimens (GF.I.05 and GF.III.20) had thickness swelling ratios that were 111.78 and 2282.24% higher, respectively, as shown in **Table IV.02**.

Furthermore, **Figure IV.10.a.** Shows that the inclusion increases of the Date Palm Fiber (DPF) mass ratio (m: 05-20%) cause the thickness swelling ratio to rise substantially in all sizes (s: I-III), indicating a clear effect of the Date Palm Fiber (DPF) addition on the thickness swelling behavior. In addition, this trend is especially pronounced in the bigger mass ratios (m: 20%) of Date Palm Fiber (DPF). The bio-composite material (GF.I.20) shows a thickness swelling ratio of  $(2.567 \pm 0.049\%)$ , while the bio-composite material (GF.II.20) shows a much higher thickness swelling ratio of  $(6.596 \pm 0.022\%)$ . However, the increase in mass ratio (m: 05-20%) leads to more Date Palm Fiber (DPF) being embedded within the gypsum matrix, which could potentially disrupt the matrix's compactness and increase the uptake of water, leading to a higher thickness swelling ratio, as confirmed by studies [16, 384, 385].

Furthermore, an increase in the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) in the bio-composite materials (GF.s.m) may enhance the mechanical properties of the bio-composite up to a certain level but also tends to increase the moisture absorption, which compromises dimensional stability. Research shows that when fiber mass ratios exceed a certain threshold, the composite may become more prone to moisture-related deformation, as seen in the dramatic increase in thickness swelling ratio at higher mass ratios (m: 15-20%) in these bio-composite materials (GF.s.m) [373, 386-388].

Furthermore, the date palm fiber-matrix interaction plays a significant role in the thickness swelling ratio behavior of bio-composite materials (GF.s.m). However, as Date Palm Fiber (DPF) mass ratios (m: 05-20%) increase, the interaction between the Date Palm Fiber (DPF) and the gypsum matrix becomes more complex. In addition, higher Date Palm Fiber (DPF) mass ratios (m: 05-20%) can lead to less effective bonding between the fiber and matrix, making the composite more susceptible to moisture absorption. This is consistent with the data showing an exponential increase in thickness swelling ratio for composites with higher fiber ratios, especially those with larger fibers, as confirmed by studies [389, 390].

Furthermore, **Figure IV.10.b.** Shows that the thickness swelling ratio generally increases as the Date Palm Fiber (DPF) size (s: I-III) increases in bio-composite materials (GF.s.m). Comparing similar mass ratios (m: 05-20%) across the three date palm sizes (s: I-III), it is evident that composites with larger-sized (s: III) Date Palm Fiber (DPF) show significantly higher thickness swelling ratios than those with smaller-size (s: I) Date Palm Fibers (DPFs). The bio-composite material (GF.I.05) has a thickness swelling ratio of  $(1.549 \pm 0.158\%)$ , whereas bio-composite material (GF.II.05) shows  $(2.789 \pm 0.110\%)$ , and bio-composite material (GF.III.05) shows  $(7.477 \pm 0.072\%)$ . This trend continues across all mass ratios (m: 05-20%), suggesting that larger-size (s: III) Date Palm Fiber (DPF) sizes lead to higher

absorption of moisture, which could result in greater dimensional changes upon thickness swelling ratios, as confirmed by studies [391, 392].

Furthermore, the relationship between Date Palm Fiber (DPF) size (s: I-III) and water absorption is well-documented in bio-composite materials (GF.s.m), as confirmed by studies [174, 393]. Larger-sized (s: III) Date Palm Fibers (DPFs) have a larger surface area for water absorption, which increases the thickness swelling ratio effect in bio-composite materials (GF.s.m), as confirmed by studies [394-396]. This effect can be attributed to the higher porosity and surface roughness associated with larger fibers, which create more spaces for water molecules to penetrate and cause a thickness swelling ratio, as confirmed by studies [373, 397]. In addition, due to their higher surface area-to-volume ratio, they might also contribute to increased water absorption, but to a lesser extent than larger fibers, as confirmed by studies [398-400].

Furthermore, Date Palm Fiber (DPF), being a natural material, has hydrophilic properties, which significantly contribute to the moisture absorption behavior of the bio-composite materials (GF.s.m), as confirmed by studies [3, 380]. The higher the fiber mass ratio and size, the greater the surface area for water interaction, as confirmed by study [382]. According to researches [171, 174, 380], natural fibers like date palm are highly sensitive to moisture, which can lead to dimensional instability when incorporated into composite materials.

Ultimately, the data presented for gypsum-Date Palm Fiber (DPF) bio-composite materials (GF.s.m) indicate that both the Date Palm Fiber (DPF) size (s: I-III) and mass ratio (m: 05-20%) have a significant effect on the thickness swelling ratio behavior. The swelling behavior reflects the fiber's water-absorbing properties, which must be carefully balanced with the desired mechanical performance of the bio-composite material (GF.s.m). For practical applications, optimizing the Date Palm Fiber (DPF) size (s: I-III) and mass ratio (m: 05-20%) could lead to bio-composite materials (GF.s.m) with an acceptable level of swelling while maintaining good mechanical properties.

#### IV.4.4. Moisture Content Results

**Figures IV.11.a, and b.** Shows the moisture content results of gypsum compared to bio-composite materials (GF.s.m), as values of physical properties. The results are presented as bar columns with number labels as follows:

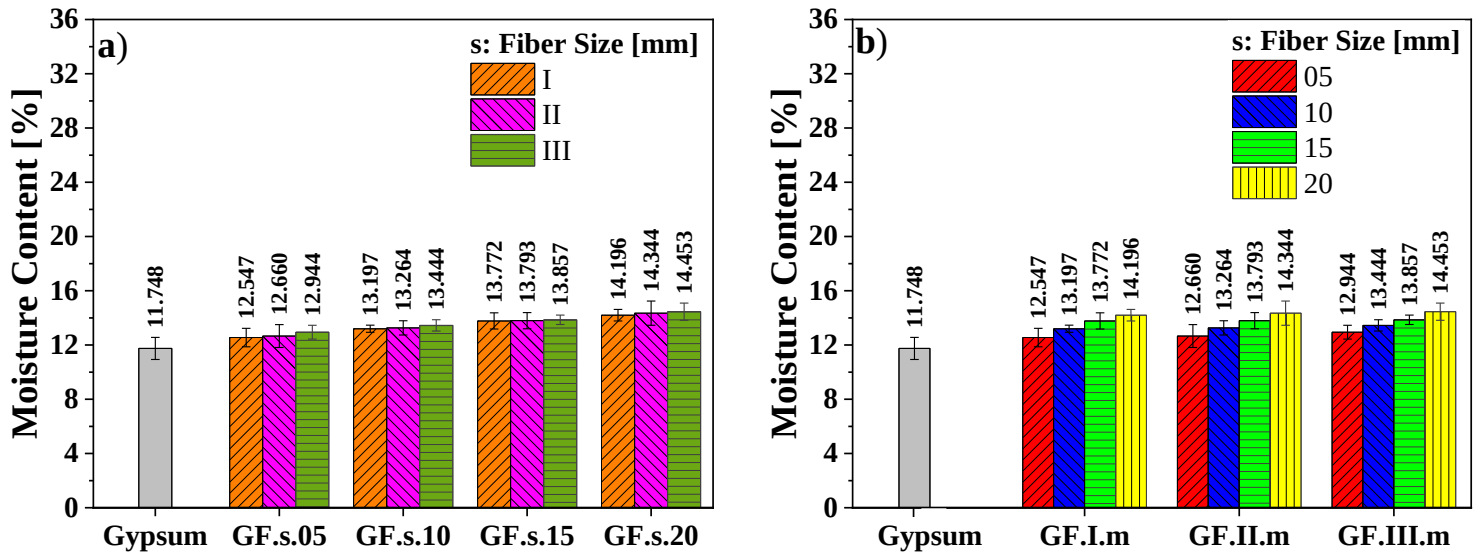


**Figure IV.11. Moisture Content Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.11.a, and b.** Shows that adding Date Palm Fibers (DPFs) to gypsum to make bio-composite materials (GF.s.m) increases the moisture content ratio in specimens compared to the normal gypsum specimen. The gypsum specimen had a moisture content ratio value of  $(11.748 \pm 0.814 \%)$ , as shown in **Table IV.02**. The bio-composite materials (GF.I.05 and GF.III.20) had the lowest and highest moisture content ratios, respectively. The bio-composite materials (GF.I.05 and GF.III.20) employed Date Palm Fibers (DPFs) in two sizes: smaller (s: I) and larger (s: III). The mass ratios were 05 and 20%, respectively. The moisture content ratios of the bio-composite materials (GF.I.05 and GF.III.20) were  $(12.547 \pm 0.681$  and  $14.453 \pm 0.634\%)$ , respectively, as shown in **Table IV.02**. This is compared to the gypsum specimen, **Table IV.02**. show that the two bio-composite material (GF.I.05 and GF.III.20) specimens had moisture content ratios that were 6.80 and 23.03% greater than the gypsum specimen, respectively.

Furthermore, **Figure IV.11.a.** Shows that although Date Palm Fiber (DPF) mass ratio (m: 05-20%) does impact the mechanical and thickness swelling ratio properties of bio-composite materials (GF.s.m), its effect on moisture content ratio is less pronounced. However, for similar mass ratios (m: 05-20%), moisture content ratio values show minimal differences between the fiber sizes. In addition, for bio-composite material (GF.I.05), which has a value of  $(12.547 \pm 0.681\%)$ , the bio-composite material (GF.II.05) has a value of  $(12.660 \pm 0.846\%)$ , and the bio-composite material (GF.III.05) has a value of  $(12.944 \pm 0.511\%)$ . However, for the mass ratio (m: 20%), the bio-composite material (GF.I.20) has a value of  $(14.196 \pm 0.424\%)$ , the bio-composite material (GF.II.20) has a value of  $(14.344 \pm 0.894\%)$ ,

and the bio-composite material (GF.III.20) has a value of  $(14.453 \pm 0.634\%)$ . In addition, the slight increase in moisture content ratio with larger-sized (s: III) Date Palm Fiber (DPF) could be explained by the larger surface area for water absorption, as fibers with greater surface area may offer more sites for moisture retention, as confirmed by studies [394, 401]. However, Date Palm Fiber (DPF), like most natural fibers, is highly hydrophilic due to the presence of cellulose and hemicellulose, as confirmed by studies [372, 402]. In addition, these fibers tend to attract water molecules, increasing the moisture content of the composites they are part of, as confirmed by studies [174, 380]. However, as the mass ratio of fibers increases, more cellulose and hemicellulose are available to absorb moisture, thus increasing the overall moisture content ratio, as confirmed by studies [373, 403].

Furthermore, as the mass ratio (m: 05-20%) of the Date Palm Fiber (DPF) increases, the overall moisture content ratio of the bio-composite materials (GF.s.m) rises. In addition, this is because the fiber, being hydrophilic, absorbs water from the environment and contributes to the overall moisture content, as confirmed by studies [404, 405]. However, higher mass ratios (m: 15-20%) provide more Date Palm Fiber (DPF) volume for water absorption, leading to higher moisture content ratios in the bio-composite materials (GF.s.m). In addition, the increase in moisture content ratio with mass ratio is commonly observed in natural fiber-reinforced composites. It is attributed to the fiber's affinity for water, as confirmed by studies [406, 407].

Furthermore, **Figure IV.11.b**. Shows that the moisture content ratio increases consistently as the Date Palm Fiber (DPF) size (s: I-III) increases in bio-composite materials (GF.s.m). In addition, in each Date Palm Fiber (DPF) size (s: I-III), this increasing trend can be explained by the larger size Date Palm Fiber (DPF) mass ratio (m: 15-20%) in the matrix, which has more hydrophilic sites for moisture absorption, as confirmed by studies [408, 409]. However, as the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) increases, the bio-composite's (GF.s.m) ability to absorb moisture naturally increases. In addition, the moisture absorption capacity is directly related to the amount and size of the fibers, as confirmed by studies [410, 411]. However, the increase in Date Palm Fiber (DPF) size (s: I-III) slightly influences the moisture content ratio, although the difference is relatively minor, as confirmed by study [412]. In addition, larger-sized fibers provide more surface area, which could result in higher moisture absorption, as confirmed by studies [413, 414]. However, the difference in moisture content ratio between the three Date Palm Fiber (DPF) sizes (s: I-III) is small, suggesting that other factors like fiber distribution and the quality of the fiber-matrix interaction might have a more significant impact on moisture retention than fiber size alone, as confirmed by studies [405, 406].

Furthermore, increased moisture content ratio in bio-composite materials (GF.s.m) can affect their dimensional stability and mechanical properties, as confirmed by studies [415, 416]. In addition, this phenomenon is evident in the thickness swelling data presented earlier, where the composites with higher moisture content also exhibited more significant swelling, as confirmed by studies [416, 417]. However, controlling moisture content is crucial for optimizing the performance of gypsum-based bio-composites in applications requiring stability and strength, as confirmed by studies [418, 419].

Ultimately, the moisture content ratio of gypsum-date palm fiber bio-composite materials (GF.s.m) increases with both Date Palm Fiber (DPF) size (s: I-III) and mass ratio (m: 05-20%). The data indicate that Date Palm Fiber's (DPFs) hydrophilic properties significantly contribute to this increase. These findings suggest that for applications where dimensional stability and resistance to moisture are crucial, the Date Palm Fiber (DPF) mass ratio and size should be carefully optimized.

IV.5. General Physical Results

**Table IV.02.** Shows the physical results. The results for the gypsum-date palm fiber bio-composite materials (GF.s.m) demonstrate a full comparison of several combinations of fiber sizes (s: I-III) and mass ratios (m: 05-20%) for each size as compared to pure gypsum. Several physical properties, including bulk density ( $\rho$ ), water absorption ratio (WAR), thickness swelling ratio ( $T_s$ ), and moisture content (MC), were used to describe these bio-composite materials (GF.s.m).

**Table IV.02. The Physical Results of Bio-composite vs. Gypsum.**

| Code      | Fiber Size | Mass Ratio | Bulk Density         | Ratio   | Water Absorption | Ratio   | Thickness Swelling | Ratio     | Moisture Content | Ratio   |
|-----------|------------|------------|----------------------|---------|------------------|---------|--------------------|-----------|------------------|---------|
| GF.s.m    | s          | m          | $\rho$               | -       | WAR              | -       | $T_s$              | -         | MC               | -       |
| Unit      | [mm]       | [%]        | [kg/m <sup>3</sup> ] | [%]     | [%]              | [%]     | [%]                | [%]       | [%]              | [%]     |
| Gypsum    | -          | -          | 1292.104 ± 19.413    | — 00.00 | 43.623 ± 1.295   | — 00.00 | 00.732 ± 0.047     | — 0000.00 | 11.748 ± 0.814   | — 00.00 |
| GF.I.05   | I          | 05         | 1229.353 ± 19.154    | ▼ 04.86 | 34.878 ± 3.082   | ▼ 20.05 | 01.549 ± 0.158     | ▲ 0111.78 | 12.547 ± 0.681   | ▲ 06.80 |
| GF.I.10   |            | 10         | 1122.351 ± 20.316    | ▼ 13.14 | 38.132 ± 1.893   | ▼ 12.59 | 02.106 ± 0.167     | ▲ 0187.92 | 13.197 ± 0.262   | ▲ 12.33 |
| GF.I.15   |            | 15         | 1091.928 ± 21.984    | ▼ 15.49 | 41.803 ± 1.157   | ▼ 04.17 | 02.404 ± 0.033     | ▲ 0228.61 | 13.772 ± 0.599   | ▲ 17.23 |
| GF.I.20   |            | 20         | 0888.355 ± 17.492    | ▼ 31.25 | 44.827 ± 1.264   | ▲ 02.76 | 02.567 ± 0.049     | ▲ 0250.89 | 14.196 ± 0.424   | ▲ 20.84 |
| GF.II.05  | II         | 05         | 1170.483 ± 16.075    | ▼ 09.41 | 37.957 ± 3.092   | ▼ 12.99 | 02.789 ± 0.110     | ▲ 0281.29 | 12.660 ± 0.846   | ▲ 07.76 |
| GF.II.10  |            | 10         | 1044.443 ± 16.058    | ▼ 19.17 | 39.913 ± 1.596   | ▼ 08.50 | 03.387 ± 0.089     | ▲ 0363.05 | 13.264 ± 0.525   | ▲ 12.90 |
| GF.II.15  |            | 15         | 0990.744 ± 18.350    | ▼ 23.32 | 43.795 ± 2.961   | ▲ 00.39 | 03.697 ± 0.105     | ▲ 0405.34 | 13.793 ± 0.596   | ▲ 17.41 |
| GF.II.20  |            | 20         | 0833.063 ± 18.131    | ▼ 35.53 | 46.431 ± 3.493   | ▲ 06.44 | 06.596 ± 0.022     | ▲ 0801.75 | 14.344 ± 0.894   | ▲ 22.10 |
| GF.III.05 | III        | 05         | 1159.658 ± 18.208    | ▼ 10.25 | 40.030 ± 2.521   | ▼ 08.24 | 07.477 ± 0.072     | ▲ 0922.17 | 12.944 ± 0.511   | ▲ 10.18 |
| GF.III.10 |            | 10         | 0990.257 ± 15.993    | ▼ 23.36 | 42.006 ± 3.224   | ▼ 03.71 | 09.515 ± 0.109     | ▲ 1200.69 | 13.444 ± 0.418   | ▲ 14.44 |
| GF.III.15 |            | 15         | 0928.280 ± 15.912    | ▼ 28.16 | 44.344 ± 1.852   | ▲ 01.65 | 15.504 ± 0.095     | ▲ 2019.52 | 13.857 ± 0.344   | ▲ 17.95 |
| GF.III.20 |            | 20         | 0806.678 ± 16.085    | ▼ 37.57 | 50.708 ± 1.267   | ▲ 16.24 | 17.426 ± 0.083     | ▲ 2282.24 | 14.453 ± 0.634   | ▲ 23.03 |

Where:

- G

: Gypsum;
- F

: Date Palm Fiber (DPF);
- GF.s.m

: The Bio-composite Material (GF.s.m) Made From Gypsum (G) Reinforced with Different Sizes (s: I, II, and III) and Mass Ratios (m: 05, 10, 15, and 20%) for each Size of Date Palm Fibre (F);
- The Symbol ▲

: The Ratio of the Value Increase of Bio-composite vs. Gypsum [%];
- The Symbol ▼

: The Ratio of the Value Decrease of Bio-composite vs. Gypsum [%];
- The Symbol —

: The Ratio of the Value Increase and Decrease of Bio-composite vs. Gypsum [%];

The results of the **Table IV.02.** were discussed as follows:

1.

**Bulk Density ( $\rho$ ):** Adding Date Palm Fibers (DPFs) in any size (s: I-III) or mass ratio (m: 05-20%) lowers the bulk density. In bio-composite materials (GF.s.m), the bulk density was reduced by (04.86 %) in bio-composite materials (GF.I.05) and by (37.57%) in bio-composite materials (GF.III.20) as compared to gypsum. Furthermore, this drop is significant, which shows that the bio-composite materials (GF.s.m) become lighter as more fibers are added, as confirmed by study [420]. In addition, these studies say that adding Date Palm Fibers (DPFs), which are less dense than gypsum, makes

the composite less dense overall [11, 23]. In addition, this is excellent for uses that need light materials, as confirmed by studies [17, 364]. Furthermore, this tendency is well-known in the literature since fibers are often utilized to make composites lighter while keeping or increasing their other mechanical characteristics [3, 17];

- 2. Water Absorption (WAR):** In bio-composite materials (GF.s.m), adding Date Palm Fibers (DPFs) usually lowers the water absorption ratio. For bio-composite material (GF.I.m), the amount of water it absorbed went down by (20.05%) for (GF.I.05), while for (GF.I.15), it went down by (04.17%) as compared to gypsum. In addition, this is confirmed by studies [408, 421], which indicate that the fibers' hydrophobic nature may make it harder for water to get through, which might explain why the lower mass ratios absorb less water. Furthermore, the water absorption ratio goes up slowly when the mass ratio goes up, as confirmed by studies [387, 422]. In addition, in other bio-composite materials, like (GF.I.20) and (GF.III.20), it went up by (2.76%) and (16.24%), respectively. However, if the fiber concentration is greater, the composite may become more porous, which would make it absorb more water, as confirmed by study [423]. In other fiber-reinforced composites, a higher fiber mass ratio may open the structure, improving water absorption, as confirmed by studies [373, 424];
- 3. Thickness Swelling (Ts):** In bio-composite materials (GF.s.m), the thickness swelling ratio changes a lot when the fiber mass ratio (m: 05-20%) and size (s: I-III) go up. Furthermore, for example, the bio-composite material (GF.I.05) swells by (111.78%), whereas the bio-composite material (GF.III.20) swells by (2282.24%) as compared to gypsum. Furthermore, the swelling has become so bad because the composite has larger fibers that soak up more water and get larger, as confirmed by studies [425, 426]. In addition, the thickness swelling ratio is a key sign of how stable the material's dimensions are under wet or humid conditions, as confirmed by studies [427, 428]. In addition, the higher swelling ratios at larger mass ratios show that gypsum composites with fibers in them may alter size when they come into contact with water. This behavior may be attributed to the hydrophilic qualities of the fiber, which are intensified by bigger fiber diameters, as confirmed by studies [373, 429];
- 4. Moisture Content (MC):** In bio-composite materials (GF.s.m), the moisture content ratio of most of the specimens goes up steadily compared to gypsum. Furthermore, for instance, in bio-composite material (GF.I.05), the moisture content ratio went up by (6.80%), and in bio-composite material (GF.III.20), it went up by (23.03%) as compared to gypsum. In addition, the higher moisture content is in line with the fact that the bio-composite materials (GF.s.m) can hold more water because date palm is fibrous, as confirmed by studies [398, 430]. Furthermore, the ability to absorb and hold moisture rises as the ratio of fiber mass increases, as confirmed by studies [404, 405]. In addition, this is confirmed by studies [37, 398], which indicate that this is important for materials that will be utilized in places with different levels of humidity. In addition, the increased moisture content ratio in larger-sized (s: III) fiber composites could be beneficial for certain uses, including building, where controlling moisture is very important.

Ultimately, adding Date Palm Fibers (DPFs) to gypsum bio-composite materials (GF.s.m) modifies their physical results a lot in the end. Bio-composites might be employed in lightweight materials and construction. However, more research is needed to determine the appropriate balance between the amount of water they can absorb, their swelling capacity, and their strength. For uses that need less weight and moderate water absorption, lower Date Palm Fiber (DPF) mass ratios (m: 05-10%) and smaller Date Palm Fiber (DPFs) sizes (s: I) seem to work well. However, larger date palm fiber size (s: III) and higher mass ratios (m: 15-20%) make the bio-composite material (GF.s.m) more stable and hold more moisture, which might be useful in certain situations.



## IV.6. Thermal Results

The results obtained from [Table IV.03](#), are discussed as thermal properties related to the bio-composite material specimens (GF.s.m), including different sizes (s: I-III) and mass ratios (m: 05-20%) of each size of palm fiber compared to the gypsum specimen, as follows:

### IV.6.1. Thermal Conductivity Results

[Figures IV.12.a, and b](#). Shows the thermal conductivity results of gypsum compared to bio-composite materials (GF.s.m), as values of thermal properties. The results are presented as bar columns with number labels as follows:

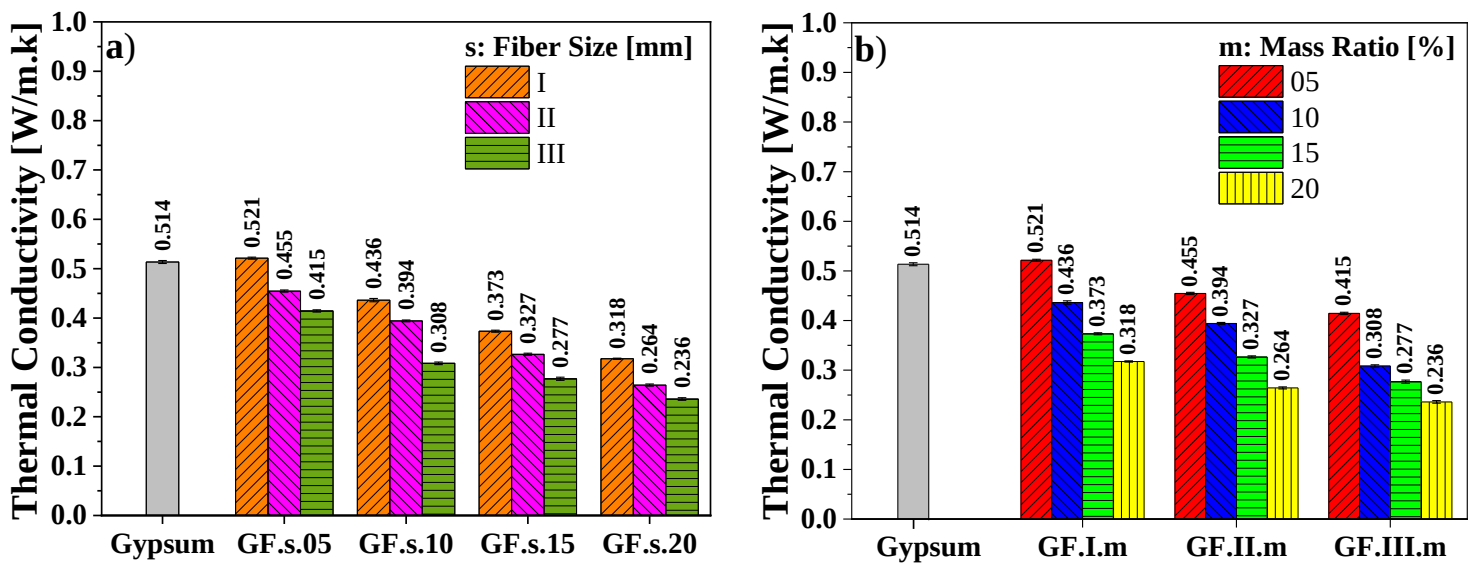


**Figure IV.12. Thermal Conductivity Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

[Figures IV.12.a, and b](#). Shows that adding Date Palm Fibers (DPFs) to gypsum to make bio-composite material (GF.s.m) specimens makes them better at thermal insulation by lowering their thermal conductivity, compared to the gypsum specimen. The gypsum specimen has a thermal conductivity value of  $(0.514 \pm 0.003 \text{ W/m}\cdot\text{K})$ , as shown in [Table IV.03](#). Among the bio-composite materials (GF.s.m), the bio-composite material (GF.III.20) exhibits the lowest thermal conductivity value, while the bio-composite material (GF.I.05) demonstrates the highest. These bio-composite materials (GF.III.20 and GF.I.05) are reinforced with larger (III) and smaller (I) sizes Date Palm Fibers (DPFs), with mass ratios of 20 and 05%, and possess thermal conductivity values of  $(0.236 \pm 0.003 \text{ and } 0.521 \pm 0.002 \text{ W/m.k})$ , respectively, in comparison to the gypsum specimen, as shown

in **Table IV.03**. Compared to the gypsum specimen, the two bio-composite materials (GF.III.20 and GF.I.05) exhibit reductions and increases in thermal conductivity values of 54.09 and 1.36%, respectively, as shown in **Table IV.03**.

Furthermore, **Figure IV.12.a**. Shows that for all sizes of Date Palm Fiber (DPF), the thermal conductivity gradually decreases as the ratio of fiber mass increases in bio-composite materials (GF.s.m). The smaller (s: I)-size Date Palm Fibers (DPFs) result in higher thermal conductivity compared to medium and larger (s: II, III) size Date Palm Fibers (DPFs) at equivalent fiber mass ratios. In addition, these results may also be consistent with studies [395, 431], which confirmed that the smaller (s: I) size Date Palm Fibers (DPFs), which are more uniformly dispersed, maintain better matrix continuity, resulting in higher thermal conductivity. The bio-composite materials (GF.III.m), which have larger (s: III) size Date Palm Fibers (DPFs), have the lowest thermal conductivity for all Date Palm Fiber (DPF) mass ratios (m: 05-20%). This is because they have many voids and not a lot of matrix continuity, as confirmed by study [432]. The bio-composites (GF.III.m) exhibit the best thermal conductivity, especially at a fiber mass ratio of (m: 20%), with a value of  $(0.236 \pm 0.003 \text{ W/m.k})$ . This makes them ideal for applications requiring superior thermal resistance, as confirmed by studies [37, 433]. The bio-composites (GF.III.m), especially those with a mass ratio of (m: 15-20%) Date Palm Fiber (DPF), are ideal for applications requiring high thermal insulation, such as wall panels or ceiling panels, as confirmed by studies [3, 17]. In addition, ensuring proper fiber dispersion and minimizing excessive voids can balance thermal and mechanical properties, especially in composites with smaller (s: I) sizes of Date Palm Fiber (DPF), as confirmed by studies [434, 435].

Furthermore, **Figure IV.12.b**. Shows that date palm fiber-reinforced bio-composite materials (GF.s.m) have low thermal conductivity, particularly when the mass ratio of Date Palm Fiber (DPF) is high (m: 20%). The thermal conductivity goes down when the mass ratio (m: 05-20%) of Date Palm Fibers (DPF) increases, no matter the size (s: I-III) of the Date Palm Fibers (DPFs). The Date Palm Fibers (DPFs) break up the dense gypsum matrix, creating holes filled with air that make the material less able to transmit heat, as confirmed by studies [9, 11]. This disruption is stronger when the mass ratio of Date Palm Fiber (DPF) is larger (m: 15-20%). This is because a greater mass ratio of Date Palm Fiber (DPF) makes the bio-composite materials (GF.s.m) more porous, which lowers their thermal conductivity, as confirmed by study [11]. In addition, the air in the voids is a powerful thermal insulator since it slows down the flow of heat, as confirmed by studies [436, 437]. Research substantiates that natural fibers enhance porosity in bio-composites, markedly diminishing heat conductivity, in alignment with observed

patterns [372, 395]. In addition, these results may also align with study [438], which confirmed that bio-composite materials (GF.s.m) containing a high mass ratio of Date Palm Fiber (DPF) or larger fiber size (s: III) are suitable for thermal conductivity applications because they exhibit lower thermal conductivity.

Ultimately, the thermal conductivity data show that the size and mass ratio of the Date Palm Fibers (DPFs) affect how well gypsum-based bio-composites (GF.s.m) insulate against heat. The bio-composite materials (GF.III.m) with a larger fiber size (s: III) and a greater fiber mass ratio are better insulators; hence, they are suitable for uses that need superior thermal resistance.

#### IV.6.2. Thermal Diffusivity Results

**Figures IV.13.a, and b.** Shows the thermal diffusivity results of gypsum compared to bio-composite materials (GF.s.m), as values of thermal properties. The results are presented as bar columns with number labels as follows:

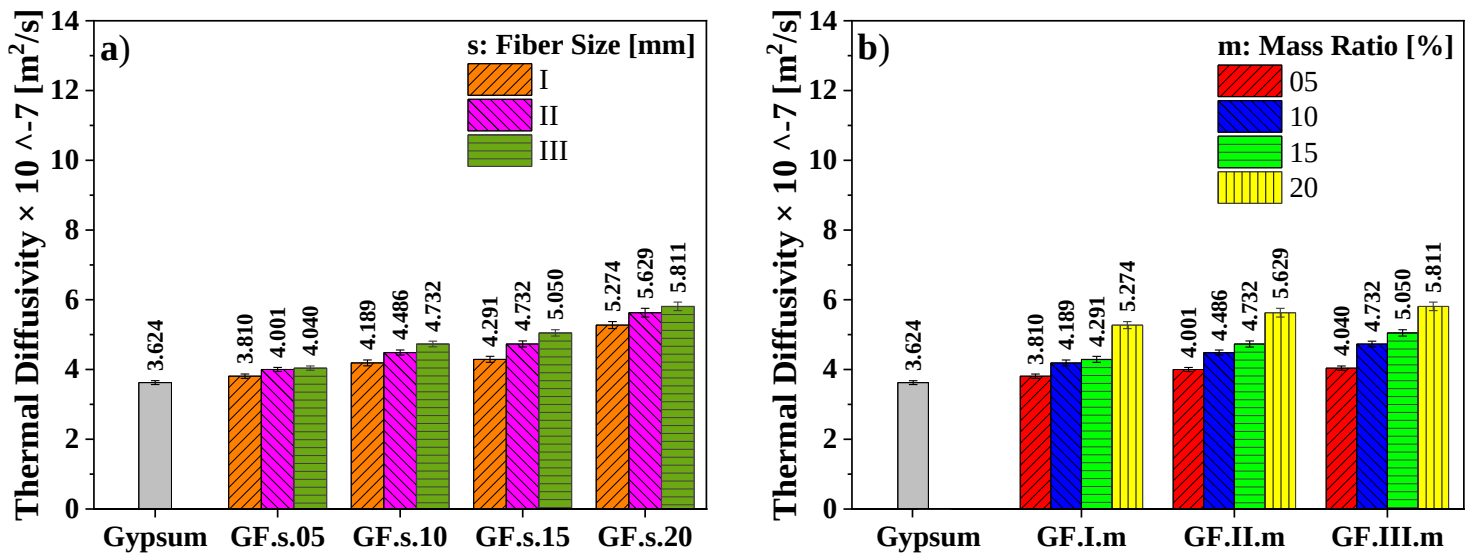


**Figure IV.13. Thermal Diffusivity Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.13.a, and b.** Shows that adding Date Palm Fibers (DPFs) to gypsum to form bio-composite material (GF.s.m) specimens makes them less thermally conductive than the gypsum specimen. The gypsum specimen has a thermal diffusivity value of  $(3.624 \pm 0.055 \times 10^{-7} m^2/s)$ , as shown in **Table IV.03**. The bio-composite material (GF.I.05) has the lowest thermal diffusivity value of all the bio-composite materials (GF.s.m), whereas the bio-composite material (GF.III.20) has the highest. These bio-composite materials (GF.I.05 and GF.III.20) are strengthened with smaller (I) and larger (III) Date Palm Fibers

(DPFs), with mass ratios of 05 and 20%, and have thermal diffusivity values of  $(3.810 \pm 0.061 \times 10^{-7} \text{ m}^2/\text{s})$  and  $(5.811 \pm 0.121 \times 10^{-7} \text{ m}^2/\text{s})$ , respectively, when compared to the gypsum specimen, as shown in **Table IV.03**. The two bio-composite materials (GF.I.05 and GF.III.20) have thermal diffusivity values that are 5.13% and 60.35% higher than those of the gypsum specimen, as shown in **Table IV.03**.

Furthermore, **Figure IV.13.a**. Shows the thermal diffusivity of the bio-composite materials (GF.s.m) goes from  $(3.810 \pm 0.061 \times 10^{-7} \text{ m}^2/\text{s})$  for Date Palm Fiber (DPF) mass ratio (m: 05%) to  $(5.811 \pm 0.121 \times 10^{-7} \text{ m}^2/\text{s})$  for Date Palm Fiber (DPF) mass ratio (m: 20%). Composites with larger-sized (s: III) Date Palm Fibers (DPFs) seem to have higher thermal diffusivity. Furthermore, for example, the bio-composite material (GF.III.m) always has greater thermal diffusivity values than those of (GF.I.m and GF.II.m), respectively. In addition, the bio-composites with a (m: 20%) mass ratio of Date Palm Fiber (DPF) have a thermal diffusivity of  $(5.811 \pm 0.121 \times 10^{-7} \text{ m}^2/\text{s})$  for the Date Palm Fiber (DPF) size (s: III). In contrast, the bio-composite material (GF.I.m), Date Palm Fiber (DPF) size (s: I), has a thermal diffusivity of  $(5.274 \pm 0.101 \times 10^{-7} \text{ m}^2/\text{s})$  for the same Date Palm Fiber (DPF) mass ratio. Furthermore, this means that larger-sized (s: III) Date Palm Fibers (DPFs) may help heat go through the material better by making more continuous paths for it to move through, as confirmed by studies [395, 439]. Also, larger-sized (s: III) Date Palm Fibers (DPFs) could make the bio-composite materials (GF.s.m) highly porous, which could make it easier for heat to move through them, as confirmed by studies [440, 441]. In addition, these results may also align with studies [442, 443], which confirmed that adding Date Palm Fiber (DPF) always makes the material's thermal diffusivity higher, no matter what size or mass ratio the fibers are. Furthermore, this shows that adding bio-based fibers like date palm to gypsum makes it better at handling heat, as confirmed by studies [17, 444]. In addition, other research on composites made from natural fibers has demonstrated that the size of the fibers can change how well they conduct heat, especially when it comes to making them better, as confirmed by studies [445, 446].

Furthermore, **Figure IV.13.b**. Shows results indicating that the thermal diffusivity of the bio-composite materials (GF.s.m) increases with higher fiber mass ratio (m: 05-20%) and Date Palm Fiber (DPF) size (s: I-III). This suggests that adding Date Palm Fiber (DPF) to gypsum reduces its thermal conductivity, especially as the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) increases, as confirmed by studies [17, 364]. In contrast, this could be a useful quality for uses that need materials that can control heat in a specific way, as confirmed by studies [3, 23]. In addition, these results may also align with studies [447, 448], which confirmed that when the fiber mass ratio (m: 05-20%) increases, the thermal diffusivity for all Date Palm Fiber (DPF) sizes (s: I-III) goes up as well. In contrast, these findings corroborate the

concept that a higher ratio of Date Palm Fiber (DPF) improves the thermal diffusivity characteristics of the composites, as confirmed by studies [449, 450]. Furthermore, this is because the fibers themselves have lower thermal conductivity, and the material's bulk density has gone down overall, as confirmed by studies [395, 451]. In addition, these results may also align with studies [452-454], which confirmed that these discoveries are especially important in areas where thermal qualities are crucial, like construction materials, insulation, and other uses. Furthermore, increasing the thermal diffusivity of these bio-composites could make them more useful in construction materials, where both the ability to keep heat in and let it out are crucial, as confirmed by studies [438, 455, 456]. In contrast, a comparable investigation into bio-composites incorporating natural fibers such as hemp, jute, and sisal revealed that increasing the fiber mass ratio improved the thermal characteristics of the composites [457-459].

Ultimately, the results of this study show that adding Date Palm Fiber (DPF) to gypsum greatly improves the thermal diffusivity of the resulting bio-composites (GF.s.m), especially when the fibers are larger and more concentrated. This finding underscores the feasibility of utilizing these materials in construction and architectural settings where thermal management is essential.

### IV.6.3. Specific Heat Results

**Figures IV.14.a, and b.** Show the specific heat results of gypsum compared to bio-composite materials (GF.s.m), as values of thermal properties. The results are presented as bar columns with number labels as follows:

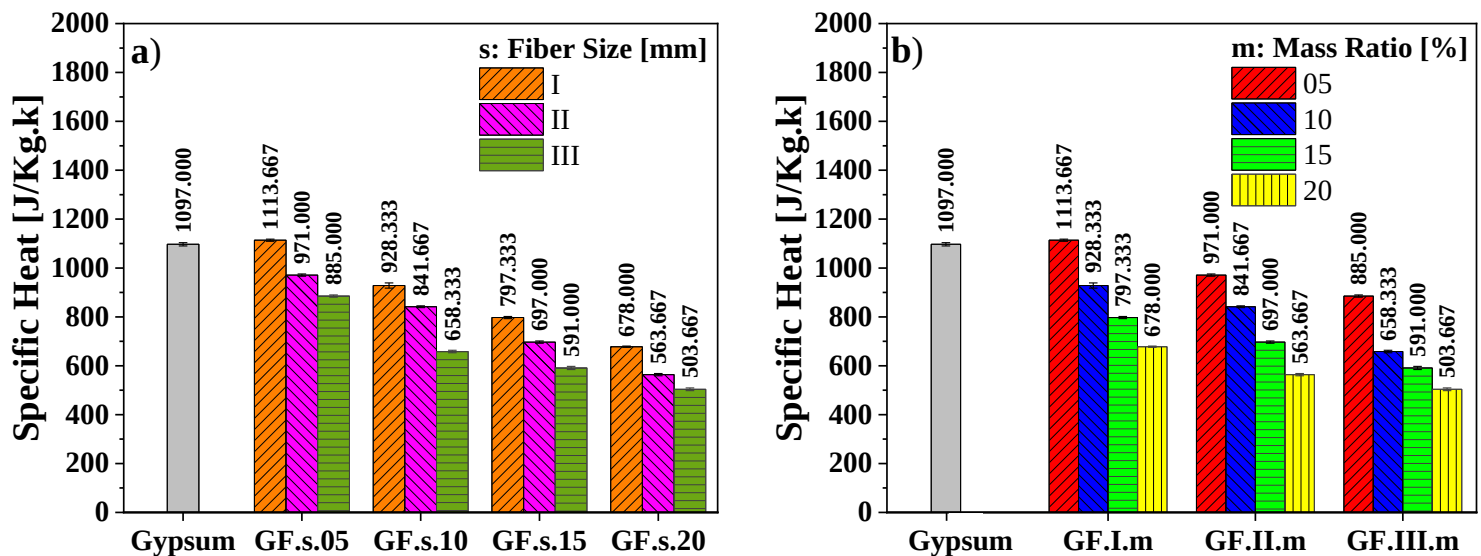


**Figure IV.14. Specific Heat Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figures IV.14.a, and b.** Shows that adding Date Palm Fibers (DPFs) to gypsum to form bio-composite material (GF.s.m) specimens makes them less specific heat than the gypsum specimen. The gypsum specimen has a specific heat value of  $(1097.000 \pm 06.667 \text{ J/kg.k})$ , as shown in **Table IV.03**. The bio-composite material (GF.II.05) has the lowest thermal diffusivity value of all the bio-composite materials (GF.s.m), whereas the bio-composite material (GF.I.05) has the highest. These bio-composite materials (GF.II.05 and GF.I.05) are strengthened with medium (s: II) smaller (s: I) Date Palm Fibers (DPFs) sizes, with a mass ratio of (m: 05%), and have specific heat values of  $(971.000 \pm 04.667 \text{ and } 1113.667 \pm 04.222 \text{ J/kg.k})$ , respectively, when compared to the gypsum specimen, as shown in **Table IV.03**. The two bio-composite materials (GF.II.05 and GF.I.05) have specific heat values that are 11.49 and 1.52% lower and higher, respectively, than those of the gypsum specimen, as shown in **Table IV.03**.

Furthermore, **Figure IV.14.a.** Shows the specific heat of bio-composite material (GF.I.m) is  $(1113.667 \pm 04.222 \text{ J/kg.k})$  at a Date Palm Fiber (DPF) mass ratio of (m: 05%). However, this value goes down as the Date Palm Fiber (DPF) mass ratio (m: 05-20%) goes up. In contrast, it goes down to  $(678.000 \pm 02.667 \text{ J/kg.k})$  when the Date Palm Fiber (DPF) mass ratio is at its greatest (m: 20%). The pattern is the same for bio-composite material (GF.II.m), with specific heat going from  $(971.000 \pm 04.667 \text{ J/kg.k})$  at (m: 05%) to  $(563.667 \pm 04.444 \text{ J/kg.k})$  at (m: 20%). In contrast, the bio-composite material (GF.III.m) of mass ratio (m: 05%) Date Palm Fiber (DPF) has a specific heat of  $(885.000 \pm 04.667 \text{ J/kg.k})$ , while for (m: 20%) Date Palm Fiber (DPF), it drops to  $(503.667 \pm 05.556 \text{ J/kg.k})$ . Furthermore, the specific heat of the bio-composite material (GF.s.m) at each Date Palm Fiber (DPF) mass ratio (m: 05-20%) normally goes down as the Date Palm Fiber (DPF) size (s: I-III) goes up. In contrast, this is because larger-sized (s: III) Date Palm Fibers (DPFs) are less able to hold heat, as confirmed by studies [63, 460]. In addition, these results may also align with studies [23, 259], which confirmed that compared to gypsum, natural fibers like Date Palm Fiber (DPF) usually have a lower specific heat capacity. In contrast, when fibers are added to gypsum, the specific heat of the bio-composite is mostly made up of the specific heat of the fibers, as confirmed by studies [438, 461]. Furthermore, adding fibers to gypsum makes the composite have a lower specific heat since fibers do not hold heat as well as gypsum does, as confirmed by study [384]. Furthermore, larger fiber sizes (s: III), such as those in bio-composite material (GF.III.m), have a bigger influence on lowering the specific heat, as confirmed by study [462]. In addition, these results may also align with studies [11, 447], which confirmed that this could be because larger-sized (s: III) Date Palm Fibers (DPFs) can create more gaps or voids in the matrix, which makes it less able to store heat. Furthermore, it is believed that smaller-

sized (s: I) Date Palm Fibers (DPFs) make the bio-composite material (GF.s.m) structure more compact and even, which leads to greater specific heat values than bio-composites with larger-sized (s: III) fibers, as confirmed by studies [9, 17, 463]. In contrast, this trend is in line with past bio-composite studies that found that smaller fibers improve thermal performance by reducing air pockets and making heat transmission channels work better [464, 465].

Furthermore, **Figure IV.14.b.** Shows that as the Date Palm Fiber (DPF) mass ratio (m: 05-20%) increases, the specific heat capacity of the bio-composites tends to decrease, with a few outliers at lower Date Palm Fiber (DPF) loadings. In contrast, when the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) in the bio-composite materials (GF.s.m) increases, the specific heat goes below that of pure gypsum. Furthermore, this means that the Date Palm Fiber (DPF) reinforcement makes the bio-composite materials (GF.s.m) less capable of storing thermal energy, as confirmed by studies [17, 443]. In addition, this is perhaps because the specific heat of Date Palm Fiber (DPF) is lower than that of gypsum itself, as confirmed by studies [17, 263, 466]. In addition, these results may also align with study [398], which confirmed that the gypsum matrix-fiber interaction and the fibers' ability to make the bio-composite materials (GF.s.m) more porous may also hinder heat storage. Furthermore, the bio-composite materials (GF.s.m) get less uniform as the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) to the total increases, which means they can hold less heat, as confirmed by study [9]. In contrast, the lower specific heat that comes with a higher Date Palm Fiber (DPF) mass ratio (m: 05-20%) suggests that these bio-composite materials (GF.s.m) might not be suitable for uses where much thermal energy needs to be stored. In addition, these results may also align with studies [11, 17, 364], which confirmed that they could be beneficial in applications where low thermal storage and faster heat dissipation are desired, such as in some types of insulation materials or temperature-sensitive applications. For instance, a bio-composite material like (GF.III.20) could be utilized in buildings or systems that need to cool down quickly and do not want to keep heat in.

Ultimately, the findings demonstrate that augmenting the Date Palm Fiber (DPF) mass ratio (m: 05-20%) and Date Palm Fiber (DPF) sizes (s: I-III) resulted in a decrease in the specific heat of gypsum-based bio-composites (GF.s.m). The main reason for this drop is that Date Palm Fiber (DPF) has a lower specific heat than gypsum. These bio-composites (GF.s.m) might not be suitable for storing thermal energy since they have a lower specific heat, but they might be good for places where heat needs to be released quickly.



IV.7. General Thermal Results

**Table IV.03.** Shows the thermal test results for gypsum bio-composite materials (GF.s.m) reinforced with Date Palm Fibers (DPFs) show how the size (s: I-III) and mass ratio (m: 05-20%) of the Date Palm Fibers (DPFs) affect thermal conductivity ( $\lambda$ ), thermal diffusivity ( $\alpha$ ), and specific heat (C). These results are very important for building materials and thermal insulation materials, where managing heat is very significant.

**Table IV.03. The Thermal Results of Bio-composite vs. Gypsum.**

| Code      | Fiber Size | Mass Ratio | Thermal Conductivity | Ratio   | Thermal Diffusivity $\times 10^{-7}$ | Ratio   | Specific Heat         | Ratio   |
|-----------|------------|------------|----------------------|---------|--------------------------------------|---------|-----------------------|---------|
| GF.s.m    | s          | m          | $\lambda$            | -       | $\alpha$                             | -       | C                     | -       |
| Unit      | [mm]       | [%]        | [W/m.k]              | [%]     | [m²/s]                               | [%]     | [J/kg.k]              | [%]     |
| Gypsum    | -          | -          | $0.514 \pm 0.003$    | — 00.00 | $3.624 \pm 0.055$                    | — 00.00 | $1097.000 \pm 06.667$ | — 00.00 |
| GF.I.05   | I          | 05         | $0.521 \pm 0.002$    | ▲ 01.36 | $3.810 \pm 0.061$                    | ▲ 05.13 | $1113.667 \pm 04.222$ | ▲ 01.52 |
| GF.I.10   |            | 10         | $0.436 \pm 0.003$    | ▼ 15.18 | $4.189 \pm 0.084$                    | ▲ 15.59 | $0928.333 \pm 10.889$ | ▼ 15.38 |
| GF.I.15   |            | 15         | $0.373 \pm 0.002$    | ▼ 27.43 | $4.291 \pm 0.086$                    | ▲ 18.41 | $0797.333 \pm 04.444$ | ▼ 27.32 |
| GF.I.20   |            | 20         | $0.318 \pm 0.001$    | ▼ 38.13 | $5.274 \pm 0.101$                    | ▲ 45.53 | $0678.000 \pm 02.667$ | ▼ 38.20 |
| GF.II.05  | II         | 05         | $0.455 \pm 0.002$    | ▼ 11.48 | $4.001 \pm 0.057$                    | ▲ 10.40 | $0971.000 \pm 04.667$ | ▼ 11.49 |
| GF.II.10  |            | 10         | $0.394 \pm 0.002$    | ▼ 23.35 | $4.486 \pm 0.073$                    | ▲ 23.79 | $0841.667 \pm 03.778$ | ▼ 23.28 |
| GF.II.15  |            | 15         | $0.327 \pm 0.002$    | ▼ 36.38 | $4.732 \pm 0.087$                    | ▲ 30.57 | $0697.000 \pm 04.667$ | ▼ 36.46 |
| GF.II.20  |            | 20         | $0.264 \pm 0.002$    | ▼ 48.64 | $5.629 \pm 0.124$                    | ▲ 55.33 | $0563.667 \pm 04.444$ | ▼ 48.62 |
| GF.III.05 | III        | 05         | $0.415 \pm 0.002$    | ▼ 19.26 | $4.040 \pm 0.063$                    | ▲ 11.48 | $0885.000 \pm 04.667$ | ▼ 19.33 |
| GF.III.10 |            | 10         | $0.308 \pm 0.003$    | ▼ 40.08 | $4.732 \pm 0.080$                    | ▲ 30.57 | $0658.333 \pm 05.111$ | ▼ 39.99 |
| GF.III.15 |            | 15         | $0.277 \pm 0.003$    | ▼ 46.11 | $5.050 \pm 0.091$                    | ▲ 39.35 | $0591.000 \pm 06.667$ | ▼ 46.13 |
| GF.III.20 |            | 20         | $0.236 \pm 0.003$    | ▼ 54.09 | $5.811 \pm 0.121$                    | ▲ 60.35 | $0503.667 \pm 05.556$ | ▼ 54.09 |

Where:

- G

: Gypsum;
- F

: Date Palm Fiber (DPF);
- GF.s.m

: The Bio-composite Material (GF.s.m) Made From Gypsum (G) Reinforced with Different Sizes (s: I, II, and III) and Mass Ratios (m: 05, 10, 15, and 20%) for each Size of Date Palm Fibre (F);
- The Symbol ▲

: The Ratio of the Value Increase of Bio-composite vs. Gypsum [%];
- The Symbol ▼

: The Ratio of the Value Decrease of Bio-composite vs. Gypsum [%];
- The Symbol —

: The Ratio of the Value Increase and Decrease of Bio-composite vs. Gypsum [%];

The results of the **Table IV.03.** were discussed as follows:

1.

**Thermal Conductivity ( $\lambda$ ):** The thermal conductivity of pure gypsum is ( $0.514 \pm 0.003$  W/m.k), and it goes down as the Date Palm Fiber (DPF) mass ratios (m: 05-20%) go up, regardless of what size (s: I-III) the fibers are. In addition, in bio-composite materials (GF.I.m), as the mass ratio increases (m: 05-20%), thermal conductivity goes down a lot, reaching ( $0.318 \pm 0.001$  W/m.k). Furthermore, in bio-composite materials (GF.II.m), the trend is the same, but the thermal conductivity values are higher than bio-composite materials (GF.I.m) for the same mass ratios. For example, the



bio-composite material (GF.II.05) value is ( $0.455 \pm 0.002$  W/m.k), whereas the bio-composite material (GF.I.05) value is ( $0.521 \pm 0.002$  W/m.k). Furthermore, in bio-composite materials (GF.III.m), this group has the lowest thermal conductivity with increased fiber concentration, with bio-composite material (GF.III.20) measuring ( $0.236 \pm 0.003$  W/m.k). This means that the insulation works better, as confirmed by studies [456, 467]. Furthermore, thermal conductivity decreases with fiber mass ratio (m: 05%), consistent with research that demonstrates natural fibers produce air pockets in the matrix [399, 408]. In addition, this is confirmed by studies [395, 414], which indicate that these areas are thermal barriers, making heat transfer harder. Larger fibers, like those in bio-composite materials (GF.III.m), may cause bigger decreases since they do not stick to the matrix as well, which makes the material more porous, as confirmed by studies [372, 468];

2.

**Thermal diffusivity ( $\alpha$ ):** As the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) increases, the thermal diffusivity also goes up in each size (s: I-III). Furthermore, this means that heat spreads quicker. In addition, the rise in thermal diffusivity is because the bio-composite materials (GF.s.m) can hold less heat (Lower Specific Heat) and transmit heat better, as confirmed by studies [438, 469]. This aligns with results from thermal composite investigations [470, 471], which indicated that diffusivity increases owing to the prevalence of fast heat conduction pathways established by fiber-matrix interactions;
3.

**Specific Heat (C):** The specific heat of pure gypsum is ( $1097.000 \pm 06.667$  J/kg.k), which means it can store much heat. In addition, as the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) increases, the specific heat goes down for all sizes (s: I-III). In addition, this is confirmed by studies [472, 473], which indicate that adding fibers to the mix lowers the specific heat, which makes sense since the low-density fibers dilute the dense gypsum matrix, which means they store less heat per unit mass. Thermal research on fiber-reinforced composites that indicate a lower heat capacity when porous reinforcements are added supports this [474, 475].

Ultimately, the sizes and composition of the Date Palm Fibers (DPFs) heavily influence the thermal properties of gypsum-date palm fiber bio-composite materials (GF.s.m). Increasing the mass ratio (m: 05-20%) of Date Palm Fiber (DPF) in a bio-composite material (GF.s.m) improves its insulation by lowering its thermal conductivity and specific heat. However, it also makes it easier for heat to go through the bio-composite materials (GF.s.m).

## IV.8. Thermo-Physical Results

The results obtained from [Table IV.02, and 03](#). are discussed as thermo-physical properties related to the bio-composite material specimens (GF.s.m), including different sizes (s: I-III) and mass ratios (m: 05-20%) of each size of Date Palm Fiber (DPF) compared to the gypsum specimen, as follows:

### IV.8.1. Thermal Conductivity-Bulk Density Results

**Figure IV.15.** Show the thermal conductivity-bulk density results of the gypsum specimen compared to bio-composite materials (GF.s.m) specimens as values of thermo-physical properties. These findings are shown as a scatter plot as follows:

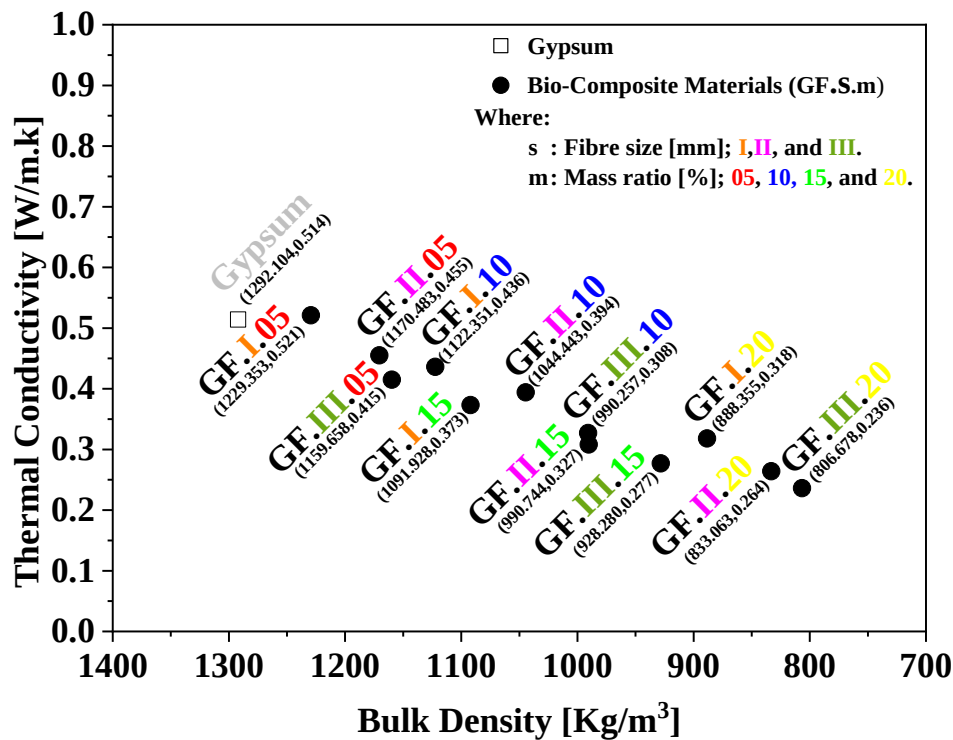


**Figure IV.15. Thermal Conductivity-Bulk Density Results of Gypsum vs. Bio-Composite Materials (GF.s.m).**

**Figure IV.15.** Shows the thermal conductivity-bulk density test results of gypsum-based bio-composite materials (GF.s.m) containing different mass ratios (m: 05-20%) of Date Palm Fibers (DPFs) used as reinforcement for each size (s: I-III) of fiber, showing significant differences in thermal conductivity and bulk density. The thermal conductivity and bulk density generally decrease with increasing mass ratio (m: 05-20%) of Date Palm Fiber (DPF) and size (s: I-III). This can be attributed to the fact that natural fibers reduce bulk density and environmental impact, as confirmed by studies [372, 476]. These changes

can be attributed to the effect of fiber inclusion on the microstructure and thermal properties of bio-composite materials by reducing them, as confirmed by study [477]. Among the bio-composite materials (GF.s.05) containing a (m: 05%) Date Palm Fiber (DPF) mass ratio, the thermal conductivity and bulk density decrease with increasing Date Palm Fiber (DPF) size (s: I-III). As observed at a Date Palm Fiber (DPF) mass ratio (m: 20%), bio-composite materials (GF.III.20) had the lowest conductivity and bulk density ( $0.236 \pm 0.003$  W/m.k and  $806.678 \pm 16.085$  kg/m<sup>3</sup>), respectively, as shown in **Tables IV.02, and 03**. This trend shows that making the fiber mass ratio and size larger improves the material's insulating characteristics by adding more voids and lowering its overall thermal conductivity and bulk density, as confirmed by study [477]. In addition, these results may also align with studies [3, 478], which confirmed that the bio-composite materials (GF.s.m) that have larger-sized (s: III) Date Palm Fibers (DPFs) are better at breaking up heat transmission routes because they make the material more porous. In addition, among the specimens studied, plain gypsum had the highest thermal conductivity ( $0.514 \pm 0.003$  W/mK) and bulk density ( $1292.104 \pm 19.413$  kg/m<sup>3</sup>), as shown in **Tables IV.02, and 03**. This is primarily due to its low voids and uniform matrix, as confirmed by studies [215, 479].

Furthermore, for bio-composite materials (GF.I.m), the thermal conductivity-bulk density decreases from ( $0.521 \pm 0.002$  W/m.k -  $1229.353 \pm 19.154$  kg/m<sup>3</sup>) at a (m: 05%) mass ratio of Date Palm Fiber (DPF) to mass ratio of (m: 20%) to ( $0.318 \pm 0.001$  W/m.k -  $888.355 \pm 17.492$  kg/m<sup>3</sup>), as shown in **Tables IV.02, and 03**. The bio-composite materials (GF.II.m. and GF.III.m) exhibit similar trends to those of (GF.I.m). In addition, these results may also align with studies [258, 480], which confirmed that the material has a lower bulk density because the bigger fibers make it more porous. The fact that Date Palm Fiber (DPF) are lighter than gypsum and the holes that are made when fibers are added to the matrix are also reasons for this decline in bulk density, as confirmed by studies [11, 364].

Furthermore, the Date Palm Fiber (DPF) size significantly influences the properties of bio-composite materials (GF.s.m). The smaller (s: I) Date Palm Fibers (DPFs) result in higher thermal conductivity and bulk density compared to larger (s: III) size Date Palm Fibers (DPFs). Larger fibers (s: III) also disrupt matrix continuity more effectively, increasing porosity, reducing bulk density, and thus reducing thermal conductivity, as confirmed by studies [395, 481]. In addition, the natural nature of natural fibers also creates small air gaps, which disrupts heat transfer and enhances thermal resistance, as confirmed by study [395]. Meanwhile, the incorporation of natural fibers, such as Date Palm Fiber (DPF), into gypsum composites is part of a growing trend toward environmentally friendly building materials, as confirmed

by studies [3, 17, 364]. In addition, these results may also align with studies [474, 482], which confirmed that these composites offer lower thermal conductivity, making them suitable for applications requiring enhanced thermal insulation. The significant reductions in density further highlight their potential for lightweight construction, as confirmed by studies [483, 484].

Ultimately, thermal conductivity and bulk density test results show that adding more Date Palm Fiber (DPF) and making it bigger (s: I-III) greatly enhances thermal insulation and lowers the bulk density of gypsum bio-composites (GF.s.m), improving them for building that lasts. These results are in line with many other scholarly studies that show that natural fiber-reinforced bio-composites could be useful, light, and eco-friendly building materials.

## IV.9. Chemical Results

Chemical results were obtained after conducting experiments on specific specimens of bio-composite materials (GF.s.m), with the aim of obtaining the chemical composition and chemical bonds that could explain the mechanical, physical, and thermal results. The chemical results were as follows:

### IV.9.1. Energy Dispersive X-ray Spectroscopy (EDS) Results

**Table IV.04.** Shows the Energy Dispersive X-ray Spectroscopy (EDS) data, which shows the weight and atomic ratios of the elements Carbon (C), Nitrogen (N), Oxygen (O), Sulfur (S), and Calcium (Ca) for three separate specimens of the bio-composite materials (GF.s.m), called (GF.I.20, GF.III.15, and GF.III.05), which are the minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{3P \text{ Max}}$ ), respectively.

The tested specimens were tested according to **Table IV.01**, where:

- The specimen (GF.I.20) was selected because it had the minimum flexural modulus value among the bio-composite materials (GF.s.m) specimens studied;
- The specimen (GF.III.05) was selected because it had the maximum flexural modulus value among the bio-composite materials (GF.s.m) specimens studied;
- The specimen (GF.III.15) was selected because it had the mean flexural modulus value among the bio-composite materials (GF.s.m) specimens studied.

Because, according to the Equation (**IV.01**):

$$\text{Calculated } E_{3P \text{ Mean}} = \frac{\text{Measured } E_{3P \text{ Max}} - \text{Measured } E_{3P \text{ Min}}}{2} = \frac{1209.741 - 81.353}{2} = 564.194 \text{ MPa} \quad (\text{IV.01})$$

Where:

- **Measured  $E_{3P \text{ Min}}$ :** The bio-composite material (GF.I.20) specimen with the minimum measured flexural modulus value [MPa];
- **Calculated  $E_{3P \text{ Mean}}$ :** The mean calculated flexural modulus value [MPa];
- **Measured  $E_{3P \text{ Max}}$ :** The bio-composite material (GF.III.05) specimen with the maximum measured flexural modulus value [MPa].

According to Equation (IV.01), since the calculated mean value of the flexural modulus was 564.194 MPa, according to Equations (IV.02, and 03), it can be said that the mean value of the flexural modulus is consistent with the bio-composite material (GF.III.15) specimen since it is closer to the value 564.194 MPa.

$$|\text{Calculated } E_{3P \text{ Mean}} - \text{Measured } E_{3P \text{ GF.II.15}}| = |564.194 - 437.975| = 126.219 \text{ MPa} \quad (\text{IV.02})$$

(Is Not Closed to Cal  $E_{3P \text{ Mean}}$  Value)

$$|\text{Calculated } E_{3P \text{ Mean}} - \text{Measured } E_{3P \text{ GF.III.15}}| = |564.194 - 667.197| = 103.003 \text{ MPa} \quad (\text{IV.03})$$

(Is Closed to Calculated  $E_{3P \text{ Mean}}$  Value)

Because:

$$|\text{Calculated } E_{3P \text{ Mean}} - \text{Measured } E_{3P \text{ GF.II.15}}| > |\text{Calculated } E_{3P \text{ Mean}} - \text{Measured } E_{3P \text{ GF.III.15}}| \quad (\text{IV.04})$$

Where:

- **Calculated  $E_{3P \text{ Mean}}$ :** The Mean Calculated Flexural Modulus Value [MPa];
- **Measured  $E_{3P \text{ GF.II.15}}$ :** The Bio-composite Material (GF.II.15) Specimen with the Measured Flexural Modulus Value [MPa];
- **Measured  $E_{3P \text{ GF.III.15}}$ :** The Bio-composite Material (GF.III.15) Specimen with the Measured Flexural Modulus Value [MPa].

### Note:

The closest two values were ( $437.975 \pm 11.588$  -  $667.197 \pm 45.744$  MPa), which are consistent with the two bio-composite material specimens (GF.II.15 and GF.III.15), respectively, as shown in Table IV.01.

- The specimen (GF.III.05) was selected because it had the maximum flexural modulus value among the bio-composite materials (GF.s.m) specimens studied.

**Table IV.04. The Quantified Weight and Atomic rate of the Elemental Makeup of Bio-composite Materials (GF.s.m).**

|                            | Atom                            | Carbon (C)      | Nitrogen (N) | Oxygen (O) | Sulfur (S) | Calcium (Ca) |
|----------------------------|---------------------------------|-----------------|--------------|------------|------------|--------------|
|                            | Bio-composite Material (GF.s.m) | Weight [%]      |              |            |            |              |
| <b>E<sub>3P</sub> Min</b>  | <b>GF.I.20</b>                  | 07.68           | 06.18        | 45.37      | 20.21      | 20.42        |
| <b>E<sub>3P</sub> Mean</b> | <b>GF.III.15</b>                | 04.33           | 04.20        | 48.37      | 19.85      | 23.26        |
| <b>E<sub>3P</sub> Max</b>  | <b>GF.III.05</b>                | 14.09           | 07.10        | 53.80      | 10.54      | 14.39        |
|                            | Atom                            | Carbon (C)      | Nitrogen (N) | Oxygen (O) | Sulfur (S) | Calcium (Ca) |
|                            | Bio-composite Material (GF.s.m) | Atomic rate [%] |              |            |            |              |
| <b>E<sub>3P</sub> Min</b>  | <b>GF.I.20</b>                  | 12.64           | 08.72        | 56.04      | 12.64      | 10.07        |
| <b>E<sub>3P</sub> Mean</b> | <b>GF.III.15</b>                | 07.38           | 06.14        | 61.92      | 12.68      | 11.89        |
| <b>E<sub>3P</sub> Max</b>  | <b>GF.III.05</b>                | 20.46           | 08.84        | 58.64      | 05.73      | 06.26        |

Where:

- **E<sub>3P</sub> Min:** The Specimen with the Minimum Value of the Flexural Modulus was Selected;
- **E<sub>3P</sub> Mean:** The Specimen with the Mean Value of the Flexural Modulus was Selected;
- **E<sub>3P</sub> Max:** The Specimen with the Maximum Value of the Flexural Modulus was Selected.

**Table IV.04.** Shows that the bulk of gypsum is calcium sulfate ( $\text{CaSO}_4$ ), thus containing a high amount of calcium (Ca), as confirmed by studies [485, 486]. Meanwhile, the bio-composite materials (GF.I.20, GF.III.15, and GF.III.05) contain carbon (C), nitrogen (N), and oxygen (O). In addition, these results may also align with studies [11, 17], which confirmed that this indicates that the inorganic gypsum matrix has been reinforced with organic materials, such as Date Palm Fiber (DPF). Furthermore, this is particularly true for the bio-composite materials (GF.s.m) containing more Date Palm Fiber (DPF). Meanwhile, the observed trend in bio-composite materials (GF.III.05) (The Specimen with Fewer Less Date Palm Fibers (DPFs)) indicates the highest carbon content (14.09%), as it is reinforced with larger-sized (s: III) Date Palm Fibers (DPFs). In addition, these results confirmed that as the mass ratio (m: 05%) of Date Palm Fiber (DPF) decreases, the overall composition changes, and the calcium (Ca) (14.39%) level decreases, indicating a lower gypsum content and the presence of voids within the bio-composite material (GF.III.05). Meanwhile, because the bio-composite material (GF.III.05) has a smaller amount of gypsum and palm fibers, although they occupy the same volume, we get a lower apparent density.

Furthermore, **Table IV.04.** Shows that the atomic distribution of elements shows a clear change in the relative amounts of carbon (C) and calcium (Ca). Meanwhile, the bio-composite material (GF.I.20) has a greater atomic percentage of oxygen (O) (56.04%), which could mean that there are more cellulose or other organic molecules that contain oxygen in the Date Palm Fibers (DPFs), as confirmed by study [487]. The more Date Palm Fiber (DPF) there is, the more carbon and oxygen there is in the bio-composite material (GF.I.20), as confirmed by studies [3, 443, 488]. Meanwhile, on the other hand, the bio-composite material (GF.III.05), which has the least amount of Date Palm Fiber (DPF), contains the least calcium (C) (06.26%).

The results of **Table IV.04.** for the Energy Dispersive X-ray Spectroscopy (EDS) test results can also be related to the results shown in **Table IV.01, 02, and 03** for the mechanical, physical, and thermal results, respectively, as follows:

- 1. Mechanical Properties:** The calcium (Ca) concentration increases with decreasing Date Palm Fiber (DPF) mass fraction (m: 0.5-20%), as shown in **Table IV.04.** This means that the flexural strength and modulus increase. Meanwhile, for example, the bio-composite material (GF.III.05) (Which Contains the Least Amount of Date Palm Fiber (DPF)) has significantly higher flexural strength and elastic modulus ( $3.263 \pm 0.115$  -  $1209.741 \pm 0.431$  MPa) than the bio-composite material (GF.I.20) ( $0.294 \pm 0.015$  -  $81.353 \pm 0.685$  MPa), as shown in **Table IV.04.** Gypsum adds additional calcium (Ca) to the material, making it more Brinell hardness value ( $31.626 \pm 0.245$  MPa) resistant and less prone to flexural modulus at the lowest strain at break of the bio-composite material (GF.III.05) ( $4.615 \pm 0.245$  mm/mm), indicating a tougher and more deformation-resistant material, which is consistent with its high calcium and sulfur (Ca, and S) (20.24 and 20.21 %) content and low organic Date Palm Fiber (DPF) mass ratio, as confirmed by studies [489, 490]. Meanwhile, the bio-composite material (GF.III.05) also has the highest compressive strength at ( $10.807 \pm 0.097$  MPa). This is because the gypsum matrix, which is the main component of the material, is compressive, as confirmed by studies [15, 384]. In addition, the bio-composite material (GF.III.05) has a lower Charpy impact strength ( $2.067 \pm 0.077$  KJ/m<sup>2</sup>), mainly due to its lower fiber mass ratio (m: 05%), carbon, nitrogen and oxygen (C, N, and O) (20.46, 08.84 and 58.64 %) content, as confirmed by studies [491, 492]. The same trend applies to bio-composite materials (GF.I.20 and GF.III.15);

- 2. Physical Properties:** The trend in bulk density shows that as the fiber mass ratio goes down, the inorganic content goes up. In addition, the highest density ( $1159.658 \pm 18.208 \text{ Kg/m}^3$ ) is in the bio-composite material (GF.III.05), which is because it has more calcium sulfate (Gypsum), as confirmed by study [493]. Furthermore, the higher organic Date Palm Fiber (DPF) mass ratio (m: 15-20%) in bio-composite material (GF.I.20) ( $888.355 \pm 17.492 \text{ kg/m}^3$ ) and bio-composite material (GF.III.15) ( $928.280 \pm 15.912 \text{ kg/m}^3$ ) makes the bulk density lower, which means that the fibers make the composite less dense overall, which proves the difference in the ratio carbon (C), nitrogen (N), and oxygen (O), as confirmed by studies [3, 11]. Meanwhile, the fact that bio-composite material (GF.I.20) has more organic material (Date Palm Fiber (DPF)) and carbon (C) (14.09%) in it makes it more likely to absorb water and swell ( $44.827 \pm 1.264 - 2.567 \pm 0.049\%$ ), respectively, as confirmed by study [37]. In addition, the bio-composite material (GF.III.05) has less Date Palm Fiber (DPF) and calcium and sulfur (Ca and S) (06.26 and 05.73 %) content, which means it absorbs less water ( $40.030 \pm 2.521 \%$ ) and swells and moisture content less when wet ( $7.477 \pm 0.072 - 12.944 \pm 0.511 \%$ ), as confirmed by studies [3, 17]. Meanwhile, this means it is more resistant to moisture because it is mostly made of gypsum. The same trend applies to bio-composite materials (GF.I.20 and GF.III.15);
- 3. Thermal Properties:** Thermal conductivity results show a similar pattern to the bulk density results. Furthermore, the bio-composite material (GF.III.05) has the highest carbon, nitrogen and oxygen (C, N and O) (14.09, 07.10 and 53.80 %) content, indicating the presence of high organic Date Palm Fibers (DPFs), and the best thermal conductivity ( $0.415 \pm 0.002 \text{ W/m}\cdot\text{K}$ ), as confirmed by studies [17, 494]. In addition, this means that the material's ability to transfer heat is more dependent on the palm fibers, as confirmed by studies [495, 496]. Furthermore, the low calcium and sulfur (Ca and S) (14.39 and 10.54 %) content also makes the bio-composite material (GF.III.05) more porous, which improves thermal conductivity, as confirmed by study [497]. In addition, it is likely that the Date Palm Fibers (DPFs) in the bio-composite material (GF.I.20 and GF.III.15) make them less thermally conductive ( $0.318 \pm 0.001$  and  $0.277 \pm 0.003 \text{ W/m}\cdot\text{K}$ ), respectively. Meanwhile, the bio-composite material (GF.III.05) has the highest specific heat capacity ( $885.000 \pm 04.667 \text{ J/kg}\cdot\text{K}$ ), meaning it can retain and store more thermal energy due to its high carbon, nitrogen and oxygen (C, N and O) (14.09, 07.10 and 53.80 %) content, as confirmed by studies [498, 499]. Furthermore, this is a common feature in materials containing a



lot of inorganic material, such as gypsum. The same trend applies to bio-composite materials (GF.I.20 and GF.III.15).

Ultimately, the data show that the size (s: I-III) and mass ratio (m: 05-20%) of Date Palm Fiber (DPF) are critical factors in changing the bio-composite's materials (GF.s.m) properties for specific uses, such as insulation or building materials. Modern materials science research could balance mechanical, thermal, and environmental performance to improve these bio-composites for many uses.

#### IV.9.2. Fourier Transform Infrared Spectroscopy (FT-IR) Results

**Figure IV.16.** Shows the FT-IR test results. The results will look at the functional groups and general spectrum properties of the gypsum-date palm fiber bio-composite materials' (GF.s.m) Fourier Transform Infrared Spectroscopy (FT-IR). This is especially true for bio-composite materials (GF.I.20, GF.III.15, and GF.III.05), which have the minimum, mean, and maximum flexural modulus values ( $E_{3P \text{ Min}}$ ,  $E_{3P \text{ Mean}}$ , and  $E_{P3 \text{ Max}}$ ), respectively.

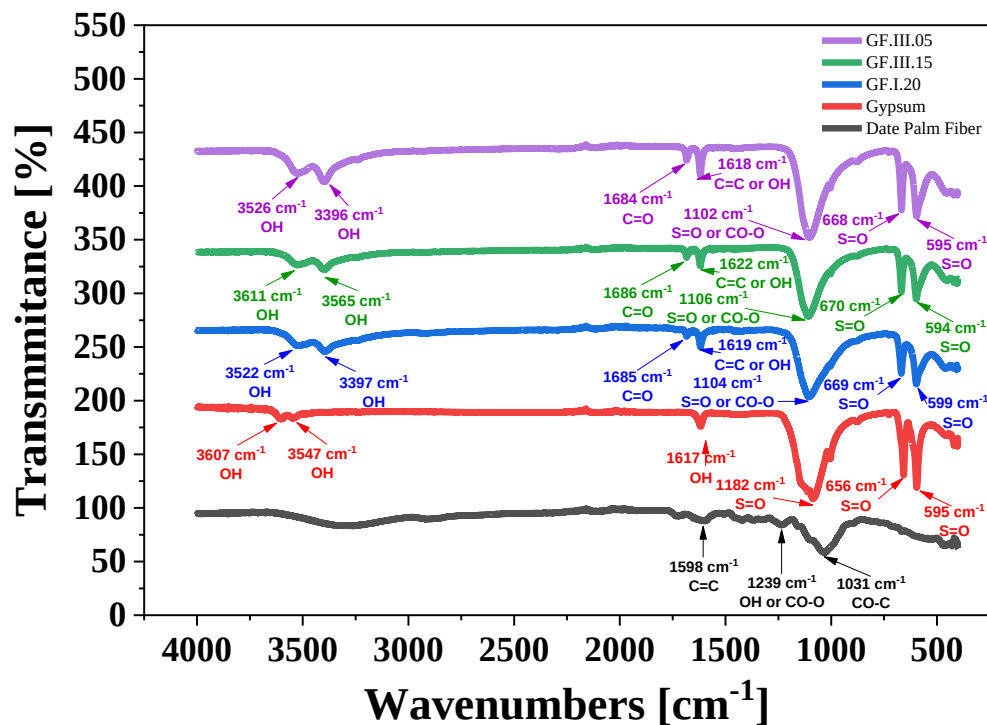


**Figure IV.16. Fourier Transform Infrared Spectroscopy (FT-IR) Results.**

**Figure IV.16.** Shows that all the spectra have a strong peak between 3611 and 3396  $\text{cm}^{-1}$ . This peak is related to the O-H stretching vibrations that are common in hydroxyl groups (OH), as confirmed by studies [500, 501]. In addition, this characteristic is a common trait of all bio-composite materials

(GF.I.20, GF.III.05, and GF.III.15), which means that both the gypsum and Date Palm Fiber (DPF) parts have hydroxyl groups in them. However, another noticeable signal between  $1634$  and  $1684\text{ cm}^{-1}$  shows C=O stretching vibrations, which are probably caused by carbonyl groups, as confirmed by studies [502, 503]. In addition, the peaks might be ester or carboxyl groups that form when the gypsum matrix and the Date Palm Fiber (DPF) come into contact with each other, as confirmed by study [504, 505]. However, peaks around  $1102$ - $1022\text{ cm}^{-1}$  and  $585$ - $595\text{ cm}^{-1}$  show S=O stretching, which is common for gypsum's sulfate bonds, as confirmed by study [506]. In addition, this finding further supports the idea that the gypsum is stable in the bio-composite materials (GF.I.20, GF.III.05, and GF.III.15).

Furthermore, the bio-composite material (GF.I.20) has the strongest O-H stretch, which means that it interacts with moisture or hydroxyl groups in the Date Palm Fiber (DPF) more strongly, as confirmed by studies [507, 508]. In addition, the sulfate groups (S=O) are visible, which means that the gypsum matrix is still intact and has not broken down too much, as confirmed by study [509]. However, the O-H stretch is still observable in the bio-composite material (GF.III.05). In addition, the spectrum is less pronounced than in the bio-composite material (GF.I.20). However, this result could mean that the Date Palm Fiber (DPF) and gypsum do not interact as much, which could be because there is less Date Palm Fiber (DPF) in the mix. In addition, the C=O peak gets stronger, which could mean that there are some chemical interactions at the interface, including ester or other carbonyl-related connections, as confirmed by studies [510, 511]. However, the bio-composite material (GF.III.15) still has the S=O stretch, but the peak intensities change, which shows that the matrix has formed differently since there are more Date Palm Fibers (DPFs) in it. In addition, this variation could mean that the Date Palm Fiber (DPF) is better spread out in the gypsum matrix, but it could also mean that the Date Palm Fiber (DPF) is breaking down or reacting with gypsum, as the C=O stretching peak has changed, as confirmed by studies [3, 23].

Furthermore, the O-H group is present in all of the bio-composite materials (GF.I.05, GF.III.05, and GF.III.15), which suggests that hydrogen bonding or moisture interaction is happening in the bio-composite, as confirmed by study [512]. In addition, the C=O groups, especially in the bio-composite materials (GF.III.05 and GF.III.15), show that the fibers and gypsum may be chemically bonded, which could change the mechanical properties, such as the flexural modulus, as confirmed by study [513].

Furthermore, the bio-composite material (GF.I.20), which has the smallest size (s: I) of Date Palm Fibers (DPFs) and the highest Date Palm Fiber (DPF) mass ratio (m: 20%), is likely to have the best mechanical performance. In addition, this is because it has strong hydroxyl and sulfate groups that could help the matrix link better, as confirmed by studies [246, 514]. However, the bio-composite material (GF.III.05),

which has large-sized (s: III) Date Palm Fibers (DPFs) and a lower Date Palm Fiber (DPF) mass ratio (m: 05%), would probably have a lower flexural modulus. In addition, this is because the Fourier Transform Infrared Spectroscopy (FT-IR) spectrum shows that the Date Palm Fiber (DPF) and gypsum don't interact as strongly, as confirmed by studies [11, 515]. However, the bio-composite material (GF.III.15), which has a larger-sized (s: III) of Date Palm Fibers (DPFs) and a mass ratio (m: 15%), may perform better overall. In addition, its greater proportion of Date Palm Fibers (DPFs) may strengthen it when bent, as confirmed by studies [516, 517]. However, its Fourier Transform Infrared Spectroscopy (FT-IR) spectrum shows that the date palm fiber-gypsum interaction is slightly weaker than that of bio-composite material (GF.I.20), as confirmed by studies [364, 515].

Ultimately, the Fourier Transform Infrared Spectroscopy (FT-IR) analyses give us critical information about how the molecules in the gypsum-date palm fiber bio-composite materials (GF.s.m) interact with each other. The functional groups seen, such as hydroxyl (O-H), carbonyl (C=O), and sulfate (S=O), show that the Date Palm Fibers (DPFs) and the gypsum matrix interact in different ways. This data is consistent with the mechanical qualities (Flexural Modulus) that were discovered. Specifically, the bio-composite material (GF.III.05) composite probably has the best mechanical qualities since the fibers are larger and the mass ratio of Date Palm Fiber (DPF) is less, which makes it easier for the gypsum to interact with the Date Palm Fibers (DPFs).

## IV.10. Morphological Results

Morphological results were obtained to confirm the interpretations of mechanical, physical, and thermal results regarding bio-composite materials (GF.s.m). The morphological results were as follows:

### IV.10.1. Scanning Electron Microscopy (SEM) Results

Scanning Electron Microscopy (SEM) images of the surface section of the fracture process after the three-point flexural test are depicted in [Figure IV.17](#). are magnified at 500 x and 85 x, respectively, and depict the specimens with the lowest and highest recorded flexural modulus values.

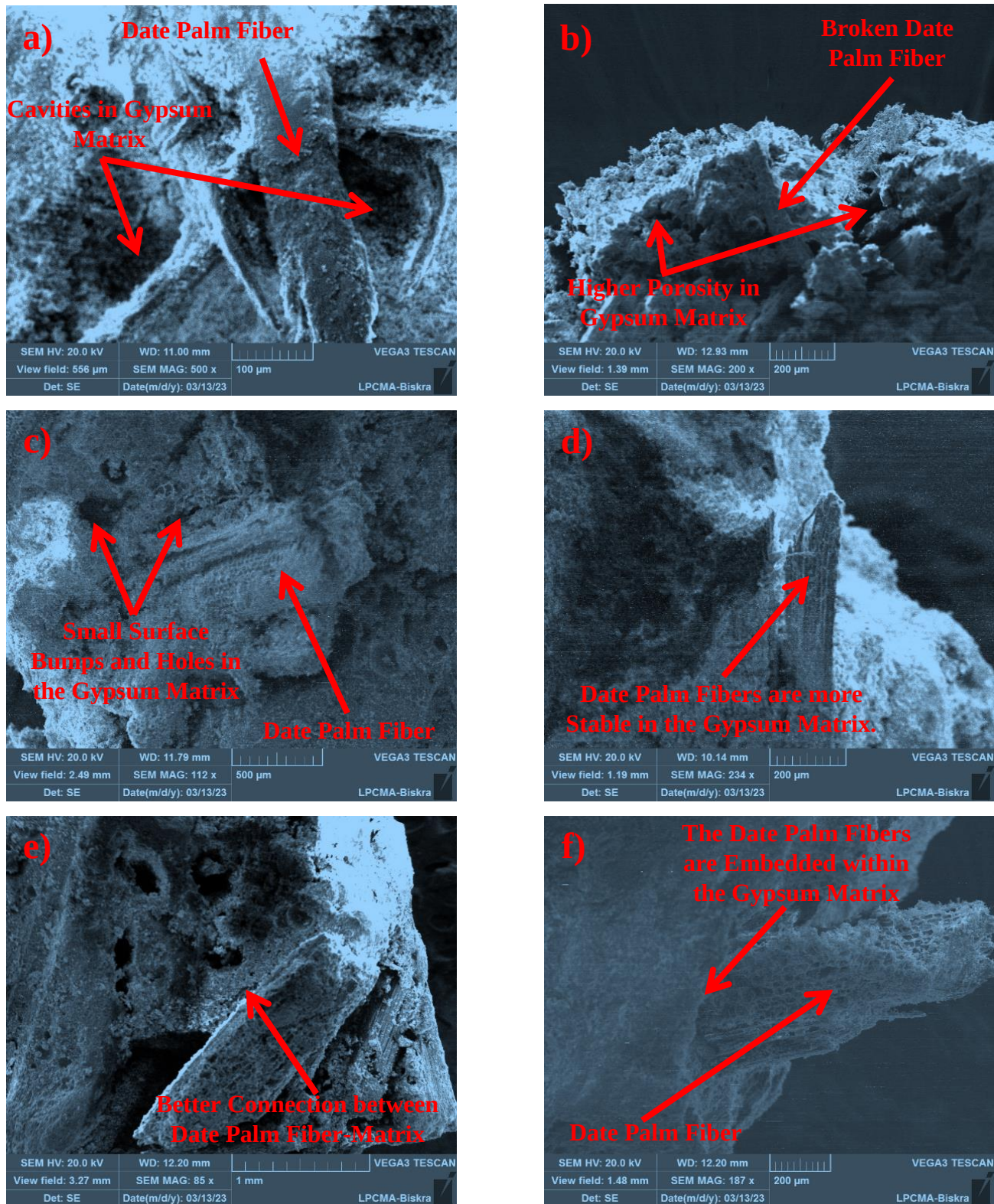


**Figure IV.17. SEM Results: a) and b) The Specimen that has a Minimum Flexural Modulus Value, c) and d) The Specimen that has a Medium Flexural Modulus Value, e) and f) The Specimen that has a Maximum Flexural Modulus Value.**



**Figure IV.17.a.** Shows that in the specimen with the minimum flexural modulus value, the complex interaction between the Date Palm Fibers (DPFs) and the gypsum matrix is highlighted, and the Date Palm Fiber (DPF) interface and diffusion distance are clearly visible. In addition, even though the Date Palm Fibers (DPFs) are spread out quite well, some of them are pulling out, which suggests that the bond between the Date Palm Fibers (DPFs) and the gypsum matrix might be a weak point in the bio-composite material's (GF.I.20) strength when it is under strain, as confirmed by studies [12, 513]. However, there are also cavities and unequal bonding, which could make the bio-composite material (GF.I.20) less useful for structural purposes, as confirmed by study [37].

Furthermore, **Figure IV.17.b.** Shows that in the specimen with the minimum flexural modulus value, the surface looks rougher and more broken up, which could mean that the bio-composite material (GF.I.20) failed or broke apart during the flexural test. In addition, the higher porosity and broken sections are linked to the lowest flexural strength seen, which suggests strain localization, micro-cracking, and early failure of the bio-composite material (GF.I.20). However, these problems could be because the gypsum matrix doesn't cling well to the Date Palm Fibers (DPFs) or because it is excessively brittle, as confirmed by study [259].

Furthermore, **Figure IV.17.c.** Shows that in the specimen with the medium flexural modulus value, this tighter link between the Date Palm Fiber (DPF) and the gypsum matrix makes the bio-composite's material (GF.III.15) better at withstanding bending stress, which makes its mechanical performance better. In addition, it shows small surface bumps and holes in the gypsum matrix that don't have a big effect on the bio-composite's material (GF.III.15) strength at a medium flexural value, but they could make it weaker in some situations, as confirmed by studies [385, 518].

Furthermore, **Figure IV.17.d.** Shows that in the specimen with the medium flexural modulus value, the Date Palm Fiber (DPF) is more firmly implanted than in the lower-flexural modulus bio-composite material (GF.I.20). In addition, there are signs of imperfect bonding at the borders of the Date Palm Fibers (DPFs), where they are partially broken and pulled out. However, the fibers' rough surface helps them link together mechanically, which makes them stick better, as confirmed by studies [519, 520]. In addition, micro-cracks along the date palm fiber-matrix interface could mean that stress is building up in some areas, which could make the bio-composite material (GF.III.15) weaker when they are under strain, as confirmed by studies [521, 522]. However, these results indicate opportunities for improvement in

processing conditions to guarantee a more uniform Date Palm Fiber (DPF) dispersion and improved date palm fiber-matrix integration.

Furthermore, **Figure IV.17.e**. Shows that the specimen with the maximum flexural modulus value shows that the gypsum matrix has been badly damaged, with cracks apparent around the Date Palm Fibers (DPFs), which is common in bio-composite materials, as confirmed by studies [63, 523]. In addition, it can show the date palm fiber-matrix contact, and some of the Date Palm Fibers (DPFs) are stuck to the gypsum matrix. However, the situation shows how strong the bio-composite material (GF.III.05) is because the Date Palm Fibers (DPFs) and the matrix connect well enough, as confirmed by studies [3, 524].

Furthermore, **Figure IV.17.f**. Shows that the sample with the maximum flexural modulus value exhibits a detailed fracture location that indicates the Date Palm Fibers (DPFs) are only partially pushed out of the gypsum matrix, particularly at the fracture edges. In addition, it also indicates that the Date Palm Fibers (DPFs) are embedded within the gypsum matrix, allowing the bio-composite material (GF.III.05) to distribute stress evenly, and that the adhesion between the Date Palm Fibers (DPFs) and the gypsum matrix is sufficiently strong, as confirmed by studies [11, 259]. However, these data suggest the potential for improving date palm fiber-matrix adhesion and gypsum matrix structure to enhance the overall performance of the bio-composite material (GF.III.05) and reduce its brittleness.

Ultimately, the SEM results show that bio-composite materials (GF.I.20, GF.III.15, and GF.III.05) could be useful in real life, but their mechanical properties and resistance to bending stress need to be improved by making the date palm fiber-matrix adhesion stronger, the Date Palm Fiber (DPF) distribution better, and the gypsum matrix formulation better.

## Conclusion

In conclusion, this fourth chapter has provided a thorough overview of the results of the gypsum-date palm fiber bio-composites (GF.s.m), denoted by different mass ratios (m: 05-20%) for each Date Palm Fiber (DPF) sizes (s: I-III), provide valuable insights into how the addition of Date Palm Fibers (DPFs) affects the mechanical, physical, thermal, thermos-physical, chemical, and morphological properties of the bio-composite. The results are displayed as follows:

**1. Mechanical Properties:** The addition of Date Palm Fibers (DPFs) as reinforcement to gypsum affects the flexural strength of bio-composites (GF.s.m). The higher the mass ratios (m: 05-20%) of Date Palm Fibers (DPFs), the lower the flexural strength of bio-composites (GF.s.m). Conversely, the higher the size (s: I-III) of Date Palm Fibers (DPFs), the higher the flexural strength of bio-composites (GF.s.m). In general, the addition of Date Palm Fibers (DPFs) as reinforcement to gypsum for making bio-composites (GF.s.m) weakens their flexural strength, as lower values are recorded than those of the gypsum specimen used as a comparison. The bio-composite materials specimens (GF.I.20 and GF.III.05) recorded the lowest and highest flexural strength values of ( $0.294 \pm 0.015$  and  $3.263 \pm 0.115$  MPa), with a decrease of (92.82 and 20.36%), respectively, compared to the gypsum specimen. Also, with increasing the mass ratio (m: 05-20%) of Date Palm Fibers (DPFs), the flexural modulus of the bio-composites (GF.s.m) decreased. While increasing the size (s: I-III) of the reinforcing Date Palm Fibers (DPFs), the flexural modulus of the bio-composite materials (GF.s.m) was higher. In general, the bio-composites (GF.s.m) have a lower flexural modulus than the gypsum specimen. The lowest and highest flexural modulus values of the bio-composites (GF.s.m) were recorded in the specimens (GF.I.20 and GF.III.05) with values of ( $81.353 \pm 06.685$  and  $1209.741 \pm 04.131$  MPa), respectively, with a decrease of (93.75 and 07.00%) compared to the gypsum specimen.

In addition, the compressive strength and compressive modulus of the bio-composite materials (GF.s.m) decreased with increasing the mass ratio (m: 05-20%) of the reinforcing Date Palm Fibers (DPFs). Whereas, the compressive strength and compressive modulus of the bio-composite materials (GF.s.m) reinforced with medium, larger, and smaller (II, III, and I) Date Palm Fibers (DPFs) was higher, respectively. In general, the bio-composite materials (GF.s.m) reinforced with medium-sized (s: II) Date Palm Fibers (DPFs) had higher compressive strength and compressive modulus values than the gypsum specimens at mass ratios (m: 05-10%). In contrast, most of the other bio-composite materials (GF.s.m) specimens had lower compressive strength and compressive modulus. The lowest and highest compressive strength and compressive modulus values of the bio-composite materials (GF.s.m) were found in the specimens (GF.I.20 and GF.II.05) with values of ( $1.329 \pm 0.261$  and  $11.964 \pm 0.687$  MPa) and ( $204.873 \pm 034.447$  and  $1279.945 \pm 025.613$  MPa), respectively, with a decrease and increase ratio of (81.01 and 70.96%), respectively, compared to the gypsum specimen.

The hardness of the bio-composite materials (GF.s.m) increased with increasing mass ratio (m: 05-20%) when reinforced with smaller-sized (s: I) Date Palm Fibers (DPFs). However, when the bio-composite materials (GF.s.m) were reinforced with medium and larger size (s: II and III) Date Palm Fibers (DPFs), and with increasing mass ratio (m: 05-20%), the Brinell surface hardness of the bio-composite materials (GF.s.m) decreased. At mass ratios (m: 05-10%), the Brinell surface hardness of the bio-composite materials (GF.s.m) increased with increasing size of (s: I-III) Date Palm Fibers (DPFs) used as reinforcement. In general, the bio-composite materials (GF.s.m) had lower Brinell hardness values than the gypsum specimen. The lowest and highest Brinell hardness values of bio-composite materials (GF.s.m) were in specimens (GF.I.05 and GF.III.05) with values of  $(14.097 \pm 0.074)$  and  $(31.626 \pm 0.245)$  MPa and a decrease and increase of (39.44 and 35.86%) respectively compared to the gypsum specimen.

With increasing mass ratio (m: 05-20%) of Date Palm Fibers (DPFs) as reinforcement for bio-composite materials (GF.s.m), the Charpy impact strength decreases when reinforced with smaller (s: I) size Date Palm Fibers (DPFs). Whereas, for bio-composite materials (GF.s.m) reinforced with medium and larger (s: II and III) size of Date Palm Fibers (DPFs), the Charpy impact strength of the specimens increases at mass ratios (m: 05-15%) and then decreases at mass ratios (m: 20%). Generally, the Charpy impact strength of bio-composite materials (GF.s.m) increases when reinforced with Date Palm Fibers (DPFs). The lowest and highest values of Charpy impact strength of bio-composite materials (GF.s.m) were found in specimens (GF.I.20 and GF.II.15) with values of  $(0.894 \pm 0.002)$  and  $(8.005 \pm 0.756)$  KJ/m<sup>2</sup>, with a decrease and increase percentage estimated at (30.32 and 523.93%) compared to the gypsum specimen.

Finally, in general, most of the bio-composite materials (GF.s.m) show lower mechanical properties than the gypsum specimen. Still, the bio-composite material (GF.I.05) specimen reinforced with the largest (s: III) size of Date Palm Fibers (DPFs) and the lowest mass ratio (m: 05%) shows the best compressive strength, Brinell hardness, and Charpy impact strength, while the flexural strength and modulus are the lowest.

- 2. Physical Properties:** The bulk density was lower with increasing mass ratio (m: 05-20%) and the size (s: I-III) of Date Palm Fiber (DPF) added as reinforcement to gypsum for making bio-composite materials (GF.s.m). In general, bio-composite materials (GF.s.m) showed lower bulk density compared to the gypsum specimen. The lowest and highest bulk density values of bio-composite materials (GF.s.m) were found in specimens (GF.III.20 and GF.I.05) with values



of  $(806.678 \pm 16.085$  and  $1229.353 \pm 19.154 \text{ kg/m}^3)$  and a decrease of (37.57 and 4.86%) compared to the gypsum specimen.

The water absorption rate was higher with increasing mass ratio (m: 05-20%) and the size (s: I-III) of Date Palm Fibers (DPFs) added as reinforcement to gypsum for making bio-composite materials (GF.s.m). In general, bio-composite materials (GF.s.m) showed a higher water absorption rate compared to the gypsum specimen. The lowest and highest water absorption rate values for bio-composite materials (GF.s.m) were found in (GF.I.05 and GF.III.20) with values of  $(34.878 \pm 3.082$  and  $50.708 \pm 1.267\%)$ , with a decrease and increase ratio of (20.05 and 16.24%) compared to the gypsum specimen.

The thickness swelling ratio was higher with increasing mass ratio (m: 05-20%) and size (s: I-III) of Date Palm Fibers (DPFs) added as reinforcement to gypsum for making bio-composite materials (GF.s.m). In general, the bio-composite materials (GF.s.m) showed a higher thickness swelling ratio compared to the gypsum specimen. The lowest and highest thickness swelling ratios for bio-composite materials (GF.s.m) specimens were found in specimens (GF.I.05 and GF.III.20) with values of  $(1.549 \pm 0.158$  and  $17.426 \pm 0.083\%)$ , with an increase of (111.78 and 2282.24%) compared to the gypsum specimen.

The moisture content ratio was higher with increasing mass ratio (m: 05-20%) and size (s: I-III) of Date Palm Fibers (DPFs) added as reinforcement to gypsum for making bio-composite materials (GF.s.m). In general, the bio-composite materials (GF.s.m) showed a higher moisture content ratio compared to the gypsum specimen. The lowest and highest moisture content ratios for bio-composite materials (GF.s.m) specimens were found in specimens (GF.I.05 and GF.III.20) with values of  $(12.547 \pm 0.681$  and  $14.453 \pm 0.634\%)$ , with an increase of (6.80 and 23.03%) compared to the gypsum specimen.

Finally, in general, most bio-composite materials (GF.s.m) exhibit improved physical properties in terms of bulk density and water absorption ratio compared to the gypsum specimen. Meanwhile, the thickness swelling ratio and moisture content ratio are higher for bio-composite materials (GF.s.m) than for the gypsum specimen.

- 3. Thermal Properties:** The results showed a decrease in thermal conductivity and specific heat in bio-composite materials (GF.s.m) with increasing mass ratio (m: 05-20%) and size (s: I-III) of Date Palm Fiber (DPF) added as reinforcement to gypsum for making bio-composite materials

(GF.s.m). At the same time, the thermal diffusivity shows the opposite results of thermal conductivity and specific heat. In general, the bio-composite materials (GF.s.m) show the lowest values of thermal conductivity and specific heat with the highest values of thermal diffusivity compared to the gypsum specimen. The lowest and highest values of thermal conductivity and specific heat in bio-composite materials (GF.s.m) were in specimens (GF.III.20 and GF.I.05) with values of  $(0.236 \pm 0.003$  and  $0.521 \pm 0.002$  W/m.k) and  $(503.667 \pm 0.556$  and  $1113.667 \pm 0.422$  J/kg.k) with a decrease and increase rate of (54.09 and 1.36%) and (54.09 and 1.52%), respectively, compared to the gypsum specimen. While the thermal diffusion of bio-composite materials (GF.s.m) recorded its lowest and highest values in specimens (GF.I.05 and GF.III.20) with values of  $(3.810 \pm 0.061$  and  $5.811 \pm 0.121$  m<sup>2</sup>/s) and an increase rate of (5.13 and 60.35%) compared to the gypsum specimen.

**4. Thermo-Physical Properties:** The thermo-physical results of the bio-composite materials (GF.s.m) represented by thermal conductivity-bulk density showed consistent results with the mass ratio (m: 0.5-20%) and Date Palm Fiber (DPF) size (s: I-III). With increasing the mass ratio (m: 0.5-20%) and Date Palm Fiber (DPF) size (s: I-III), the thermal conductivity-bulk density values decreased in the bio-composite materials (GF.s.m). In general, the bio-composite materials (GF.s.m) showed lower values of thermal conductivity-bulk density compared to the gypsum specimen. The lowest and highest values of thermal conductivity-bulk density among the bio-composite materials (GF.s.m) were in specimens (GF.III.20 and GF.I.05) with values of  $(0.236 \pm 0.003$  W/m.k -  $806.678 \pm 16.085$  Kg/m<sup>3</sup> and  $0.521 \pm 0.002$  W/m.k -  $1229.353 \pm 19.154$  Kg/m<sup>3</sup>) and a decrease of (54.09 - 37.57% and 01.36 - 04.86%) compared to the gypsum specimen.

**5. Chemical Properties:** Energy Dispersive X-ray Spectroscopy (EDS) results showed that the composition and properties of bio-composite materials (GF.s.m) composed of Date Palm Fibers (DPFs) and gypsum were characterized by their elemental content, showing varying levels of carbon (C), nitrogen (N), oxygen (O), sulfur (S), and calcium (Ca). The results indicate that the addition of Date Palm Fibers (DPFs) increases the organic content, particularly by increasing the levels of carbon (C), nitrogen (N), and oxygen (O), with an increase in the mass ratio (m: 05-20%) of the Date Palm Fibers (DPFs), which is consistent with the mechanical (Lower Flexural Modulus, Lower Compressive Strength, Lower Surface Hardness, and Higher Impact Strength), physical (Light Weight, Lower Water Absorption, Higher Thickness Swelling Upon Exposure to Moisture, and Increased Moisture Content), and thermal (Lower Thermal

Conductivity) results. While high levels of sulfur (S) and calcium (Ca) characterize the bio-composite materials (GF.s.m), a lower mass ratio (m: 05-20%) of Date Palm Fibers (DPFs) results in poorer mechanical, physical, and thermal properties. Energy-dispersive X-ray spectroscopy (EDS) results indicate that modifying the mass ratio (m: 05-20%) and size (s: I-III) of Date Palm Fibers (DPFs) can significantly alter their elemental content and hence the mechanical, physical, and thermal properties of bio-composite materials (GF.s.m), making them suitable for a variety of applications, including insulation and construction.

The Fourier Transform Infrared Spectroscopy (FT-IR) results of bio-composite materials (GF.I.20, GF.III.05, and GF.III.15) indicate substantial molecular interactions between gypsum and Date Palm Fibers (DPFs). A pronounced peak ranging from 3526 to 3396  $\text{cm}^{-1}$ , linked to O-H stretching vibrations, is evident in all materials, signifying the existence of hydroxyl groups. Also, the C=O stretching between 1634 and 1684  $\text{cm}^{-1}$  implies that carbonyl groups were formed at the fiber-matrix contact. The peaks for S=O stretching, which are common in gypsum's sulfate bonds, show that the gypsum matrix is stable. The peaks' strength and position change, which suggests that the way fibers and matrices interact depends on the size and mass ratio of the fibers. The bio-composite material with bigger-sized Date Palm Fibers (DPFs) and a lower mass ratio (GF.III.05) has stronger interactions and better mechanical properties. The bio-composite material with smaller-sized Date Palm Fibers (DPFs) and a bigger mass ratio (GF.I.20) has weaker interactions, which makes it less flexible. These results shed light on the relationship between molecular structure and the mechanical properties of bio-composite materials (GF.s.m).

**6. Morphological Properties:** The Scanning Electron Microscopy (SEM) results of bio-composite materials (GF.I.20, GF.III.15, and GF.III.05) show that the mechanical properties are different because of how the Date Palm Fibers (DPFs) and the gypsum matrix interact. The specimen with the lowest flexural modulus (GF.I.20) shows that the Date Palm Fibers (DPFs) do not connect well with the gypsum matrix. This causes the fibers to pull out, causing the material to collapse in a brittle way with more holes. These things make the material's flexural strength poor, which shows that the fiber-matrix adhesion needs to be better. The medium flexural modulus specimen (GF.III.15) has a better bond between the fibers and the matrix, which improves mechanical performance even when the surface is not flawless. However, micro-cracking at the fiber-matrix interface indicates stress concentration, which could make the material weaker when it is under strain. The specimen with the highest flexural modulus

(GF.III.05) shows that the fibers are better embedded in the gypsum matrix, which leads to a more even distribution of stress and stronger strength. Even though these results are good, the gypsum matrix is showing signs of brittleness and degradation, which means that there is still potential for improvement in the way the fibers are spread out and the way the matrix is made. In general, these bio-composite materials (GF.I.20, GF.III.15, and GF.III.05) look promising, but to make them better for practical use, we need to increase the adhesion, distribution, and composition of the fibers and matrix.

Ultimately, the fourth chapter shows that the gypsum-date palm fiber bio-composites (GF.s.m) show promise for energy-efficient and sustainable construction. Their suitability depends on balancing the benefits of thermal insulation and lightweight properties with the challenges posed by reduced mechanical strength and moisture susceptibility.

## Conclusion

In conclusion, the second part of this thesis has provided a comprehensive exploration of the fabrication, evaluation, and performance of bio-composite materials (GF.s.m) made by reinforcing gypsum with Date Palm Fibers (DPFs).

The third chapter presented a detailed methodology for preparing and evaluating these bio-composites, including the processes of extracting and sizing the palm fibers and integrating them with gypsum. The various testing methods, ranging from mechanical to physical, thermal, chemical, and morphological tests, were systematically employed to understand the impact of different fiber sizes (s: I-III) and mass ratios (m: 05-20%) on the performance of the bio-composites.

The fourth chapter further extended the analysis, presenting a detailed interpretation of the results obtained from the various tests. It highlighted the relationship between the mass ratio (m: 05-20%) of the fibers and the properties of the bio-composites, with specific emphasis on how different sizes (s: I-III) of Date Palm Fibers (DPFs) influenced their performance. The fourth chapter underscored that while the addition of Date Palm Fibers (DPFs) typically led to a decrease in mechanical strength, it also contributed to improvements in thermal insulation and reduced density and water absorption, making the bio-composites promising for energy-efficient and sustainable construction. However, challenges such as increased thickness swelling and moisture absorption were also noted, necessitating careful consideration when selecting the appropriate bio-composite for specific applications.

Ultimately, the second part contributes valuable knowledge on the development and optimization of gypsum-date palm fiber bio-composites, paving the way for their potential use in eco-friendly construction and insulation materials.

**Part 3**

**Statistical Analysis and Optimization**

**Study**

## Introduction

In part three, the focus shifts to the advanced methodologies and optimization techniques used to evaluate and enhance the performance of bio-composite materials and models under study. This third part combines the principles of regression modeling and statistical analysis with the development of bio-composite materials designed for high performance, sustainability, and cost-effectiveness in industrial applications.

The fifth chapter outlines a comprehensive approach to regression modeling and statistical analysis, focusing on regression techniques such as linear, quadratic and pure quadratic models. It emphasizes the importance of assessing model performance through various metrics, including p-values, Standard Errors (SE), and goodness-of-fit statistics like Root Mean Squared Error (RMSE) and R-squared. These statistical tools, alongside Multi-Objective Optimization (MOO) techniques, are applied to ensure robust, reliable analysis and optimal model fitting. The fifth chapter also showcases the use of MATLAB for implementing regression models, analyzing data, and providing predictions, all while ensuring the validity of results through statistical rigor.

The sixth chapter, on the other hand, extends the discussion to the optimization of bio-composite materials, with a particular emphasis on properties such as impact strength, water absorption, and thermal conductivity. Through a Multi-Objective Optimization (MOO) approach, this chapter explores how conflicting material properties can be optimized simultaneously to achieve the best balance for specific industrial applications. Utilizing tools like Analysis of Variance (ANOVA) and Pareto optimization, it presents tailored solutions for optimizing bio-composite materials, specifically those combining Date Palm Fibers (DPFs) and gypsum matrix. This optimization process ensures that the bio-composite materials meet required performance standards, paving the way for their use in diverse engineering applications.

Ultimately, these third part provide a comprehensive methodology for both statistical modeling and bio-composite material optimization, illustrating how data-driven approaches can enhance both theoretical analysis and practical material design.

The third part can be summarized by the hierarchy, as shown in [Figure 04](#).

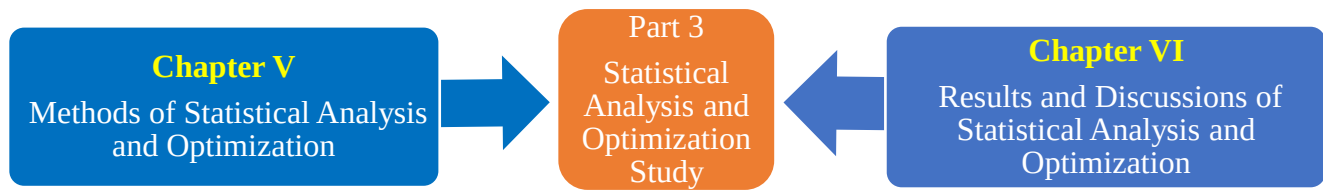


**Figure 04. Graphical Abstract of the Third Part of the Thesis.**



# Chapter V

## Methods of Statistical Analysis and Optimization

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## Introduction

In this fifth chapter, the methodology employed to evaluate and analyze the data is outlined, with a primary focus on regression models and statistical tools. The aim is to establish the most suitable models for the analysis and evaluate their performance through several metrics and techniques. The fifth chapter begins with the data collection process, followed by an exploration of different regression models, including linear and quadratic approaches, and their respective methods for estimating coefficients.

A critical section of the fifth chapter delves into statistical analysis, including the estimation of coefficients, Standard Errors (SE), and t-statistics, which provide insight into the reliability and significance of the models. The p-values associated with these estimations allow for the evaluation of hypothesis tests, which is crucial for understanding the validity of the models. Model fit statistics, such as Root Mean Squared Error (RMSE), R-squared, and F-statistic, are also examined to assess the overall accuracy and robustness of the regression models.

The Analysis of Variance (ANOVA) is another key aspect of the fifth chapter, offering a deeper understanding of how the models explain the variability within the data. The Sum of Squares (SumSq), degrees of freedom, and the associated F-statistics are calculated to determine the goodness of fit and the significance of the predictors.

Further, the fifth chapter includes a section on Multi-Objective Optimization (MOO), where the Pareto optimality and the Pareto front are discussed. This provides insights into how multiple objectives can be optimized simultaneously, considering trade-offs between them. The practical application of these methods is demonstrated through the use of MATLAB, which is utilized for data analysis, model fitting, and prediction generation. The MATLAB code is provided to show how the data is processed, regression models are implemented, and performance metrics are calculated. The final sections offer predictions and the corresponding output, concluding with an overall evaluation of the methodology's effectiveness.

Ultimately, this fifth chapter aims to provide a comprehensive approach to statistical analysis and regression modeling, serving as the foundation for the conclusions drawn in the subsequent chapter.

### V.1. Data Collection

The dataset utilized in this study consists of experimental measurements that describe the relationship between the size of Date Palm Fibers (DPFs) (As Reinforcement) and their Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) behavior in bio-composite materials. The

independent variables include the Date Palm Fiber (DPF) size (Denoted as  $x_1$ ) and the date palm fiber-to-matrix mass ratio (Denoted as  $x_2$ ). In contrast, the dependent variable is the measured Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) of the bio-composite material.

### 1. Independent Variables:

- a. **Fiber Size ( $x_1$ ):** The size of the Date Palm Fiber (DPF) used as reinforcement was categorized into three distinct groups: 0.080, 0.315, and 0.500 mm. These sizes are representative of the variations in Date Palm Fiber (DPF) lengths typically encountered in bio-composite materials.
- b. **Fiber-to-Matrix Mass Ratio ( $x_2$ ):** The mass ratio of Date Palm Fiber (DPF) to matrix (Gypsum) was varied across three levels: 05, 10, and 15%. This ratio significantly influences the mechanical, physical and thermal properties of bio-composite materials, including Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) behavior, respectively.

### 2. Dependent Variable:

- a. **Charpy Impact Strength (Measured):** The dependent variable is the Charpy impact strength (G) capacity of the bio-composite material, measured experimentally for each combination of Date Palm Fiber (DPF) size ( $x_1$ ) and fiber-to-matrix mass ratio ( $x_2$ ). The Charpy impact strength (G) data were collected under controlled laboratory conditions, ensuring consistency and reliability of the measurements.
- b. **Water Absorption (Measured):** The dependent variable is the water absorption (WAR) capacity of the bio-composite material, measured experimentally for each combination of Date Palm Fiber (DPF) size ( $x_1$ ) and fiber-to-matrix mass ratio ( $x_2$ ). The water absorption (WAR) data were collected under controlled laboratory conditions, ensuring consistency and reliability of the measurements.
- c. **Thermal Conductivity (Measured):** The dependent variable is the thermal conductivity ( $\lambda$ ) capacity of the bio-composite material, measured experimentally for each combination of Date Palm Fiber (DPF) size ( $x_1$ ) and fiber-to-matrix mass ratio ( $x_2$ ). The thermal conductivity ( $\lambda$ ) data were collected under controlled laboratory conditions, ensuring consistency and reliability of the measurements.

Ultimately, the dataset consists of nine data points, with each point corresponding to a unique combination of Date Palm Fiber (DPF) size ( $x_1$ ) and fiber-to-matrix mass ratio ( $x_2$ ), and the associated Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) value.

## V.2. Regression Models

The primary objective of this study was to develop statistical models that could predict the Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) of date palm fiber-reinforced bio-composites based on the Date Palm Fiber (DPF) size ( $x_1$ ) and fiber-to-matrix mass ratio ( $x_2$ ). Three different regression models were considered: a linear regression model, a quadratic regression model, and a pure quadratic regression model.

**1. Linear Model Equation:** The linear model equation, as shown in Equation (V.01):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 \quad (\text{V.01})$$

Where:

- **y:** The Dependent Variable (Response) [-];
- **$x_1$  and  $x_2$ :** Independent Variables (Predictors) [-];
- **$\beta_0$ :** The Intercept [-];
- **$\beta_1$  and  $\beta_2$ :** The Coefficients for Respectively [-].

The model suggests that the dependent variable is linearly related to ( $x_1$ ) and ( $x_2$ ), along with a constant intercept term.

**2. Quadratic Model Equation:** The quadratic model equation, as shown in Equation (V.02):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_1 x_2 + \beta_4 x_1^2 + \beta_5 x_2^2 \quad (\text{V.02})$$

Where:

- **y:** The Dependent Variable (Response) [-];
- **$x_1$  and  $x_2$ :** The Independent Variables (Predictors) [-];
- **$x_1^2$  and  $x_2^2$ :** The Quadratic Terms of the Predictors [-];
- **$\beta_0, \beta_1, \beta_2, \beta_3, \beta_4$  and  $\beta_5$ :** The Parameters to be Estimated [-].

**3. Pure Quadratic Model Equation:** The pure quadratic model equation, as shown in Equation (V.03):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_1^2 + \beta_4 x_2^2 \quad (\text{V.03})$$

Where:

- **y** : The Dependent Variable (Response) [-];
- **x<sub>1</sub> and x<sub>2</sub>**: The Independent Variables (Predictors) [-];
- **x<sub>1</sub><sup>2</sup> and x<sub>2</sub><sup>2</sup>**: The Quadratic Terms of the Predictors [-];
- **β<sub>0</sub>, β<sub>1</sub>, β<sub>2</sub>, β<sub>3</sub> and β<sub>4</sub>**: The Parameters to be Estimated [-].

Ultimately, this developed statistical models to predict the Charpy impact strength (G), water absorption (WAR), and thermal conductivity (λ) of date palm fiber-reinforced bio-composites. These models provide useful insights into the influence of fiber properties on bio-composite performance, offering a foundation for optimizing material design.

### V.3. Statistical Analysis

Statistical analysis is the process of collecting, organizing, interpreting, and presenting data to identify patterns, relationships, and trends. It involves applying various mathematical and statistical methods to assess the validity of hypotheses, draw conclusions, or make predictions based on the data. Statistical analysis can be descriptive (Summarizing Data) or inferential (Drawing Conclusions or Testing Hypotheses Based on Specimen Data). It is widely used in fields such as research, business, economics, healthcare, and social sciences to make informed decisions, improve processes, or validate scientific findings.

#### V.3.1. Estimated Coefficients (β) or (p)

The estimated coefficients in a regression model are the values that best fit the model to the data. These coefficients are determined by the least squares estimation method, which minimizes the sum of the squared differences between the observed values and the values predicted by the model.

##### V.3.1.1. General Method for Estimating Coefficients

To estimate the coefficients, the used of the Ordinary Least Squares (OLS) method was used, which minimizes the following sum of squared residuals, as shown in Equation (V.04):

$$RSS = \sum_{i=1}^n (y_i - \hat{y}_i)^2 \quad (\text{V.04})$$

Where:

- **RSS:** The Sum of Squared Residuals [-];
- **y<sub>i</sub>:** The Observed Values [-];
- **$\hat{y}_i$ :** The Predicted Values Based on the Model [-];
- **n:** The Number of Data Points [-].

### V.3.1.2. Linear Model Coefficients Estimation

The linear model is presented, as shown in Equation (V.05):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 \quad (\text{V.05})$$

Where:

- **y:** The Dependent Variable (Response) [-];
- **x<sub>1</sub> and x<sub>2</sub>:** Independent Variables (Predictors) [-];
- **$\beta_0$ :** The Intercept [-];
- **$\beta_1$  and  $\beta_2$ :** The Coefficients for Respectively [-].

The matrix form of this model can be written, as shown in Equations (V.06, 07, and 08):

$$y = X \beta + \epsilon \quad (\text{V.06})$$

$$X = \begin{bmatrix} 1 & x_{1,1} & x_{2,1} \\ 1 & x_{1,2} & x_{2,2} \\ \vdots & \vdots & \vdots \\ 1 & x_{1,n} & x_{2,n} \end{bmatrix} \quad (\text{V.07})$$

$$\beta = \begin{bmatrix} \beta_0 \\ \beta_1 \\ \beta_2 \end{bmatrix} \quad (\text{V.08})$$

Where:

- **y:** The Vector of Observed Values [y<sub>1</sub>, y<sub>2</sub>, ... y<sub>n</sub>] [-];
- **X:** The Design Matrix that Includes the Independent Variables x<sub>1</sub> and x<sub>2</sub> Augmented with a Column of Ones for the Intercept [-];
- **$\beta$ :** The Vector of Coefficients to be Estimated [-];

- $\epsilon$ : The Vector of Residuals (Errors) between the Observed and Predicted Values [-].

The least squares solution for  $\beta$  is obtained, as shown in Equation (V.09)

$$\beta = (X^T X)^{-1} X^T y \quad (\text{V.09})$$

Where:

- $X^T$ : The Transpose of the Design Matrix [-];
- $(X^T X)^{-1}$ : The Inverse of the Matrix Product  $X^T X$  [-];
- $X^T y$ : The Matrix Product of the Transposed Design Matrix and the Observed Values Vector [-].

Ultimately, this equations gives the estimated coefficients  $\beta$ , including the intercept,  $\beta_0$  and the coefficients for  $x_1$  and  $x_2$ .

### V.3.1.3. Quadratic Model Coefficients Estimation

The quadratic model is presented, as shown in Equation (V.10):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_1 x_2 + \beta_4 x_1^2 + \beta_5 x_2^2 \quad (\text{V.10})$$

Where:

- $y$ : The Dependent Variable (Response) [-];
- $x_1$  and  $x_2$ : The Independent Variables (Predictors) [-];
- $x_1^2$  and  $x_2^2$ : The Quadratic Terms of the Predictors [-];
- $\beta_0, \beta_1, \beta_2, \beta_3, \beta_4$  and  $\beta_5$ : The Parameters to be Estimated [-].

The matrix approach and the design matrix  $X$  now includes the linear terms  $x_1, x_2$  and the quadratic terms  $x_1^2, x_2^2$ . The matrix is presented, as shown in Equations (V.11, and 12):

$$X = \begin{bmatrix} 1 & x_{1,1} & x_{2,1} & x_{1,1}^2 & x_{2,1}^2 \\ 1 & x_{1,2} & x_{2,2} & x_{1,2}^2 & x_{2,2}^2 \\ \vdots & \vdots & \vdots & \vdots & \vdots \\ 1 & x_{1,n} & x_{2,n} & x_{1,n}^2 & x_{2,n}^2 \end{bmatrix} \quad (\text{V.11})$$

The coefficients are then estimated using the same equation:

$$\beta = (X^T X)^{-1} X^T y \quad (\text{V.12})$$



Where:

- $\beta$  : Now Includes  $\beta_0, \beta_1, \beta_2, \beta_3, \beta_4$  and  $\beta_5$  [-];
- $\mathbf{X}^T$ : The Transpose of the Design Matrix [-];
- $(\mathbf{X}^T\mathbf{X})^{-1}$ : The Inverse of the Matrix Product  $\mathbf{X}^T\mathbf{X}$  [-];
- $\mathbf{X}^T\mathbf{y}$ : The Matrix Product of the Transposed Design Matrix and the Observed Values Vector [-].

#### V.3.1.4. Pure-Quadratic Model Coefficients Estimation

The pure-quadratic model is presented, as shown in Equation (V.13):

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_1^2 + \beta_4x_2^2 \quad (\text{V.13})$$

Where:

- $y$  : The Dependent Variable (Response) [-];
- $x_1$  and  $x_2$ : The Independent Variables (Predictors) [-];
- $x_1^2$  and  $x_2^2$ : The Quadratic Terms of the Predictors [-];
- $\beta_0, \beta_1, \beta_2, \beta_3$  and  $\beta_4$ : The Parameters to be Estimated [-].

In pure-quadratic model, the design matrix  $\mathbf{X}$  only includes the intercept and the quadratic terms, as shown in Equation (V.14):

$$\mathbf{X} = \begin{bmatrix} 1 & x_{1,1}^2 & x_{2,1}^2 \\ 1 & x_{1,2}^2 & x_{2,2}^2 \\ \vdots & \vdots & \vdots \\ 1 & x_{1,n}^2 & x_{2,n}^2 \end{bmatrix} \quad (\text{V.14})$$

Where:

- $\mathbf{X}$  : The Design Matrix [-];
- $x_1^2$  and  $x_2^2$ : The Quadratic Terms of the Predictors [-].

The coefficients  $\beta_0, \beta_1, \beta_2, \beta_3$  and  $\beta_4$  are estimated using the least squares formula, as shown in Equation (V.15):

$$\beta = (\mathbf{X}^T\mathbf{X})^{-1}\mathbf{X}^T\mathbf{y} \quad (\text{V.15})$$

Where:

- $\beta$  : Now Includes  $\beta_0, \beta_1, \beta_2, \beta_3$ , and  $\beta_4$  [-];
- $X^T$ : The Transpose of the Design Matrix [-];
- $(X^T X)^{-1}$ : The Inverse of the Matrix Product  $X^T X$  [-];
- $X^T y$ : The Matrix Product of the Transposed Design Matrix and the Observed Values Vector [-].

Ultimately, the estimated coefficients in linear, quadratic, and pure-quadratic models are determined using the least squares estimation method. This involves forming a design matrix based on the variables and solving the normal Equations ( $\beta = (X^T X)^{-1} X^T y$ ) to get the coefficient estimates. These coefficients are then used to predict the response variable and assess the model's fit to the data.

### V.3.2. Standard Error (SE)

The Standard Error (SE) of the coefficients in a regression model quantifies the variability or precision of the estimated coefficients. In other words, it indicates how much the estimated coefficient values would vary if you were to repeatedly specimen from the population and fit a regression model to each specimen.

In regression analysis, SE is calculated using the residuals (errors) of the model, the design matrix, and the number of observations.

The Standard Error (SE) of the coefficients is calculated, as shown in Equation (V.16):

$$SE(\beta) = \sqrt{\hat{\sigma}^2 \cdot (X^T X)^{-1}} \quad (\text{V.16})$$

Where:

- $SE(\beta)$ : The Standard Error of the Coefficients  $\beta$  [-];
- $\hat{\sigma}^2$ : The Estimated Variance of the Residuals [-];
- $X$ : The Design Matrix (Which Contains the Values of the Independent Variables) [-];
- $(X^T X)^{-1}$ : The Inverse of the Matrix Product of  $X^T$  (The Transpose of the Design Matrix) and  $X$  [-].

To calculate the SE, the regression model using the least squares method and obtaining the residuals is the first step. The residuals  $\epsilon_i$  are the differences between the observed values  $y_i$  and the predicted values  $\hat{y}_i$ , as shown in Equation (V.17):

$$\epsilon_i = y_i - \hat{y}_i \quad (\text{V.17})$$

Where:

- $\epsilon_i$ : The Residuals [-];
- $y_i$ : The Observed Values [-];
- $\hat{y}_i$ : The Predicted Values Based on the Model [-].

The residuals represent the error or deviation of the observed values from the predicted values.

The residual variance  $\hat{\sigma}^2$  is the mean squared residual, and it is calculated, as shown in Equation (V.18):

$$\hat{\sigma}^2 = \frac{\sum_{i=1}^n \epsilon_i^2}{n - p} \quad (\text{V.18})$$

Where:

- $\hat{\sigma}^2$ : The Estimated Variance of the Residuals [-];
- $\epsilon_i$ : The Residuals [-];
- $n$ : The Number of Observations [-];
- $p$ : The Number of Coefficients (Including the Intercept) in the Model [-].

**Note:** The denominator is  $n-p$ , which is the number of degrees of freedom. This accounts for the fact that the model has  $p$  coefficients, and we lose one degree of freedom for each coefficient we estimate.

Ultimately, the Standard Error (SE) of the coefficients in a regression model serves as a measure of the variability or precision of the estimated coefficients. It reflects how much the estimated values would fluctuate if the regression model were repeatedly fitted to different samples from the population, allowing one to judge the reliability of the model's estimates.

### V.3.3. t-Statistic (tStat)

The t-statistic is used to determine if a coefficient is significantly different from zero (Null Hypothesis). It's calculated by dividing the coefficient estimate by its Standard Error (SE), as shown in Equation (V.19):

$$tStat = \frac{\beta}{SE(\beta)} \quad (V.19)$$

Where:

- **tStat:** The t-statistic [-];
- **$\beta$ :** The Coefficients for Respectively [-];
- **SE ( $\beta$ ):** The Standard Error of the Coefficients  $\beta$  [-].

Ultimately, a higher absolute t-statistic means the corresponding coefficient is more likely to be significantly different from zero.

### V.3.4. p-Value (p-value)

The p-value in regression analysis helps to determine whether a particular independent variable (Or Predictor) has a statistically significant relationship with the dependent variable. It is used to test the null hypothesis that the coefficient of the independent variable is equal to zero, meaning that the variable does not have an effect on the dependent variable.

For each regression coefficient  $\beta_j$  (Where: j: The Index of the Coefficient), we perform the following hypothesis tests:

1. **Null Hypothesis ( $H_0$ ):**  $\beta_j = 0$ , meaning that the coefficient for  $x_j$  has no effect (The Independent Variable  $x_j$  does not Influence y ).
2. **Alternative Hypothesis ( $H_1$ ):**  $\beta_j \neq 0$ , meaning that the coefficient for  $x_j$  is significantly different from zero (The Independent Variable  $x_j$  has a Significant Effect on y).

The goal is to test whether we can reject the null hypothesis based on the observed data. A small p-value (Typically below a Significance Level, such as 0.05) indicates strong evidence against the null hypothesis, suggesting that the coefficient is significantly different from zero.

Once we have the t-statistic, we calculate the p-value using the t-distribution. The p-value is the probability of observing a t-statistic as extreme as the one calculated, assuming that the null hypothesis is true.

The p-value is calculated as the two-tailed probability from the t-distribution, as shown in Equation (V.20):

$$p - \text{value} = 2 \times (1 - \text{CDF}(|t_j|)) \quad (\text{V.20})$$

Where:

- **p – value:** The p-value [-];
- **CDF:** The Cumulative Distribution Function of the t-Distribution [-];
- **|t<sub>j</sub>|:** The Absolute Value of the Calculated t-Statistic [-].

This formula gives the probability that the observed t-statistic would occur under the null hypothesis. The factor of two accounts for the fact that we are conducting a two-tailed test (Testing for both Positive and Negative Deviations from Zero).

The t-statistic is used to test the null hypothesis for each coefficient. It is calculated, as shown in Equation (V.21):

$$t_j = \frac{\hat{\beta}_j}{\text{SE}(\hat{\beta}_j)} \quad (\text{V.21})$$

Where:

- **t<sub>j</sub>:** The t-Statistic [-];
- **$\hat{\beta}_j$ :** The Estimated Coefficient [-];
- **SE( $\hat{\beta}_j$ ):** The Standard Error of the Estimated Coefficient [-].

Ultimately, the t-statistic measures how many Standard Errors (SE) the estimated coefficient is away from zero. A larger absolute t-statistic suggests that the coefficient is further from zero, increasing the likelihood of rejecting the null hypothesis.

The p-value is a critical value used to assess the significance of each coefficient in a regression model. Smaller p-values (Typically Less Than 0.05) indicate that the coefficient is significantly different from zero and that the independent variable has an effect on the dependent variable. Larger p-values suggest that the variable may not have a significant effect on the outcome.

In the examples provided, you would repeat the above calculations for each of the coefficients  $\beta_0$ ,  $\beta_1$ ,  $\beta_3$ , etc., in the linear, quadratic, and pure-quadratic models to evaluate their significance.

## V.4. Model Fit Statistics

Model fit statistics refer to a set of measures used to evaluate how well a statistical model explains or fits the observed data. These statistics help determine whether the model is appropriate and provides an accurate representation of the relationships within the data.

### V.4.1. Root Mean Squared Error (RMSE)

The Root Mean Squared Error (RMSE) is a commonly used metric to evaluate the performance of a regression model. It provides a measure of how well the model's predictions match the observed data. The Root Mean Squared Error (RMSE) gives the standard deviation of the residuals (Errors), or in simpler terms, it tells us how far off our model's predictions are from the actual data points.

The Root Mean Squared Error (RMSE) is calculated, as shown in Equation (V.22):

$$\text{RMSE} = \sqrt{\frac{1}{n} \sum_{i=1}^n (y_i - \hat{y}_i)^2} \quad (\text{V.22})$$

Where:

- $y_i$ : The Actual Observed Value for the i-th Data Point [-];
- $\hat{y}_i$ : The Predicted Value for the i-th Data Point, Calculated from the Regression Model [-];
- $n$ : The Total Number of Observations (Data Points) [-];
- $(y_i - \hat{y}_i)$ : The Residual or Error for the i-th Observation [-];
- $(y_i - \hat{y}_i)^2$ : The Squares each Residual, which Eliminates Negative Values and Gives more Weight to Larger Errors [-].

Ultimately, the Root Mean Squared Error (RMSE) provides a useful, interpretable measure of prediction accuracy, and it is particularly helpful when comparing different regression models.

### V.4.2. R-squared ( $R^2$ )

The R-squared (Denoted  $R^2$ ) is a statistical measure that indicates how well the regression model explains the variation in the dependent variable. In simple terms, it tells us what proportion of the variance in the dependent variable can be explained by the independent variables in the model.

The R-squared is calculated, as shown in Equation (V.23):

$$R^2 = 1 - \frac{SS_{\text{Residual}}}{SS_{\text{Total}}} \quad (\text{V.23})$$

Where:

- **$R^2$** : The R-squared [-];
- **$SS_{\text{Residual}}$** : The Residual is the Residual Sum of Squares (SumSq) (Also Known as the Error Sum of Squares, RSS) [-];
- **$SS_{\text{Total}}$** : The Total is the Total Sum of Squares (SumSq) [-].

**1. Residual Sum of Squares ( $SS_{\text{Residual}}$ ):** The residual Sum of Squares (SumSq) measures the variation in the dependent variable that the model does not explain. It is calculated as the sum of squared differences between the observed values and the predicted values, as shown in Equation (V.24):

$$SS_{\text{Residual}} = \sum_{i=1}^n (y_i - \hat{y}_i)^2 \quad (\text{V.24})$$

Where:

- **$SS_{\text{Residual}}$** : The Residual Sum of Squares (SumSq) [-];
- **$y_i$** : The Observed Value of the Dependent Variable [-];
- **$\hat{y}_i$** : The Predicted Value of the Dependent Variable (From the Regression Model) [-];
- **$n$** : The Number of Observations [-].

**2. Total Sum of Squares ( $SS_{\text{Total}}$ ):** The total Sum of Squares (SumSq) measures the total variation in the dependent variable around its mean. It is calculated as the sum of squared differences between the observed values and the mean of the observed values, as shown in Equation (V.25):

$$SS_{\text{Total}} = \sum_{i=1}^n (y_i - \bar{y}_i)^2 \quad (\text{V.25})$$

Where:

- **SS<sub>Total</sub>**: The Total Sum of Squares (SumSq) [-];
- **y<sub>i</sub>**: The Observed Value of the Dependent Variable [-];
- **$\bar{y}_i$** : The Mean of the Observed Values of the Dependent Variable [-].

**3. Sum of Squares (SS<sub>Regression</sub>):** The explained Sum of Squares (SumSq) measures the variation explained by the regression model. It is the difference between the Total Sum of Squares (SS<sub>Total</sub>) and the residual Sum of Squares (SumSq), as shown in Equation (V.26):

$$SS_{Regression} = SS_{Total} - SS_{Residual} \quad (V.26)$$

Where:

- **SS<sub>Regression</sub>**: The Regression Sum of Squares (SumSq) [-];
- **SS<sub>Total</sub>**: The Total Sum of Squares (SumSq) [-];
- **SS<sub>Residual</sub>**: The Residual Sum of Squares (SumSq) [-].

The R-squared ( $R^2$ ) is the proportion of the total variation explained by the model, as shown in Equation (V.27):

$$R^2 = \frac{SS_{Regression}}{SS_{Total}} \quad (V.27)$$

Where:

- **$R^2$** : The R-squared [-];
- **SS<sub>Regression</sub>**: The Regression Sum of Squares (SumSq) [-];
- **SS<sub>Total</sub>**: The Total Sum of Squares (SumSq) [-].

Ultimately, the R-squared value helps to assess the goodness of fit of the model and indicates how well the independent variables explain the variation in the dependent variable.

#### V.4.3. Adjusted R-squared ( $R^2_{Adj}$ )

The Adjusted R-squared ( $R^2_{Adj}$ ) is a modification of the R-squared statistic that adjusts for the number of predictors (Independent Variables) in a regression model. While R-squared always increases or stays the same when more predictors are added to a model (Even if those Predictors are Irrelevant), Adjusted



R-squared accounts for this and provides a more accurate measure of model fit by penalizing the inclusion of irrelevant predictors.

The adjusted R-squared is calculated, as shown in Equation (V.28):

$$R^2_{Adj} = 1 - \left( \frac{(1 - R^2) \cdot (n - 1)}{n - p - 1} \right) \quad (\text{V.28})$$

Where:

- $R^2_{Adj}$ : The Adjusted R-squared [-];
- $R^2$ : The R-squared [-];
- $n$ : The Number of Observations (Data Points) [-];
- $p$ : The Number of Predictors (Independent Variables) in the Model [-].

Ultimately, the Adjusted R-squared gives a more realistic measure of how well the model explains the data, especially when comparing models with different numbers of predictors.

#### V.4.4. F-statistic (F)

The F-statistic is a measure used in regression analysis to test the overall significance of the model, i.e., whether the model as a whole provides a better fit to the data than a model with no predictors (i.e., A Constant-Only Model).

The F-statistic compares two models:

1. **The Full Model:** Which includes the predictors (Independent Variables);
2. **The Null Model:** (Constant Model), which only includes an intercept (No Predictors).

The F-statistic is calculated, as shown in Equation (V.29):

$$F = \frac{\left( \frac{SS_{Regression}}{p} \right)}{\left( \frac{SS_{Residual}}{n - p - 1} \right)} \quad (\text{V.29})$$

Where:

- $F$ : The F-statistic [-];
- $SS_{Regression}$ : The Sum of Squares (SumSq) for Regression, Representing the Variation Explained by the Model [-];

- **SS<sub>Residual</sub>**: The Sum of Squares (SumSq) for Residuals, Representing the Variation Unexplained by the Model [-];
- **p**: The Number of Predictors (Independent Variables) [-];
- **n**: The Number of Observations [-].

Ultimately, the F-statistic helps assess whether the regression model provides a significantly better fit to the data compared to a model with no predictors (Just the Intercept).

#### V.4.5. p-value for F-statistic

The p-value in regression analysis is used to assess the statistical significance of individual regression coefficients or the overall regression model. It helps determine whether the relationships between the independent variables and the dependent variable are statistically significant. A smaller p-value indicates that the relationship is more likely to be real and not due to random chance.

In the context of the F-statistic and regression analysis, the p-value associated with the F-statistic tests the null hypothesis that none of the predictors (Independent Variables) in the model are significantly related to the dependent variable. A smaller p-value (Typically Less Than 0.05) suggests that the model provides a better fit than a model with no predictors.

For a regression model, the p-value associated with the F-statistic is computed by comparing the calculated F-statistic to an F-distribution with the appropriate degrees of freedom for the numerator and denominator.

The p-value for the F-statistic is calculated, as shown in Equation (V.30):

$$p - \text{value} = P(F > F_{\text{Calculated}} | DF_{\text{Model}}, DF_{\text{Residual}}) \quad (\text{V.30})$$

Where:

- **p – value**: The p-value for the F-statistic [-];
- **F<sub>Calculated</sub>**: The Computed F-statistic from the Model [-];
- **DF<sub>Model</sub>**: The Degrees of Freedom Associated with the Regression Model [-];
- **DF<sub>Residual</sub>**: The Degrees of Freedom Associated with the Residuals [-].

Ultimately, the p-value for the F-statistic is calculated using the F-distribution with the degrees of freedom for the regression model and residuals. The p-value gives the probability of obtaining an F-statistic as extreme as the one calculated, assuming that the null hypothesis is true. If the p-value is less

than the significance level (Usually 0.05), you reject the null hypothesis and conclude that the regression model significantly explains the variation in the dependent variable.

In conclusion, these statistics provide insight into the model's predictive accuracy and whether it generalizes well to new data.

## V.5. Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) is a statistical technique used to compare the means of three or more groups to determine if there are statistically significant differences between them. Analysis of Variance (ANOVA) tests the null hypothesis that all group means are equal against the alternative hypothesis that at least one group mean is different.

The basic idea is to partition the total variance observed in the data into components attributable to different sources, such as the variance between groups (Due to the Treatment or Factor) and the variance within groups (Due to Random Error or Individual Variation). By comparing these variances, Analysis of Variance (ANOVA) determines whether the observed differences between group means are greater than what would be expected by chance.

There are different types of Analysis of Variance (ANOVA):

1. **One-Way Analysis of Variance (ANOVA):** Compares means across multiple groups based on one factor (e.g., Different Treatments);
2. **Two-Way Analysis of Variance (ANOVA):** Compares means across groups based on two factors, and can also examine interactions between the factors;
3. **Multivariate ANOVA (MANOVA):** Extends ANOVA to multiple dependent variables.

Ultimately, the Analysis of Variance (ANOVA) provides an F-statistic, which is the ratio of the variance between the groups to the variance within the groups. A high F-statistic indicates that the group means are significantly different from each other. The associated p-value helps assess the statistical significance of the results.

### V.5.1. Sum of Squares (SumSq)

In regression analysis, the Sum of Squares (SumSq) quantifies the variability in the dependent variable. It is used to measure how much of the variation in the data is explained by different components of the

model. These components include the total variability (Total), the variability explained by the model (Model), the linear terms (Linear), the nonlinear terms (Nonlinear), and the unexplained variability (Residuals).

1. **Sum of Squares for Total ( $SS_{\text{Total}}$ ):** The Sum of Squares (SumSq) for Total ( $SS_{\text{Total}}$ ) quantifies the total variation in the dependent variable ( $y$ ) from its mean. It is calculated as the sum of the squared differences between each observed value and the mean of the observed values, as shown in Equation (V.31):

$$SS_{\text{Total}} = \sum_{i=1}^n (y_i - \bar{y})^2 \quad (\text{V.31})$$

Where:

- **$SS_{\text{Total}}$ :** The Sum of Squares (SumSq) for Total [-];
- **$y_i$ :** The Observed Value of the Dependent Variable for the  $i$ -th Observation [-];
- **$\bar{y}$ :** The Mean of the Observed Values of the Dependent Variable [-];
- **$n$ :** The Mean of the Observed Values of the Dependent Variable [-].

2. **Sum of Squares for Model ( $SS_{\text{Model}}$ ):** The Sum of Squares (SumSq) for Model ( $SS_{\text{Model}}$ ) quantifies the variation explained by the regression model. It measures how much of the total variation in ( $y$ ) is accounted for by the predictors (Independent Variables), as shown in Equation (V.32):

$$SS_{\text{Model}} = \sum_{i=1}^n (\hat{y}_i - \bar{y})^2 \quad (\text{V.32})$$

Where:

- **$SS_{\text{Model}}$ :** The Sum of Squares (SumSq) for Model [-];
- **$\hat{y}_i$ :** The Predicted Value of ( $y$ ) for the  $i$ -th Observation based on the Model [-];
- **$\bar{y}$ :** The Mean of the Observed Values of ( $y$ ) [-];
- **$n$ :** The Mean of the Observed Values of the Dependent Variable [-].

3. **Sum of Squares for Linear Model ( $SS_{\text{Linear}}$ ):** The Sum of Squares (SumSq) for Linear ( $SS_{\text{Linear}}$ ) corresponds to the part of the model that is explained by the linear predictors (i.e., The

Terms in the Model that are Linear in the Predictors). These are the terms like  $x_1, x_2$  etc., but without considering any interaction or nonlinear terms like  $x_1^2, x_2^2$  or  $x_1 \times x_2$ . It is calculated, as shown in Equation (V.33):

$$SS_{\text{Linear}} = \sum_{i=1}^n (\hat{y}_{\text{Linear},i} - \bar{y})^2 \quad (\text{V.33})$$

Where:

- **$SS_{\text{Linear}}$** : The Sum of Squares (SumSq) for Linear Model [-];
- **$\hat{y}_{\text{Linear},i}$** : The Predicted Value of (y) for the i-th Observation based Only on the Linear Predictors (Without Considering Nonlinear or Interaction Terms) [-];
- **$\bar{y}$** : The Mean of the Observed Values of (y) [-];
- **$n$** : The Mean of the Observed Values of the Dependent Variable [-].

**4. Sum of Squares for Nonlinear Model ( $SS_{\text{Nonlinear}}$ )**: The Sum of Squares (SumSq) for Nonlinear ( $SS_{\text{Nonlinear}}$ ) captures the portion of the variation explained by the nonlinear components of the model, such as squared terms  $x_1^2, x_2^2$  or interaction terms  $x_1 \times x_2$ . It is calculated, as shown in Equation (V.34):

$$SS_{\text{Nonlinear}} = \sum_{i=1}^n (\hat{y}_{\text{Nonlinear},i} - \hat{y}_{\text{Linear},i})^2 \quad (\text{V.34})$$

Where:

- **$SS_{\text{Linear}}$** : The Sum of Squares (SumSq) for Nonlinear Model [-];
- **$\hat{y}_{\text{Nonlinear},i}$** : The Predicted Value of (y) for the i-th Observation based on the Entire Model (Including Nonlinear Terms) [-];
- **$\hat{y}_{\text{Linear},i}$** : The Predicted Value of (y) based only on the Linear Terms [-];
- **$n$** : The Mean of the Observed Values of the Dependent Variable [-].

**5. Sum of Squares for Residuals ( $SS_{\text{Residual}}$ )**: The Sum of Squares (SumSq) for Residuals ( $SS_{\text{Residual}}$ ) represents the unexplained variability in the dependent variable after accounting for the model's predictions. It is the difference between the total variability and the variability explained by the model. It is calculated, as shown in Equation (V.35):

$$SS_{\text{Residual}} = \sum_{i=1}^n (y_i - \hat{y}_i)^2 \quad (\text{V.35})$$

Where:

- **$SS_{\text{Residual}}$** : The Sum of Squares (SumSq) for Residual [-];
- **$y_i$** : The Observed Value of the Dependent Variable for the i-th Observation [-];
- **$\hat{y}_i$** : The Predicted Value of (y) for the i-th Observation based on the Model [-];
- **$n$** : The Mean of the Observed Values of the Dependent Variable [-].

Ultimately, the Sum of Squares (SumSq) calculations are essential for understanding the different sources of variation in regression analysis. They help partition the total variation in the dependent variable into components explained by the model (linear and nonlinear terms) and the residual error. These sums of squares are then used to compute important statistics such as the F-statistic and the Mean Squares (MeanSq).

### V.5.2. Number of Observations (n)

The number of observations in a regression analysis refers to the total count of data points used in fitting the regression model. It plays a crucial role in determining the degrees of freedom, calculating residual variance, and assessing model accuracy.

The number of observations refers to the total number of data points or cases in the dataset. Each observation corresponds to a row in your dataset, containing values for both the dependent variable (Also Called the Response Variable) and the independent variables (Predictors).

The number of observations is important because it impacts:

1. **Degrees of Freedom:** It is used to calculate the number of degrees of freedom, which is crucial for estimating variances, Standard Errors (SE), and performing hypothesis tests;
2. **Estimating the Variance:** The number of observations is used to estimate the variance of the residuals (Errors). The variance is critical for computing Standard Errors (SE) and conducting hypothesis tests.

Ultimately, for a model with two independent variables, the number of observations would simply be the total number of rows in the dataset.

### V.5.3. Degrees of Freedom (DF)

The Degrees of Freedom (DF) for different components in regression analysis are important for partitioning the total variability in the data. These components include the total variability (Degrees of Freedom (DF) for Total), the variability explained by the model (Degrees of Freedom (DF) for Model), and the residual variability (Degrees of Freedom (DF) for Residual). These degrees of freedom are crucial for various statistical tests, such as calculating the F-statistic and the Mean Square.

1. **The Degrees of Freedom (DF) for Total:** The DF for Total represents the total number of observations minus one. This accounts for all the observations in the dataset and provides a measure of the total variation in the dependent variable before any model is fitted. It is calculated, as shown in Equation (V.36):

$$DF_{\text{Total}} = n - 1 \quad (\text{V.36})$$

Where:

- **DF<sub>Total</sub>:** The Degrees of Freedom Total [-];
- **n:** The Number of Observations (Data Points) [-].

2. **The Degrees of Freedom (DF) for Model:** The DF for Model refers to the number of predictors (Independent Variables) included in the regression model. This represents the number of independent variables or parameters estimated in the model. It accounts for the number of components in the regression model that explain the variation in the dependent variable. It is calculated, as shown in Equation (V.37):

$$DF_{\text{Model}} = p \quad (\text{V.37})$$

Where:

- **DF<sub>Model</sub>:** The Degrees of Freedom Model [-];
- **p:** The Number of Estimated Parameters in the Model (Including the Intercept) [-].

3. **The Degrees of Freedom (DF) for Linear Model:** The DF for Linear is the DF for the Model associated with the linear part of the regression model, which includes only the linear predictors (Excluding Interactions and Nonlinear Terms). This represents how much variability is explained by the linear terms in the model. It is calculated, as shown in Equation (V.38):

$$DF_{\text{Linear}} = p_{\text{Linear}} \quad (\text{V.38})$$

Where:

- **DF<sub>Linear</sub>**: The Degrees of Freedom Linear [-];
- **p<sub>Linear</sub>**: The Number of Linear Predictors (Independent Variables) in the Model [-].

**4. The Degrees of Freedom (DF) for Nonlinear Model:** The DF for Nonlinear corresponds to the degrees of freedom associated with the nonlinear components of the regression model, such as interaction terms or squared terms (e.g.,  $x_1^2$ ,  $x_2^2$  or  $x_1$ ,  $x_2$ ). This represents the portion of the model that captures nonlinear relationships between the predictors and the dependent variable. It is calculated, as shown in Equation (V.39):

$$DF_{\text{Nonlinear}} = p_{\text{Nonlinear}} \quad (\text{V.39})$$

Where:

- **DF<sub>Nonlinear</sub>**: The Degrees of Freedom Nonlinear [-];
- **p<sub>Nonlinear</sub>**: The Number of Nonlinear Components in the Model (e.g., Squared Terms or Interactions) [-].

**5. The Degrees of Freedom (DF) for Residuals:** The DF for Residuals refers to the degrees of freedom for the error or residuals, which represents the variability in the dependent variable that is not explained by the model. It is calculated as the total number of observations minus the number of estimated parameters (Including the Intercept), as shown in Equation (V.40):

$$DF_{\text{Residual}} = n - p - 1 \quad (\text{V.40})$$

Where:

- **DF<sub>Residuals</sub>**: The Degrees of Freedom Residual [-];
- **n**: The Number of Observations (Data Points) [-];
- **p**: The Number of Estimated Parameters in the Model (Including the Intercept) [-].

Ultimately, the Degrees of Freedom (DF) are crucial for understanding the structure of a regression model. The Degrees of Freedom (DF)<sub>Total</sub> accounts for all data points, DF<sub>Model</sub> captures the Degrees of Freedom (DF) associated with the predictors, DF<sub>Linear</sub> and DF<sub>Nonlinear</sub> split the model's components into linear and nonlinear parts, and DF<sub>Residual</sub> accounts for the unexplained variability. These calculations are



essential for statistical tests like the F-statistic, which helps evaluate the overall significance of the regression model.

#### V.5.4. Error Degrees of Freedom ( $df_{\text{Error}}$ )

The Error Degrees of Freedom ( $df_{\text{Erreur}}$ ) refers to the amount of information available for estimating the residual variance or error variance in a regression model. It is crucial for determining the Standard Errors (SE) of the estimated coefficients and performing hypothesis tests.

In regression analysis, degrees of freedom refers to the number of independent pieces of information used to estimate parameters or variability. It can be broken down into two types:

1. **Degrees of Freedom for the Model:** This is the number of independent variables (Predictors) in the model, excluding the intercept;
2. **Degrees of Freedom for the Error:** This is the number of observations minus the number of estimated parameters in the model.

The Error Degrees of Freedom is calculated by subtracting the number of parameters (Including the Intercept) from the number of observations, as shown in Equation (V.41):

$$df_{\text{Error}} = n - p \quad (\text{V.41})$$

Where:

- **$df_{\text{Error}}$ :** The Error Degrees of Freedom [-];
- **$n$ :** The Number of Observations (Data Points) [-];
- **$p$ :** The Number of Estimated Parameters in the Model (Including the Intercept) [-].

Ultimately, the Error Degrees of Freedom represents the amount of data available to estimate the error variance in the regression model. It is calculated by subtracting the number of estimated parameters from the number of observations. This value is critical for calculating Standard Errors (SE), conducting hypothesis tests, and assessing the precision of the model's estimates.

#### V.5.5. The Mean Squares (MeanSq)

The Mean Squares (MeanSq) are the average values of the Sum of Squares (SumSq) for each component in a regression analysis. These are used to quantify the average variability explained by the different components (Total, Model, Linear, Nonlinear) and the residual variability.

- 1. Mean Square for Total ( $MS_{Total}$ ):** The Mean Square for Total ( $MS_{Total}$ ) quantifies the total variability in the dependent variable, averaged over the total number of observations. It is essentially the Sum of Squares (SumSq) for Total ( $SS_{Total}$ ) divided by its corresponding degrees of freedom, as shown in Equations (V.42, and 43):

$$MS_{Total} = \frac{SS_{Total}}{DF_{Total}} \quad (V.42)$$

$$DF_{Total} = n - 1 \quad (V.43)$$

Where:

- **$MS_{Total}$ :** The Mean Square for Total [-];
- **$SS_{Total}$ :** The Sum of Squares (SumSq) for Total [-];
- **$DF_{Total}$ :** The Degrees of Freedom for Total [-];
- **$n$ :** The Number of Observations (Data Points) [-].

- 2. Mean Square for Model ( $MS_{Model}$ ):** The Mean Square for Model ( $MS_{Model}$ ) quantifies how much variability is explained by the regression model, averaged over the degrees of freedom associated with the model. It is the Sum of Squares (SumSq) for the Model ( $SS_{Model}$ ) divided by its corresponding degrees of freedom, as shown in Equations (V.44, and 45):

$$MS_{Model} = \frac{SS_{Model}}{DF_{Model}} \quad (V.44)$$

$$DF_{Model} = p \quad (V.45)$$

Where:

- **$MS_{Model}$ :** The Mean Square for Model [-];
- **$SS_{Model}$ :** The Sum of Squares (SumSq) for Model [-];
- **$DF_{Model}$ :** The Degrees of Freedom for Model [-];
- **$p$ :** The Number of Estimated Parameters in the Model (Including the Intercept) [-].

- 3. Mean Square for Linear Model ( $MS_{Linear}$ ):** The Mean Square for Linear ( $MS_{Linear}$ ) quantifies how much variability is explained by the linear components of the regression model, averaged over the degrees of freedom associated with the linear terms, as shown in Equations (V.46, and 47):

$$MS_{\text{Linear}} = \frac{SS_{\text{Linear}}}{DF_{\text{Linear}}} \quad (\text{V.46})$$

$$DF_{\text{Linear}} = p_{\text{Linear}} \quad (\text{V.47})$$

Where:

- **MS<sub>Linear</sub>**: The Mean Square for Linear [-];
- **SS<sub>Linear</sub>**: The Sum of Squares (SumSq) for Linear [-];
- **DF<sub>Linear</sub>**: The Degrees of Freedom for Linear [-];
- **p<sub>Linear</sub>**: The Number of Linear Predictors (Independent Variables) in the Model [-].

**4. Mean Square for Nonlinear Model (MS<sub>Nonlinear</sub>):** The Mean Square for Nonlinear (MS<sub>Nonlinear</sub>) quantifies how much variability is explained by the nonlinear components (Squared Terms, Interaction Terms, etc.) of the regression model, averaged over the Degrees of Freedom (DF) associated with the nonlinear terms, as shown in Equations (V.48, and 49):

$$MS_{\text{Nonlinear}} = \frac{SS_{\text{Nonlinear}}}{DF_{\text{Nonlinear}}} \quad (\text{V.48})$$

$$DF_{\text{Nonlinear}} = p_{\text{Nonlinear}} \quad (\text{V.49})$$

Where:

- **MS<sub>Nonlinear</sub>**: The Mean Square for Nonlinear [-];
- **SS<sub>Nonlinear</sub>**: The Sum of Squares (SumSq) for Nonlinear [-];
- **DF<sub>Nonlinear</sub>**: The Degrees of Freedom for Nonlinear [-];
- **p<sub>Nonlinear</sub>**: The Number of Nonlinear Components in the Model (e.g., Squared Terms or Interactions) [-].

**5. Mean Square for Residuals (MS<sub>Residual</sub>):** The Mean Square for Residuals (MS<sub>Residual</sub>) quantifies the average residual error or unexplained variability after accounting for the model's predictions. It is the Sum of Squares (SumSq) for Residuals (SS<sub>Residual</sub>) divided by its corresponding degrees of freedom, as shown in Equations (V.50, and 51):

$$MS_{\text{Residual}} = \frac{SS_{\text{Residual}}}{DF_{\text{Residual}}} \quad (\text{V.50})$$

$$DF_{\text{Residual}} = n - p - 1 \quad (\text{V.51})$$

Where:

- **MS<sub>Residual</sub>**: The Mean Square for Residual [-];
- **SS<sub>Residual</sub>**: The Sum of Squares (SumSq) for Residual [-];
- **DF<sub>Residual</sub>**: The Degrees of Freedom for Residual [-];
- **n**: The Number of Observations (Data Points) [-];
- **p**: The Number of Estimated Parameters in the Model (Including the Intercept) [-].

Ultimately, Mean Square for Residuals (MS<sub>Residual</sub>) are key to calculating the F-statistic in Analysis of Variance (ANOVA), which helps assess the overall significance of the regression model.

### V.5.6. The F-statistic (F)

The F-statistic (F) is a ratio used to test the overall significance of a regression model. It compares the variability explained by the model to the unexplained variability (Residuals). In regression analysis, F-statistic (F) is computed for various components such as Total, Model, Linear, Nonlinear, and Residual to assess how well the model fits the data.

1. **F for Total (F<sub>Total</sub>)**: The F for Total is not typically computed directly, as the F-statistic is more commonly used to assess the significance of the model. However, we can still define it conceptually. The Total Sum of Squares (SS<sub>Total</sub>) represents the total variability in the dependent variable, and the F<sub>Total</sub> is the ratio of explained variance to the unexplained variance.

Since SS<sub>Total</sub> is essentially the total variance in the dependent variable, F<sub>Total</sub> can be conceptually considered as the comparison between the SS<sub>Total</sub> and the residual variance. Still, it's not computed in the same way as the model F-statistic. It is calculated, as shown in Equation (V.52):

$$F_{\text{Total}} = \frac{SS_{\text{Model}}}{SS_{\text{Residual}}} = 1 \quad (\text{V.52})$$

Where:

- **F<sub>Total</sub>**: The F for Total [-];
- **SS<sub>Model</sub>**: The Sum of Squares (SumSq) for Model [-];
- **SS<sub>Residual</sub>**: The Sum of Squares (SumSq) for Residual [-].

**2. F for Model ( $F_{\text{Model}}$ ):** The F-statistic for Model ( $F_{\text{Model}}$ ) assesses the overall significance of the regression model. It compares the Mean Square for the Model ( $MS_{\text{Model}}$ ) to the Mean Square for Residuals ( $MS_{\text{Residual}}$ ) to see if the model explains a significant portion of the variation in the dependent variable, as shown in Equation (V.53):

$$F_{\text{Model}} = \frac{MS_{\text{Model}}}{MS_{\text{Residual}}} \quad (\text{V.53})$$

Where:

- **$F_{\text{Model}}$** : The F for Model [-];
- **$MS_{\text{Model}}$** : The Mean Square for the Model [-];
- **$MS_{\text{Residual}}$** : The Mean Square for the Residual [-].

**3. F for Linear ( $F_{\text{Linear}}$ ):** The F-statistic for Linear ( $F_{\text{Linear}}$ ) assesses how much of the variation is explained by the linear terms in the model. It is calculated by comparing the Mean Square for Linear to the Mean Square for Residuals, as shown in Equation (V.54):

$$F_{\text{Linear}} = \frac{MS_{\text{Linear}}}{MS_{\text{Residual}}} \quad (\text{V.54})$$

Where:

- **$F_{\text{Linear}}$** : The F for Linear [-];
- **$MS_{\text{Linear}}$** : The Mean Square for the Linear Terms [-];
- **$MS_{\text{Residual}}$** : The Mean Square for the Residuals [-].

**4. F for Nonlinear ( $F_{\text{Nonlinear}}$ ):** The F-statistic for Nonlinear ( $F_{\text{Nonlinear}}$ ) assesses the significance of the nonlinear terms in the regression model, such as squared terms or interaction terms. It is calculated by comparing the Mean Square for Nonlinear to the Mean Square for Residuals, as shown in Equation (V.55):

$$F_{\text{Nonlinear}} = \frac{MS_{\text{Nonlinear}}}{MS_{\text{Residual}}} \quad (\text{V.55})$$

Where:

- **$F_{\text{Nonlinear}}$** : The F for Nonlinear [-];
- **$MS_{\text{Nonlinear}}$** : The Mean Square for the Nonlinear Terms [-];

- **MS<sub>Residual</sub>**: The Mean Square for the Residuals [-].

**5. F for Residual (F<sub>Residual</sub>):** The F-statistic for Residual (F<sub>Residual</sub>) is not commonly calculated explicitly in regression analysis. The residual Mean Square (MS<sub>Residual</sub>) is used in the calculations of the F-statistics for the model, linear, and nonlinear components. The residual represents the unexplained variability after fitting the model. However, conceptually, the F-statistic for Residual can be considered as the ratio of the Mean Square for Residuals to itself. It is calculated, as shown in Equation (V.56):

$$F_{\text{Residual}} = \frac{MS_{\text{Residual}}}{MS_{\text{Residual}}} = 1 \quad (\text{V.56})$$

Where:

- **F<sub>Residual</sub>**: The F for Residual [-];
- **MS<sub>Residual</sub>**: The Mean Square for the Residuals [-].

Ultimately, in practice, F-statistics are used to compare the contribution of different parts of the model (Linear vs. Nonlinear) and assess if the model as a whole is statistically significant.

### V.5.7. The p-value (p-value)

The p-value is used in regression analysis to test the significance of different components of the model, such as the Total, Model, Linear, Nonlinear, and Residual components. A smaller p-value indicates stronger evidence against the null hypothesis (Which Typically Asserts that there is no Relationship Between the Variables).

**1. p-Value for Model (p-Value<sub>Model</sub>):** The p-value for the Model tests the overall significance of the regression model, which includes both linear and nonlinear terms. It assesses whether at least one predictor variable has a significant relationship with the dependent variable. The p-value for the Model is calculated based on the F-statistic for the model. The F-statistic follows an F-distribution with degrees of freedom based on the number of predictors in the model and the residual degrees of freedom. It is calculated, as shown in Equation (V.57):

$$p - \text{Value}_{\text{Model}} = P(F_{\text{Calculated}} > F_{\text{Critical}}) \quad (\text{V.57})$$

Where:

- **p – Value<sub>Model</sub>**: The p-value for the Model [-];
- **F<sub>Calculated</sub>**: The Computed F-statistic for the Mode [-];
- **F<sub>Critical</sub>**: The Critical Value of the F-distribution based on the Degrees of Freedom [-].

**2. p-Value for Linear (p<sub>Linear</sub>):** The p-value for the Linear terms tests whether the linear components (e.g.,  $x_1, x_2$ ) of the model have a significant relationship with the dependent variable. The p-value for Linear is typically calculated for each predictor. The p-value for each Linear term is calculated based on its t-statistic and follows a t-distribution, as shown in Equation (V.58):

$$p - \text{Value}_{\text{Linear}} = P(t_{\text{Calculated}} > t_{\text{Critical}}) \quad (\text{V.58})$$

Where:

- **p – Value<sub>Linear</sub>**: The p-value for the Linear [-];
- **t<sub>Calculated</sub>**: The t-statistic for the Individual Predictor [-];
- **t<sub>Critical</sub>**: The Critical Value of the t-distribution based on the Degrees of Freedom for the Residuals [-].

**3. p-Value for Nonlinear (p<sub>Nonlinear</sub>):** The p-value for Nonlinear terms assesses the significance of the nonlinear components (e.g.,  $x_1^2, x_2^2$  or interaction terms like  $x_1 \times x_2$ ) in the regression model. It helps determine if these terms provide additional explanatory power for the dependent variable, beyond the linear components. The p-value for the Nonlinear terms is calculated based on the F-statistic for the Nonlinear terms. It follows a similar approach to the p-value for the overall model, comparing the F-statistic for the nonlinear terms to the critical value from the F-distribution, as shown in Equation (V.59):

$$p - \text{Value}_{\text{Nonlinear}} = P(F_{\text{Calculated}} > F_{\text{Critical}}) \quad (\text{V.59})$$

Where:

- **p – Value<sub>Model</sub>**: The p-value for the Nonlinear [-];
- **F<sub>Calculated</sub>**: The Computed F-statistic for the Nonlinear Terms [-];
- **F<sub>Critical</sub>**: The Critical Value of the F-distribution based on the Degrees of Freedom for the Nonlinear Terms and Residuals [-].

Ultimately, p-values are used to test the significance of the coefficients or components in the regression model.

In conclusion, the Analysis of Variance (ANOVA) is widely used in experimental design, hypothesis testing, and to evaluate the effectiveness of treatments or interventions across multiple groups.

## V.6. Multi-Objective Optimization (MOO)

Multi-Objective Optimization (MOO) refers to the process of optimizing two or more conflicting objectives simultaneously (Date Palm Fiber (DPF) Size ( $x_1$ ) and Fiber-to-Matrix Mass Ratio ( $x_2$ )), where the goal is to find solutions that strike a balance between the competing objectives. In contrast to single-objective optimization, where only one goal is optimized, Multi-Objective Optimization (MOO) deals with situations where multiple criteria need to be considered and optimized at the same time. This approach is particularly useful in fields in as bio-composite material design, where various factors (e.g., Charpy Impact Strength (G), Water Absorption (WAR), and Thermal Conductivity ( $\lambda$ )) need to be optimized concurrently. The challenge in Multi-Objective Optimization (MOO) lies in identifying solutions that provide the best trade-offs between the different objectives, as shown in Equations (V.60, 61, 62, and 63).

$$\text{Min } (-G), \text{WAR}, \lambda \quad (\text{V.60})$$

Subject to:

$$0.080 \leq x_1 \leq 0.500 \quad (\text{V.61})$$

$$05 \leq x_2 \leq 15 \quad (\text{V.62})$$

Where:

- $\lambda$ : The Thermal Conductivity [w/m.k];
- **WAR**: The Water Absorption [%];
- **G**: The Charpy Impact Strength [KJ/m<sup>2</sup>];
- $x_1$ : The Date Palm Fiber (DPF) Size (Decision Variable) [mm];
- $x_2$ : The Fiber Mass Ratio (Decision Variable) [%].



Ultimately, a constraint was added for the gypsum compressive strength, as shown in Equation (V.63), ensuring that the optimized bio-composite material had adequate mechanical strength.

$$f_m \geq \bar{f}_m = 6.998 \text{ MPa; Gypsum Compressive Strength} \quad (\text{V.63})$$

Where:

- $f_m$ : The Gypsum Compressive Strength (Constraint) [MPa];
- $\bar{f}_m$ : The Average Compressive Strength of Gypsum [MPa].

### V.6.1. Objectives

In the context of Multi-Objective Optimization (MOO), objectives refer to the specific goals or criteria that are to be optimized. Each objective represents a particular property or characteristic of the system under consideration. In the Multi-Objective Optimization (MOO), the three primary objectives are:

- 1. Maximizing Impact Strength (G):** Charpy Impact strength (G) measures the bio-composite material's resistance to sudden forces. Higher impact strength is desirable for durability and mechanical performance in applications subjected to external stresses;
- 2. Minimizing Water Absorption (WAR):** Water absorption (WAR) is a critical property for bio-composite materials exposed to moisture. A lower water absorption (WAR) percentage is preferred to prevent material degradation and maintain its structural integrity;
- 3. Minimizing Thermal Conductivity ( $\lambda$ ):** Thermal conductivity ( $\lambda$ ) reflects the bio-composite material's ability to conduct heat, with lower values typically being desired in applications requiring insulation.

Ultimately, each objective requires specific decision variables (Date Palm Fiber (DPF) Size ( $x_1$ ) and Fiber-to-Matrix Mass Ratio ( $x_2$ )) to be adjusted in order to achieve the desired outcome.

### V.6.2. Pareto Optimality

Pareto optimality refers to a concept in Multi-Objective Optimization (MOO) where no objective can be improved without deteriorating another. A solution is said to be Pareto-optimal if, given the trade-offs between the objectives, there is no other solution that dominates it. In other words, a Pareto-optimal

solution is one where all objectives are balanced in such a way that improving one would lead to the deterioration of at least one other. This concept is fundamental in Multi-Objective Optimization (MOO) because it provides a set of solutions that are considered efficient, meaning that no further improvements can be made in one objective without causing harm to the others.

A solution is Pareto-optimal if there is no other solution that improves one objective without worsening another. Mathematically, for two solutions  $X = (x_1, x_2)$  and  $Y = (y_1, y_2)$ , solution  $x$  is Pareto-optimal if, as shown in Equation (V.64):

$$\forall i, f_i(X) \leq f_i(Y) \text{ and } \exists j, f_j(X) < f_j(Y) \quad (\text{V.64})$$

Where:

- $f_i(X)$   $f_i(Y)$ : The Values of the Objectives for Solutions  $X$  and  $Y$ , Respectively [-].

### V.6.3. Pareto Front

The Pareto front is the graphical representation of the set of Pareto-optimal solutions. In Multi-Objective Optimization (MOO), the Pareto front shows the trade-offs between different objectives. It is essentially a curve or surface where each point represents a different solution, and no point on the Pareto front is dominated by any other. A solution that lies on the Pareto front is considered to be efficient because it represents the best possible trade-off between the competing objectives. In the case of bio-composite materials, the Pareto front might show a balance between minimizing thermal conductivity ( $\lambda$ ) and water absorption (WAR) while maximizing impact strength (G). The goal is to identify solutions along this front that offer the most desirable combination of properties for the intended application.

The Pareto front is the set of Pareto-optimal solutions, represented graphically, as shown in Equation (V.65):

$$\{X^* | \forall Y, f(X^*) \leq f(Y) \text{ For at Least One Objective}\} \quad (\text{V.65})$$

Where:

- **$f(\mathbf{X}^*)$** : The Vector of Objectives for a Pareto-Optimal Solution  $\mathbf{X}^*$ , and the Goal is to Find a Set of such Solutions Where no Further Improvement Can be Made in One Objective Without Worsening Another [-].

#### V.6.4. Trade-off

A trade-off refers to the compromise made between two or more conflicting objectives when optimizing them simultaneously. In Multi-Objective Optimization (MOO), trade-offs are inevitable because improving one objective often leads to the deterioration of another. For example, in the case of bio-composite materials, minimizing thermal conductivity ( $\lambda$ ) may increase water absorption (WAR), and maximizing charpy impact strength (G) may increase thermal conductivity. These trade-offs must be carefully considered when selecting the best solution. Understanding the trade-offs is crucial for decision-makers to choose a solution that aligns with their priorities or specific application needs. The Pareto front helps in visualizing and understanding these trade-offs, allowing for a better-informed decision regarding which compromise to accept.

A trade-off occurs when improving one objective results in the deterioration of another. This can be expressed, as shown in Equation (V.66):

$$\Delta f_1(\mathbf{X}) > 0 \text{ implies } \Delta f_2(\mathbf{X}) < 0 \quad (\text{V.66})$$

Where:

- $f_1(\mathbf{X})$  and  $f_2(\mathbf{X})$ : Two Conflicting Objectives [-];
- $\Delta f_1(\mathbf{X})$ : The Change in Objective 1 [-];
- $\Delta f_2(\mathbf{X})$ : The Change in Objective 2 [-].

Ultimately, Multi-Objective Optimization (MOO) seeks to find a balanced set of solutions that best satisfy all objectives, acknowledging that optimizing for one may come at the expense of another.

## V.7. MATLAB Code

This MATLAB code performs a regression analysis to model the relationship between the size and the mass ratio of Date Palm Fiber (DPF) (Reinforcement in Bio-composite Material) and the measured Charpy impact strength (G). The code evaluates three different regression models: a linear model, a quadratic model, and a pure quadratic model. The models are then used to predict the Charpy Impact Strength (G) values, and the prediction accuracy is assessed using the Root Mean Square Error (RMSE).

### V.7.1. Measured Data

The independent variables  $x_1$  (Size of Date Palm Fiber (DPF)) and  $x_2$  (Mass Ratio of Date Palm Fiber) are given as vectors:

```
% Measured Data
x1 = [0.08, 0.08, 0.08, 0.315, 0.315, 0.315, 0.5, 0.5, 0.5]; % Size
x2 = [5, 10, 15, 5, 10, 15, 5, 10, 15]; % Mass Ratio
measured = [2.460, 1.840, 0.983, 3.167, 5.243, 8.005, 2.067, 3.597, 4.519]; % Charpy Impact Strength
```

These variables are the primary data points used in the analysis.

### V.7.2. Design Matrices for Regression Models

- 1. Linear Model:** The linear model includes the intercept (Constant Term),  $x_1$ , and  $x_2$  as the independent variables:

```
X_linear = [ones(length(x1), 1), x1', x2']; % Linear Model (with intercept)
```

The `ones(length(x1), 1)` column represents the intercept term ( $\beta_0$ ), and the subsequent columns represent  $x_1$  and  $x_2$ .

- 2. Quadratic Model:** The quadratic model includes interaction terms (Multiplication of  $x_1$  and  $x_2$ ) and squared terms ( $x_1^2$  and  $x_2^2$ ):

```
X_quadratic = [ones(length(x1), 1), x1', x2', x1'.*x2', x1'.^2, x2'.^2]; % Quadratic Model (with interaction and squared terms)
```

The interaction term ( $x_1 * x_2$ ) accounts for potential synergistic effects between the two variables, while the squared terms account for non-linear relationships.

- 3. Pure Quadratic Model:** The pure quadratic model includes the intercept,  $x_1$ ,  $x_2$ , and the squared terms ( $x_1^2$  and  $x_2^2$ ) but omits the interaction term:

```
X_pure_quadratic = [ones(length(x1), 1), x1', x2', x1'.^2, x2'.^2]; % Pure Quadratic Model
(with squared terms only)
```

This model evaluates the effect of each independent variable individually with respect to its quadratic term.

### V.7.3. Regression Analysis Using Regress

The regress function is used to perform the regression analysis for each model:

#### 1. For the Linear Model:

```
[b_linear, bint_linear, r_linear, rint_linear, stats_linear] = regress(measured', X_linear);
```

This function estimates the regression coefficients (**b\_linear**) and provides statistics, including the **R-squared** value, which measures the goodness of fit.

#### 2. For the Quadratic and Pure Quadratic Models:

```
[b_quadratic, bint_quadratic, r_quadratic, rint_quadratic, stats_quadratic] =
regress(measured', X_quadratic);
[b_pure_quadratic, bint_pure_quadratic, r_pure_quadratic, rint_pure_quadratic,
stats_pure_quadratic] = regress(measured', X_pure_quadratic);
```

### V.7.4. Model Evaluation

#### 1. The **R-squared** Value is Printed for Each Model:

```
fprintf('R-squared for Linear Model: %.4f\n', stats_linear(1)); % R-squared value
fprintf('R-squared for Quadratic Model: %.4f\n', stats_quadratic(1)); % R-squared value
fprintf('R-squared for Pure Quadratic Model: %.4f\n', stats_pure_quadratic(1)); % R-squared value
```

The **R-squared** value indicates how well the model explains the variability of the data, with values closer to 1 representing better fits.

### V.7.5. Predictions

The models are used to predict Charpy impact strength (G) values based on the fitted regression coefficients:

```
pred_linear = X_linear * b_linear;
pred_quadratic = X_quadratic * b_quadratic;
pred_pure_quadratic = X_pure_quadratic * b_pure_quadratic;
```

These predictions represent the expected Charpy impact strength (G) values according to each model.

### V.7.6. Root Mean Square Error (RMSE)

To assess the accuracy of the models, the Root Mean Squared Error (RMSE) is computed. Root Mean Squared Error (RMSE) quantifies the average prediction error:

```
RMSE_linear = sqrt(mean((measured' - pred_linear).^2));
RMSE_quadratic = sqrt(mean((measured' - pred_quadratic).^2));
RMSE_pure_quadratic = sqrt(mean((measured' - pred_pure_quadratic).^2));
```

The lower the RMSE value, the better the model's predictions match the actual measured values.

### V.7.7. Final Output

The coefficients for each model (Linear, Quadratic, and Pure Quadratic) and their corresponding R-squared and Root Mean Squared Error (RMSE) values are displayed:

```
fprintf('\nRMSE for Linear Model: %.4f\n', RMSE_linear);
fprintf('RMSE for Quadratic Model: %.4f\n', RMSE_quadratic);
fprintf('RMSE for Pure Quadratic Model: %.4f\n', RMSE_pure_quadratic);
```

### V.7.8. Finale Code

```
% Measured Data
x1 = [0.08, 0.08, 0.08, 0.315, 0.315, 0.315, 0.5, 0.5, 0.5]; % Size
x2 = [5, 10, 15, 5, 10, 15, 5, 10, 15]; % Mass Ratio
measured = [2.460, 1.840, 0.983, 3.167, 5.243, 8.005, 2.067, 3.597, 4.519]; % Charpy Impact Strength

% Matrix for independent variables
X_linear = [ones(length(x1), 1), x1', x2']; % Linear Model (with intercept)
X_quadratic = [ones(length(x1), 1), x1', x2', x1'.*x2', x1'.^2, x2'.^2]; % Quadratic Model (with interaction and squared terms)
X_pure_quadratic = [ones(length(x1), 1), x1', x2', x1'.^2, x2'.^2]; % Pure Quadratic Model (with squared terms only)

% Linear Model
[b_linear, bint_linear, r_linear, rint_linear, stats_linear] = regress(measured', X_linear);
fprintf('Linear Model Coefficients (β):\n');
disp(b_linear);
fprintf('Linear Model Standard Error (SE):\n');
disp(bint_linear(:, 2)); % Standard error
fprintf('Linear Model t-Statistic (tStat):\n');
disp(b_linear ./ bint_linear(:, 2)); % t-statistics
fprintf('Linear Model p-Value:\n');
disp(2 * (1 - tcdf(abs(b_linear ./ bint_linear(:, 2)), length(measured) - length(b_linear)))); % p-value

fprintf('R-squared for Linear Model: %.4f\n', stats_linear(1)); % R-squared value
fprintf('Adjusted R-squared for Linear Model: %.4f\n', 1 - (1 - stats_linear(1)) * (length(measured) - 1) / (length(measured) - length(b_linear) - 1)); % Adjusted R-squared
fprintf('F-statistic for Linear Model: %.4f\n', stats_linear(2)); % F-statistic
fprintf('p-value for F-statistic for Linear Model: %.4f\n', stats_linear(3)); % p-value for F-statistic
```

```

% Quadratic Model
[b_quadratic, bint_quadratic, r_quadratic, rint_quadratic, stats_quadratic] = regress(measured', X_quadratic);
fprintf('\nQuadratic Model Coefficients ( $\beta$ ):\n');
disp(b_quadratic);
fprintf('Quadratic Model Standard Error (SE):\n');
disp(bint_quadratic(:, 2)); % Standard error
fprintf('Quadratic Model t-Statistic (tStat):\n');
disp(b_quadratic ./ bint_quadratic(:, 2)); % t-statistics
fprintf('Quadratic Model p-Value:\n');
disp(2 * (1 - tcdf(abs(b_quadratic ./ bint_quadratic(:, 2)), length(measured) - length(b_quadratic)))); % p-value

fprintf('R-squared for Quadratic Model: %.4f\n', stats_quadratic(1)); % R-squared value
fprintf('Adjusted R-squared for Quadratic Model: %.4f\n', 1 - (1 - stats_quadratic(1)) * (length(measured) - 1) / (length(measured) - length(b_quadratic) - 1)); % Adjusted R-squared
fprintf('F-statistic for Quadratic Model: %.4f\n', stats_quadratic(2)); % F-statistic
fprintf('p-value for F-statistic for Quadratic Model: %.4f\n', stats_quadratic(3)); % p-value for F-statistic

% Pure Quadratic Model
[b_pure_quadratic, bint_pure_quadratic, r_pure_quadratic, rint_pure_quadratic, stats_pure_quadratic] =
regress(measured', X_pure_quadratic);
fprintf('\nPure Quadratic Model Coefficients ( $\beta$ ):\n');
disp(b_pure_quadratic);
fprintf('Pure Quadratic Model Standard Error (SE):\n');
disp(bint_pure_quadratic(:, 2)); % Standard error
fprintf('Pure Quadratic Model t-Statistic (tStat):\n');
disp(b_pure_quadratic ./ bint_pure_quadratic(:, 2)); % t-statistics
fprintf('Pure Quadratic Model p-Value:\n');
disp(2 * (1 - tcdf(abs(b_pure_quadratic ./ bint_pure_quadratic(:, 2)), length(measured) - length(b_pure_quadratic)))); % p-value

fprintf('R-squared for Pure Quadratic Model: %.4f\n', stats_pure_quadratic(1)); % R-squared value
fprintf('Adjusted R-squared for Pure Quadratic Model: %.4f\n', 1 - (1 - stats_pure_quadratic(1)) * (length(measured) - 1) / (length(measured) - length(b_pure_quadratic) - 1)); % Adjusted R-squared
fprintf('F-statistic for Pure Quadratic Model: %.4f\n', stats_pure_quadratic(2)); % F-statistic
fprintf('p-value for F-statistic for Pure Quadratic Model: %.4f\n', stats_pure_quadratic(3)); % p-value for F-statistic

% Predictions (using the models to predict values for the given x1, x2)
pred_linear = X_linear * b_linear;
pred_quadratic = X_quadratic * b_quadratic;
pred_pure_quadratic = X_pure_quadratic * b_pure_quadratic;

% Display predicted values
fprintf('\nPredictions for Linear Model:\n');
disp(pred_linear);

fprintf('\nPredictions for Quadratic Model:\n');
disp(pred_quadratic);

fprintf('\nPredictions for Pure Quadratic Model:\n');
disp(pred_pure_quadratic);

% Calculate RMSE for each model
RMSE_linear = sqrt(mean((measured' - pred_linear).^2));
RMSE_quadratic = sqrt(mean((measured' - pred_quadratic).^2));
RMSE_pure_quadratic = sqrt(mean((measured' - pred_pure_quadratic).^2));

fprintf('\nRMSE for Linear Model: %.4f\n', RMSE_linear);

```

```

fprintf('RMSE for Quadratic Model: %.4f\n', RMSE_quadratic);
fprintf('RMSE for Pure Quadratic Model: %.4f\n', RMSE_pure_quadratic);

% ANOVA
% For Linear Model (ANOVA on model residuals)
[~, tbl_linear, ~] = anova1(r_linear, x1);
fprintf('\nANOVA for Linear Model:\n');
disp(tbl_linear);

% For Quadratic Model (ANOVA on model residuals)
[~, tbl_quadratic, ~] = anova1(r_quadratic, x1);
fprintf('\nANOVA for Quadratic Model:\n');
disp(tbl_quadratic);

% For Pure Quadratic Model (ANOVA on model residuals)
[~, tbl_pure_quadratic, ~] = anova1(r_pure_quadratic, x1);
fprintf('\nANOVA for Pure Quadratic Model:\n');
disp(tbl_pure_quadratic);

```

Ultimately, this MATLAB script provides a comprehensive way to analyze the influence of different factors on the Charpy impact strength (G) of bio-composite materials, offering valuable insights for optimizing material properties.

## Conclusion

In conclusion, this fifth chapter has provided a detailed and systematic approach to the methodology used in the regression analysis and statistical evaluation of the collected data. The first step, data collection, ensured that accurate and relevant data were gathered, forming the foundation for subsequent analyses. Various regression models were explored, including linear, quadratic, and pure-quadratic models, to assess the relationships between the variables.

Through statistical analysis, including the estimation of coefficients and the evaluation of their significance using t-statistics and p-values, the robustness of the models was verified. Additionally, Standard Error (SE) and model fit statistics, such as Root Mean Squared Error (RMSE),  $R^2$ , and adjusted  $R^2$ , were utilized to measure the accuracy and explanatory power of the models. The calculation of the F-statistic and p-value for F further validated the reliability of the regression models. The Analysis of Variance (ANOVA) provided an in-depth understanding of how the data's variability was explained by the models, with the results of Sum of Squares (SumSq), Degrees of Freedom, and Mean Squares (MeanSq) enhancing the statistical foundation of the analysis.

The section on Multi-Objective Optimization (MOO) offered valuable insights into how different objectives can be optimized simultaneously. By analyzing the Pareto optimality and Pareto front, this part of the fifth chapter highlighted the trade-offs involved in balancing multiple objectives in real-world



scenarios. The use of MATLAB facilitated the entire analysis, from data preprocessing and model fitting to predictions and the final output. The MATLAB code provided a clear, reproducible framework for implementing the regression models and evaluating their performance and using Multi-Objective Optimization (MOO).

Ultimately, this fifth chapter has demonstrated a comprehensive methodology for performing regression analysis and statistical evaluation. The techniques outlined, spanning data collection, regression modeling, statistical testing, and optimization, establish a solid foundation for understanding the relationships within the data. These methods provide crucial insights that will inform the conclusions and recommendations in the subsequent chapters of this thesis.

# Chapter VI

## Results and Discussions of Statistical Analysis and Optimization

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## Introduction

In this sixth chapter, the development of bio-composite materials with optimized properties is crucial for a wide range of industrial applications, particularly in fields where performance, durability, and environmental impact are key considerations. The bio-composite materials, which combine Date Palm Fibers (DPFs) with gypsum matrix, offer potential advantages such as sustainability, low weight, and cost-effectiveness. However, to maximize their performance, it is essential to optimize multiple material properties simultaneously. These properties include Charpy impact strength ( $G$ ), thermal conductivity ( $\lambda$ ), and water absorption (WAR), all of which play significant roles in determining the overall suitability of bio-composites for specific applications.

Charpy Impact strength ( $G$ ) measures a bio-composite material's resistance to sudden mechanical forces, which is essential in applications requiring high durability and resistance to stress. Water absorption (WAR) is an important factor, especially for bio-composite materials exposed to moisture, as high water absorption (WAR) can lead to a degradation of mechanical properties. Thermal conductivity ( $\lambda$ ) refers to a bio-composite material's ability to conduct heat, and in many applications, low thermal conductivity ( $\lambda$ ) is desirable for insulation purposes.

The evaluation is carried out using a combination of statistical tools, model fitting techniques, and advanced methods such as Analysis of Variance (ANOVA) to ensure the reliability and validity of the results. The sixth chapter is structured into three main results: Charpy impact strength ( $G$ ), water absorption (WAR), and thermal conductivity ( $\lambda$ ). Each result begins with the presentation of statistical analysis results, followed by model fit statistics, Analysis of Variance (ANOVA) findings, and predicted results, offering a detailed examination of the bio-composite material's behavior under different conditions.

Given the interdependencies between these properties, optimizing them in isolation is not practical. A Multi-Objective Optimization (MOO) approach allows for the simultaneous optimization of thermal conductivity, water absorption, and impact strength, resulting in bio-composite material configurations that strike the best possible balance between these objectives.

In this sixth chapter, a Multi-Objective Optimization (MOO) problem is formulated to minimize thermal conductivity ( $\lambda$ ) and water absorption (WAR), while maximizing Charpy impact strength ( $G$ ). The optimization variables considered are Date Palm Fiber (DPF) size ( $x_1$ ) and Date Palm Fiber (DPF) mass

ratio ( $x_2$ ), which significantly influence the bio-composite material's thermal and mechanical properties. The constraints on these variables, as well as a mechanical strength requirement for the gypsum, are integrated into the optimization problem.

Through the application of Pareto optimization, the study identifies a set of optimal solutions that represent the best trade-offs between these conflicting objectives. The results of this optimization process are presented in terms of Pareto-optimal solutions, which help identify the most balanced combinations of Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) to achieve desired bio-composite material properties. Furthermore, specific limiting solutions are examined to assess the performance of the bio-composite material under different optimization strategies, and the best compromise solution is identified.

Ultimately, this sixth chapter contributes valuable insights into the design of bio-composite materials with tailored properties that meet specific performance requirements, offering a pathway for developing high-performance, sustainable materials in engineering applications.

## VI.1. Charpy Impact Strength

The statistical significances were as follows:

### VI.1.1. Statistical Analysis Results

The statistical analysis results show that the Charpy impact strength ( $G$ ) was tested using three distinct types of models: linear, quadratic, and pure quadratic. Every model tries to explain how the predictors, the Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ), are related to the Charpy impact strength ( $G$ ), which is a feature of a bio-composite material that tells how tough it is. [Table VI.01](#). Shows the estimated coefficients ( $\beta$  or  $p$ ), Standard Error (SE), t-statistics (tStat), and p-values (p-value) for each model.

Table VI.01. The Statistical Results of Charpy Impact Strength.

| Model          |                  | Estimated Coefficients<br>( $\beta$ ) or (p) | Standard Error<br>(SE) | t-Statistic (tStat) | p-Value<br>(p-value) |
|----------------|------------------|----------------------------------------------|------------------------|---------------------|----------------------|
| Linear         | Intercept        | 0.2876                                       | 2.1910                 | 0.1313              | 9.00E-01             |
|                | $x_1$            | 4.4149                                       | 4.0286                 | 1.0959              | 3.15E-01             |
|                | $x_2$            | 19.3770                                      | 16.9600                | 1.1425              | 2.97E-01             |
| Quadratic      | Intercept        | -0.1275                                      | 4.2514                 | -0.0300             | 9.78E-01             |
|                | $x_1$            | 31.0720                                      | 14.7580                | 2.1054              | 1.26E-01             |
|                | $x_2$            | -08.7215                                     | 81.6680                | -0.1068             | 9.22E-01             |
|                | $x_1 \times x_2$ | 101.2900                                     | 65.8870                | 1.5373              | 2.22E-01             |
|                | $x_1^2$          | -64.3310                                     | 22.6100                | -2.8452             | 6.54E-02             |
|                | $x_2^2$          | -10.6000                                     | 392.2700               | -0.0270             | 9.80E-01             |
| Pure Quadratic | Intercept        | -3.1493                                      | 4.3652                 | -0.7215             | 5.11E-01             |
|                | $x_1$            | 41.2010                                      | 15.2920                | 2.6943              | 5.44E-02             |
|                | $x_2$            | 21.4970                                      | 91.7870                | 0.2342              | 8.26E-01             |
|                | $x_1^2$          | -64.3310                                     | 26.1820                | -2.4571             | 6.99E-02             |
|                | $x_2^2$          | -10.6000                                     | 454.2300               | -0.0233             | 9.83E-01             |

The results were as follows:

- 1. Linear Model:** The p-value for the intercept ( $\beta_0=0.2876$ ) is (p-value = 9.00E-01), which is very high. This means that the intercept doesn't actually assist explain the Charpy impact strength (G). The coefficient ( $\beta_1=4.4149$ ) for the size ( $x_1$ ) of the Date Palm Fiber (DPF) is what does. The p-value for this coefficient is (p-value = 3.15E-01), which is greater than the conventional cutoff for significance, which is 0.05. This shows that the size ( $x_1$ ) of the Date Palm Fiber (DPF) doesn't have a big effect on the Charpy impact strength (G). The coefficient ( $\beta_2=19.3770$ ) for the Date Palm Fiber (DPF) mass ratio ( $x_2$ ) has a p-value (p-value = 2.97E-01), which is also higher than 0.05. This suggests that the fiber mass ratio ( $x_2$ ) doesn't have a big effect on Charpy impact strength (G).
- 2. Quadratic Model:** The p-value for the intercept ( $\beta_0= -0.1275$ ) is (p-value = 9.78E-01), which means it is not statistically significant. The coefficient for the size ( $x_1$ ) of Date Palm Fiber (DPF)

is ( $\beta_1=31.0720$ ), and the p-value (p-value = 1.26E-01), which is 0.05 above the limit. This quadratic model demonstrates that Charpy impact strength (G) is unchanged by the size ( $x_1$ ) of Date Palm Fiber (DPF). The mass ratio ( $x_2$ ) coefficient ( $\beta_2=-8.7215$ ) has a p-value (p-value = 9.22E-01), which means that mass ratio ( $x_2$ ) does not have a big effect on the outcome.

The interaction term between Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) is ( $\beta_3= 101.2900$ ), with a p-value (p-value = 2.22E-01). This means that this interaction does not have a big effect on Charpy impact strength (G). The quadratic term for the size ( $x_1$ ) of Date Palm Fibers (DPFs) is ( $\beta_4= -64.3310$ ), which is written as ( $x_1^2$ ). The p-value (p-value = 6.54E-02), which is very near to the 0.05 level of significance. This implies a potential marginal influence of ( $x_1^2$ ) on Charpy impact strength (G). The quadratic term for mass ratio ( $x_2$ ), which is ( $x_2^2$ ), is ( $\beta_5= -10.6000$ ). The p-value (p-value = 9.80E-01), which means that ( $x_2^2$ ) does not have a big effect on the Charpy impact strength (G).

**3. Pure Quadratic Model:** Intercept ( $\beta_0 = -3.1493$ ): the p-value (p-value = 5.11E-01), indicating that the intercept is not statistically significant. For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = 41.2010$ ), the p-value (p-value = 5.44E-02), which is marginally greater than 0.05. This suggests a borderline significance for Date Palm Fiber (DPF) size ( $x_1$ ), possibly showing some influence on Charpy impact strength (G) but not strong enough to be definitive. For the date palm mass ratio ( $x_2$ ) coefficient ( $\beta_2 = 21.4970$ ), the p-value (p-value = 8.26E-01), which indicates that date palm mass ratio ( $x_2$ ) does not significantly impact the Charpy impact strength (G). For the ( $x_1^2$ ) coefficient ( $\beta_3 = -64.3310$ ), the p-value (p-value = 6.99E-02), which is also close to 0.05, indicating a potential but not significant influence of ( $x_1^2$ ) on the outcome. For the ( $x_2^2$ ) coefficient ( $\beta_4 = -10.6000$ ), the p-value (p-value = 9.83E-01), indicating no significant effect from ( $x_2^2$ ) on Charpy impact strength (G).

Across all models (Linear, Quadratic, and Pure Quadratic), no predictor shows strong statistical significance at the 0.05 level. The best candidates are the quadratic terms for Date Palm Fiber (DPF) size ( $x_1$ ) and the linear term for Date Palm Fiber (DPF) size ( $x_1$ ) in the pure quadratic model, though they are only marginally significant. Given the high p-values in most of the predictors across all models, we could infer that either the chosen predictors.

Ultimately, the current models provide limited insight into predicting Charpy impact strength (G), and further exploration or adjustment of the experimental design may be necessary.

### VI.1.2. Models Fit Statistics Results

**Table VI.02.** Evaluates the performance of three different models for predicting Charpy impact strength (G): linear, quadratic, and pure quadratic. The results are given in terms of the following performance metrics: number of observations (n), error degrees of freedom ( $df_{\text{Error}}$ ), Root Mean Squared Error (RMSE), R-squared ( $R^2$ ), adjusted R-squared ( $R^2_{\text{Adj}}$ ), F-statistic, and p-value.

**Table VI.02. Models Evaluation Statistics for Charpy Impact Strength Prediction.**

| Model          | Number of observations<br>(n) | Error degrees of freedom<br>( $df_{\text{Error}}$ ) | Root Mean Squared Error<br>(RMSE) | R-squared<br>( $R^2$ ) | Adjusted R-Squared<br>( $R^2_{\text{Adj}}$ ) | F-statistic vs. constant model<br>(F) | p-Value<br>(p-value) |
|----------------|-------------------------------|-----------------------------------------------------|-----------------------------------|------------------------|----------------------------------------------|---------------------------------------|----------------------|
| Linear         | 9                             | 6                                                   | 2.080                             | 0.295                  | 0.059                                        | 1.250                                 | 3.51E-01             |
| Quadratic      | 6                             | 3                                                   | 1.390                             | 0.843                  | 0.581                                        | 3.220                                 | 1.83E-01             |
| Pure Quadratic | 9                             | 4                                                   | 1.610                             | 0.719                  | 0.438                                        | 2.560                                 | 1.93E-01             |

The results were as follows:

- 1. Linear Model:** Number of observations ( $n=9$ ), error degrees of freedom ( $df_{\text{Error}} = 6$ ), and Root Mean Squared Error (RMSE) measures the average magnitude of errors in the model's predictions. A higher Root Mean Squared Error (RMSE) indicates larger prediction errors. In this case, the Root Mean Squared Error (RMSE = 2.080) suggests moderate error in the model's predictions. R-squared indicates the proportion of the variance in the dependent variable (Charpy impact strength (G)) that is explained by the model. An  $R^2$  ( $R^2 = 0.295$ ) means that only about 29.5% of the variation in Charpy impact strength (G) is explained by the linear model. This is relatively low, indicating that the linear model does not fit the data well. Adjusted R-squared accounts for the number of predictors in the model. A low  $R^2_{\text{Adj}}$  ( $R^2_{\text{Adj}} = 0.059$ ) suggests that even after adjusting for the number of predictors, the model still does not explain much of the

variability in Charpy impact strength (G). The F-statistic compares the fit of the model against a baseline model (A Constant Model). A value of ( $F = 1.250$ ) is relatively low, indicating that the linear model does not provide a much better fit than a constant model. The p-value associated with the F-statistic tests the null hypothesis that all model coefficients are equal to zero (i.e., No Relationship between the Predictors and the Outcome). A p-value ( $p\text{-value} = 3.51\text{E-}01$ ) is much higher than the typical significance threshold of 0.05, suggesting that the linear model is not statistically significant in predicting Charpy impact strength (G).

**2. Quadratic Model:** Number of observations ( $n = 6$ ), error degrees of freedom ( $df_{\text{Error}} = 3$ ), and the Root Mean Squared Error (RMSE) for the quadratic model is lower than the linear model ( $\text{RMSE} = 1.390$  vs.  $2.080$ ), indicating that the quadratic model has smaller errors in its predictions. This suggests that the quadratic model might provide a better fit to the data than the linear model. The  $R^2$  ( $R^2 = 0.843$ ) means that the quadratic model explains 84.3% of the variance in Charpy impact strength (G). This is a significant improvement compared to the linear model ( $R^2 = 0.295$ ), suggesting that the quadratic model fits the data much better. The Adjusted R-squared  $R^2$  ( $R^2_{\text{Adj}} = 0.581$ ) indicates that after accounting for the number of predictors, the quadratic model still explains a substantial portion of the variance in Charpy impact strength (G). The F-statistic ( $F = 3.220$ ) is relatively higher than that of the linear model ( $F = 1.250$ ), indicating that the quadratic model provides a significantly better fit than the constant model. The p-value ( $p\text{-value} = 1.83\text{E-}01$ ) suggests that the overall quadratic model is not statistically significant at the 0.05 significance level. While the model fits the data better than the linear model, this p-value indicates that there is still a reasonable probability that the observed fit is due to random chance.

**3. Pure Quadratic Model:** Number of observations ( $n = 9$ ), error degrees of freedom ( $df_{\text{Error}} = 4$ ), and the Root Mean Squared Error (RMSE) for the pure quadratic model is ( $\text{RMSE} = 1.610$ ), which is higher than that of the quadratic model ( $\text{RMSE} = 1.390$ ) but lower than the linear model ( $\text{RMSE} = 2.080$ ). This suggests that the pure quadratic model offers some improvement over the linear model but is less effective than the quadratic model. The  $R^2$  ( $R^2 = 0.719$ ) means that the pure quadratic model explains 71.9% of the variance in Charpy impact strength (G), which is better than the linear model ( $R^2 = 0.295$ ) but not as good as the quadratic model ( $R^2 = 0.843$ ). The adjusted  $R^2$  ( $R^2_{\text{Adj}} = 0.438$ ) indicates that, after adjusting for the number of predictors, the pure quadratic model explains a moderate portion of the variance



in Charpy impact strength (G). The F-statistic ( $F = 2.560$ ) is higher than that of the linear model ( $F = 1.250$ ), suggesting a better fit compared to the constant model. However, it is lower than the quadratic model ( $F = 3.220$ ), indicating that the pure quadratic model does not fit the data as well as the quadratic model. The p-value ( $p\text{-value} = 1.93\text{E-}01$ ) is greater than the typical 0.05 significance threshold, suggesting that the pure quadratic model is not statistically significant. While it performs better than the linear model, it does not provide a statistically significant improvement over the constant model.

The linear model has poor predictive performance, with low  $R^2$ , adjusted  $R^2$ , and a high p-value. It does not significantly improve upon using just a constant model. In addition, quadratic model, the quadratic model significantly improves over the linear model, with a much higher  $R^2$  and a lower Root Mean Squared Error (RMSE). However, the p-value ( $p\text{-value} = 1.83\text{E-}01$ ) suggests that the model is not statistically significant at the 0.05 level. In addition, the pure quadratic model: pure quadratic model provides some improvement over the linear model, but it is less effective than the quadratic model. The p-value ( $p\text{-value} = 1.93\text{E-}01$ ) indicates that this improvement is not statistically significant.

Ultimately, although the quadratic model provides the best fit in terms of  $R^2$  and Root Mean Squared Error (RMSE), the p-value indicates that the model's improvement over the linear model is not statistically significant at the 0.05 level. Therefore, while the quadratic model appears to fit the data better, the overall statistical significance is not strong enough to conclusively suggest that it is a better model than the linear one. The linear model still appears to be the most appropriate for predicting Charpy impact strength (G), given the context of these results.

### VI.1.3. Analysis of Variance (ANOVA) Results

**Table VI.03.** Shown the Sum of Squares (SumSq), degrees of freedom (DF), Mean Squares (MeanSq), F-statistic (F), and p-Value (p-value) for three models linear, quadratic, and pure quadratic to assess the significance of the predictor's Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) in modeling Charpy impact strength (G).

Table VI.03. ANOVA Summary for Models Comparison in Charpy Impact Strength Prediction.

| Model          | -         | Sum of Squares<br>(SumSq) | Degrees of Freedom<br>(DF) | The Mean Squares<br>(MeanSq) | The F-statistic<br>(F) | p-Value<br>(p-value) |
|----------------|-----------|---------------------------|----------------------------|------------------------------|------------------------|----------------------|
| Linear         | Total     | 36.7020                   | 8                          | 4.5877                       | 1.2531                 | 3.51E-01             |
|                | Model     | 10.8140                   | 2                          | 5.4068                       |                        |                      |
|                | Residual  | 25.8880                   | 6                          | 4.3147                       |                        |                      |
| Quadratic      | Total     | 36.7020                   | 8                          | 4.5877                       | 3.2162                 | 1.83E-01             |
|                | Model     | 30.9310                   | 5                          | 6.1863                       |                        |                      |
|                | Linear    | 10.8140                   | 2                          | 5.4068                       |                        |                      |
|                | Nonlinear | 20.1180                   | 3                          | 6.7059                       |                        |                      |
| Pure Quadratic | Residual  | 5.7703                    | 3                          | 1.9234                       | 2.5577                 | 1.93E-01             |
|                | Total     | 36.7020                   | 8                          | 4.5877                       |                        |                      |
|                | Model     | 26.3850                   | 4                          | 6.5963                       |                        |                      |
|                | Linear    | 10.8140                   | 2                          | 5.4068                       |                        |                      |
|                | Nonlinear | 15.5720                   | 2                          | 7.7859                       |                        |                      |
|                | Residual  | 10.3160                   | 4                          | 2.5791                       |                        |                      |

The results were as follows:

- 1. Linear Model:** Total Sum of Squares (SumSq = 36.7020), representing the total variability in the data. Model Sum of Squares (SumSq = 10.8140), which is the variation explained by the linear model. Residual Sum of Squares (SumSq = 25.8880), which indicates the unexplained variability (Error). The Mean Squares (MeanSq) for the model (MeanSq = 5.4068) and residuals (MeanSq = 4.3147) are calculated by dividing the respective Sum of Squares by their degrees of freedom (DF). F-statistic (F = 1.2531), which compares the variance explained by the model to the residual variance. The F-statistic is relatively low, suggesting that the linear model does not explain a significant proportion of the variability in Charpy impact strength (G) compared to the error. The p-value (p-value = 3.51E-01), which is greater than the typical threshold of 0.05, indicating that the linear model is not statistically significant in explaining the variability in Charpy impact strength (G).

**2. Quadratic Model:** Total Sum of Squares (SumSq = 36.7020), unchanged from the linear model, as it reflects the total variation in the dependent variable. Model Sum of Squares (SumSq = 30.9310), indicating a significant increase in the explained variation compared to the linear model (SumSq = 10.8140). This suggests that the quadratic model explains much more of the variance in Charpy impact strength (G). Residual Sum of Squares (SumSq = 5.7703), which is lower than the residual sum for the linear model (SumSq = 10.8140), indicating improved fit and less unexplained variability. Mean Squares (MeanSq), the quadratic model has a mean square of (MeanSq = 6.1863) for the model and (MeanSq = 1.9234) for the residuals. F-statistic ( $F = 3.2162$ ), which is higher than the linear model's F-statistic ( $F = 1.2531$ ), indicating that the quadratic model better explains the data. The p-value (p-value =  $1.83\text{E-}01$ ), which is still greater than 0.05, meaning the quadratic model is not statistically significant at the 5% significance level. Despite the higher F-statistic, the p-value suggests that the quadratic model does not provide enough evidence to reject the null hypothesis.

**3. Pure Quadratic Model:** Total Sum of Squares (SumSq = 36.7020), unchanged across all models. Model Sum of Squares (SumSq = 26.3850), which is slightly lower than the quadratic model's (SumSq = 30.9310), suggesting a marginally reduced explained variation compared to the quadratic model. Residual Sum of Squares (SumSq = 10.3160), which is higher than the quadratic model's residuals (SumSq = 5.7703), suggesting that the pure quadratic model leaves more unexplained variability. Mean squares (MeanSq), the pure quadratic model has a mean square of (MeanSq = 6.5963) for the model and (MeanSq = 2.5791) for the residuals. F-statistic ( $F = 2.5577$ ), which is slightly lower than the quadratic model's F-statistic ( $F = 3.2162$ ). This suggests that the pure quadratic model does not explain the data as well as the quadratic model. The p-value (p-value =  $1.93\text{E-}01$ ), which is greater than 0.05, indicating that the pure quadratic model is also not statistically significant.

In linear model, provides a poor fit for Charpy impact strength (G), with low F-statistic ( $F = 1.2531$ ) and a high p-value (p-value =  $3.51\text{E-}01$ ), indicating that it does not significantly explain the variance. In addition, quadratic model, explains more variance than the linear model F-statistic ( $F = 3.2162$ ), but still lacks statistical significance (p-value =  $1.83\text{E-}01$ ), suggesting limited improvement over the linear model. In addition, the pure quadratic model shows slightly reduced explained variance compared to the quadratic model F-statistic ( $F = 2.5577$ ), and is not statistically significant (p-value =  $1.93\text{E-}01$ ).

Ultimately, none of the models (Linear, Quadratic, or Pure Quadratic) is statistically significant at the 5% level ( $p$ -values  $> 0.05$ ). While the quadratic model explains more variance than the linear model, none of neither models provides conclusive evidence for a strong relationship between the predictors and Charpy impact strength ( $G$ ). This suggests that additional variables or a different modeling approach may be needed to predict Charpy impact strength ( $G$ ) better.

#### VI.1.4. The Predicted Results

**Table VI.04.** Shown the performance of bio-composite materials under various conditions defined by the size ( $x_1$ ) of the Date Palm Fiber (DPF) and mass ratio ( $x_2$ ). The materials were tested to determine their Charpy impact strength ( $G$ ) properties, measured in kilojoules per square meter ( $\text{KJ/m}^2$ ). Three different bio-composite materials were tested (GF.I, GF.II, and GF.III), with each having, variations in both Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ), to find three predictive results models (Linear, Quadratic, and Pure-Quadratic).

**Table VI.04. Predicted and Measured Charpy Impact Strength for Various Fiber Sizes and Mass Ratios.**

| Bio-composite Material | Test | $x_1$ (Size) | $x_2$ (Mass Ratio) | Measured [ $\text{KJ/m}^2$ ] | Linear [ $\text{KJ/m}^2$ ] | Quadratic [ $\text{KJ/m}^2$ ] | Pure-Quadratic [ $\text{KJ/m}^2$ ] |
|------------------------|------|--------------|--------------------|------------------------------|----------------------------|-------------------------------|------------------------------------|
| GF.I.05                | 1    | 0.080        | 05                 | $2.460 \pm 0.242$            | 1.610                      | 1.889                         | 0.783                              |
| GF.I.10                | 2    | 0.080        | 10                 | $1.840 \pm 0.043$            | 2.578                      | 1.779                         | 1.779                              |
| GF.I.15                | 3    | 0.080        | 15                 | $0.983 \pm 0.008$            | 3.547                      | 1.615                         | 2.721                              |
| GF.II.05               | 4    | 0.315        | 05                 | $3.167 \pm 0.123$            | 2.647                      | 4.410                         | 4.494                              |
| GF.II.10               | 5    | 0.315        | 10                 | $5.243 \pm 0.809$            | 3.616                      | 5.489                         | 5.489                              |
| GF.II.15               | 6    | 0.315        | 15                 | $8.005 \pm 0.756$            | 4.585                      | 6.516                         | 6.432                              |
| GF.III.05              | 7    | 0.500        | 05                 | $2.067 \pm 0.077$            | 3.464                      | 1.395                         | 2.417                              |
| GF.III.10              | 8    | 0.500        | 10                 | $3.597 \pm 0.106$            | 4.433                      | 3.412                         | 3.412                              |
| GF.III.15              | 9    | 0.500        | 15                 | $4.519 \pm 0.239$            | 5.402                      | 5.376                         | 4.354                              |

**Table VI.04.** Shown a clear relationship between size and mass ratio on Charpy impact strength ( $G$ ) across the different formulations. For instance, the bio-composite material (GF.I) series (With a Smaller Date Palm Fiber (DPF) Size of 0.080 mm) exhibited lower Charpy impact strength ( $G$ ) values, with a notable decrease as the size increased. In contrast, the bio-composite material (GF.II) series (With a Medium Date Palm Fiber (DPF) size of 0.315 mm) demonstrated significantly higher Charpy impact strength ( $G$ ), especially at larger sizes, with the highest Charpy impact strength ( $G$ ) observed at bio-composite material (GF.II.15) ( $8.005 \pm 0.756 \text{ KJ/m}^2$ ). The bio-composite material (GF.III) series

(With a Larger Date Palm Size of 0.500) showed a more consistent increase in Charpy impact strength (G), with bio-composite material (GF.III.15) achieving  $(4.519 \pm 0.239 \text{ KJ/m}^2)$ . The data also reveal variations between the measured, linear, quadratic, and pure-quadratic values, offering insight into the bio-composite's behavior under different conditions.

Ultimately, these findings underscore the influence of both date palm size ( $x_1$ ) and mass ratio ( $x_2$ ) on the bio-composite material's Charpy impact strength (G) capacity, highlighting the potential for optimizing material design based on these parameters.

**Figure VI.01.** Shown are the Charpy impact strength (G) values (Measured in  $\text{KJ/m}^2$ ) for various test conditions, including Date Palm Fiber (DPF) size ( $x_1$ ), mass ratio ( $x_2$ ), and three models (Linear, Quadratic, and Pure-Quadratic) results for prediction.

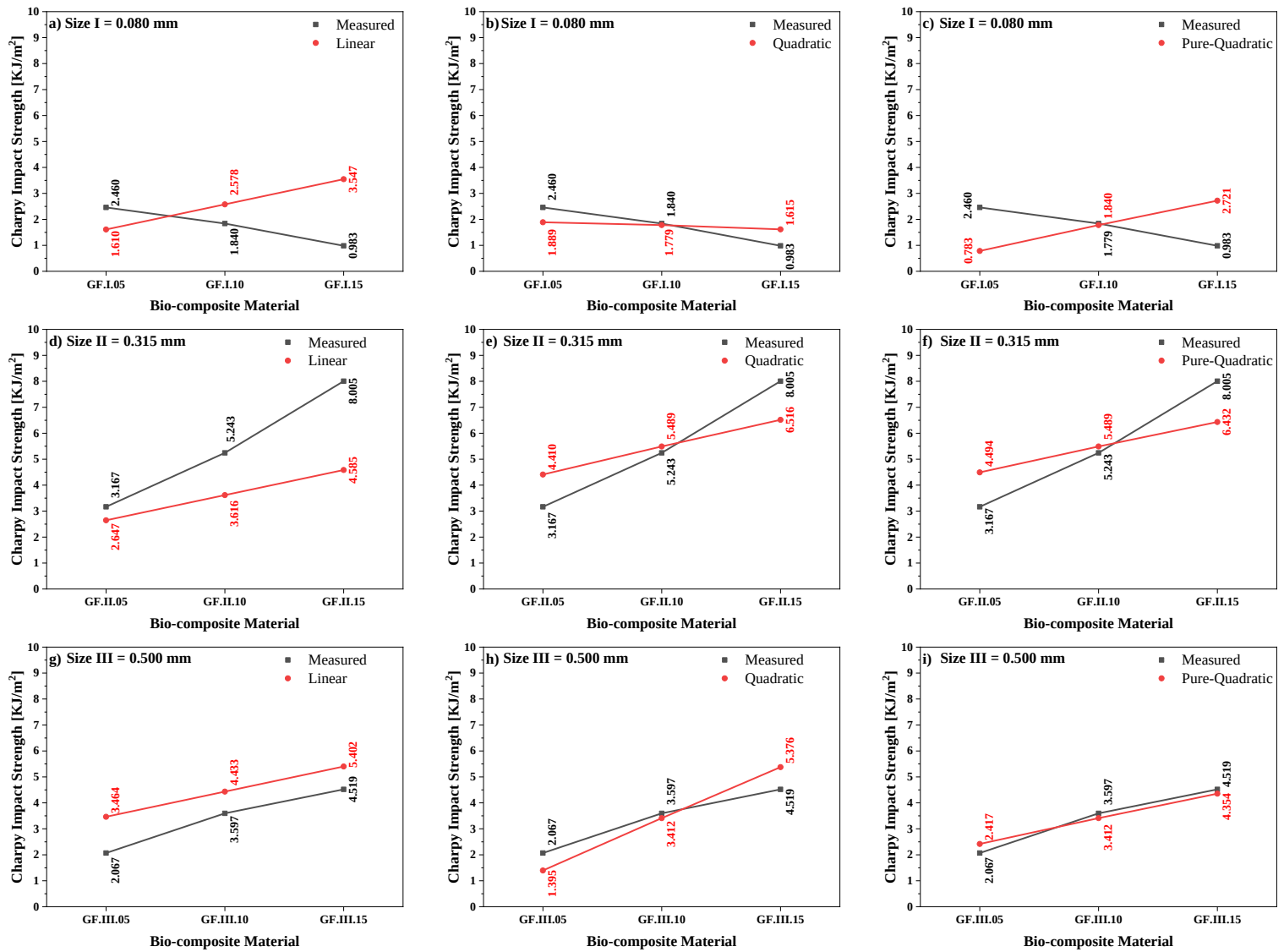


**Figure VI.01. Charpy Impact Strength Measurements versus Prediction Models:**  
a, d, and g) Linear; b, e, and h) Quadratic; and c, f, and i) Pure-Quadratic.

**Figure VI.01.** Shown that the bio-composite material (GF.I.05), the quadratic model provides the closest prediction to the measured value, but all models show discrepancies. The pure-quadratic model significantly underestimates the Charpy impact strength (G). The bio-composite material (GF.I.10), the pure-quadratic and quadratic models provide similar predictions, and both are reasonably close to the measured value. The linear model overestimates the impact strength. The bio-composite material (GF.I.15), the linear model provides a substantial overestimate, while the quadratic and pure-quadratic models are closer to the measured value, though still off. The bio-composite material (GF.II.05), both quadratic and pure-quadratic models overestimate the impact strength, but they are more accurate than the linear model, which underestimates the measured value. The bio-composite material (GF.II.10), the

quadratic and pure-quadratic models predict the measured value well, while the linear model underestimates the strength. The bio-composite material (GF.II.15), both the quadratic and pure-quadratic models predict values closer to the measured strength, but they still tend to underestimate the value. The linear model provides a more significant underestimation. The bio-composite material (GF.III.05), the pure-quadratic model provides the closest prediction to the measured value, while the linear model significantly overestimates and the quadratic model underestimates. The bio-composite material (GF.III.10), both the quadratic and pure-quadratic models, predict closely to the measured value. The linear model overestimates the strength. The bio-composite material (GF.III.15), the quadratic model provides the closest prediction, followed by the pure-quadratic model. The linear model overestimates.

Ultimately, the quadratic and pure-quadratic models tend to offer the best predictions for Charpy impact strength (G) across the test conditions, providing values closest to the measured values, especially for tests with a larger size ( $x_1$ ) and mass ratio ( $x_2$ ). The linear model generally underestimates or overestimates the impact strength, particularly when the combination of ( $x_1$ ) and ( $x_2$ ) is extreme (e.g., Higher Mass Ratios). For some tests (e.g., The Bio-composite Material (GF.I.05) and (GF.III.05)), the pure-quadratic model is more accurate than the quadratic model, suggesting it may better capture the non-linear interactions between size and mass ratio in these cases.

## VI.2. Water Absorption

The statistical significances were as follows:

### VI.2.1. Statistical Analysis Results

The statistical analysis results show that the water absorption (WAR) was tested using three distinct types of models: linear, quadratic, and pure quadratic. Every model tries to explain how the predictors, the Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ), are related to the water absorption, which is a feature of a bio-composite material that tells how tough it is. **Table VI.05.** Shows the estimated coefficients ( $\beta$  or  $p$ ), Standard Errors (SE), t-statistics (tStat), and p-values (p-value) for each model.

Table VI.05. The Statistical Results of Water Absorption.

| Model          |                  | Estimated Coefficients<br>( $\beta$ ) or (p) | Standard Error<br>(SE) | t-Statistic (tStat) | p-Value<br>(p-value) |
|----------------|------------------|----------------------------------------------|------------------------|---------------------|----------------------|
| Linear         | Intercept        | 31.8790                                      | 0.6701                 | 47.5780             | 5.78E-09             |
|                | $x_1$            | 9.2040                                       | 1.2320                 | 7.4705              | 2.97E-04             |
|                | $x_2$            | 56.9230                                      | 5.1868                 | 10.9750             | 3.40E-05             |
| Quadratic      | Intercept        | 31.3970                                      | 0.9837                 | 31.9160             | 6.76E-05             |
|                | $x_1$            | 17.0160                                      | 3.4149                 | 4.9829              | 1.55E-02             |
|                | $x_2$            | 39.1930                                      | 18.8970                | 2.0740              | 1.30E-01             |
|                | $x_1 \times x_2$ | -61.4630                                     | 15.2460                | -4.0316             | 2.74E-02             |
|                | $x_1^2$          | -2.9135                                      | 5.2318                 | -0.5569             | 6.16E-01             |
|                | $x_2^2$          | 180.3300                                     | 90.7670                | 1.9868              | 1.41E-01             |
| Pure Quadratic | Intercept        | 33.2300                                      | 1.9137                 | 17.3640             | 6.46E-05             |
|                | $x_1$            | 10.8700                                      | 6.7041                 | 1.6214              | 1.80E-01             |
|                | $x_2$            | 20.8570                                      | 40.2400                | 0.5183              | 6.32E-01             |
|                | $x_1^2$          | -2.9135                                      | 11.4780                | -0.2538             | 8.12E-01             |
|                | $x_2^2$          | 180.3300                                     | 199.1400               | 0.9056              | 4.16E-01             |

The results were as follows:

**1. Linear Model:** For the intercept ( $\beta_0 = 31.8790$ ), the p-value for the intercept is (p-value = 5.78E-09), which is highly significant. This suggests that the intercept is statistically important in explaining water absorption. For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = 9.2040$ ), the p-value for this coefficient is (p-value = 2.97E-04), which is extremely significant, indicating that Date Palm Fiber (DPF) size ( $x_1$ ) has a strong and significant effect on water absorption. For the mass ratio ( $x_2$ ) coefficient ( $\beta_2 = 56.9230$ ), the p-value is (p-value = 3.40E-05), indicating that mass ratio ( $x_2$ ) also has a highly significant effect on water absorption.

**2. Quadratic Model:** For the intercept ( $\beta_0 = 31.3970$ ), the p-value (p-value = 6.76E-05), indicating that the intercept is highly significant, although slightly lower than in the linear model.



For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = 17.0160$ ), the p-value for Date Palm Fiber (DPF) size ( $x_1$ ) (p-value =  $1.55E-02$ ), which is statistically significant at the 0.05 level. This shows that Date Palm Fiber (DPF) size ( $x_1$ ) has a significant linear effect on water absorption (WAR) in the quadratic model. For the mass ratio ( $x_2$ ) coefficient ( $\beta_2 = 39.1930$ ), the p-value for mass ratio ( $x_2$ ) is (p-value =  $1.30E-01$ ), which is greater than 0.05, suggesting that mass ratio ( $x_2$ ) is not statistically significant in this quadratic model.

For the interaction term ( $\beta_3 = -61.4630$ ), the p-value (p-value =  $2.74E-02$ ), which is less than 0.05, indicating that the interaction between Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) significantly affects water absorption. For the ( $x_1^2$ ) ( $\beta_4 = -2.9135$ ), the p-value for ( $x_1^2$ ) is (p-value =  $6.16E-01$ ), which is greater than 0.05, suggesting that the quadratic term for Date Palm Fiber (DPF) size ( $x_1$ ) does not have a significant impact on water absorption. For the ( $x_2^2$ ) ( $\beta_5 = 180.3300$ ), the p-value is (p-value =  $1.41E-01$ ), which is greater than 0.05, indicating no significant effect of ( $x_2^2$ ) on water absorption (G).

**3. Pure Quadratic Model:** For the intercept ( $\beta_0 = 33.2300$ ), the p-value is (p-value =  $1.80E-01$ ), indicating that the intercept remains highly significant in the pure quadratic model. For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = 10.8700$ ), the p-value for Date Palm Fiber (DPF) size ( $x_1$ ) is (p-value =  $6.32E-01$ ), which is greater than 0.05, suggesting that Date Palm Fiber (DPF) size ( $x_1$ ) is not significantly related to water absorption (WAR) in the pure quadratic model. For the mass ratio ( $x_2$ ) coefficient ( $\beta_2 = 20.8570$ ), the p-value (p-value =  $8.12E-01$ ), which is much higher than 0.05, indicating that mass ratio ( $x_2$ ) does not have a significant effect on water absorption (WAR) in this model. For the ( $x_1^2$ ) coefficient ( $\beta_3 = -2.9135$ ), the p-value is (p-value =  $4.16E-01$ ), which is very high, suggesting that ( $x_1^2$ ) has no significant impact on water absorption (WAR) in the pure quadratic model. For the ( $x_2^2$ ) coefficient ( $\beta_4 = 180.3300$ ), the p-value is (p-value =  $5.78E-09$ ), which also indicates a significant effect of ( $x_2^2$ ) on water absorption.

In the linear model, both Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) are highly significant predictors of water absorption, with p-values well below 0.05. The model suggests a strong, positive relationship between both variables and the outcome. In addition, in the quadratic model, the quadratic model shows that Date Palm Fiber (DPF) size ( $x_1$ ) and the interaction term ( $x_1 \times x_2$ ) are significant, with mass ratio ( $x_2$ ) and the quadratic terms showing no significant contribution. This suggests that while the

interaction between Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) might modify the effect on water absorption (G), the quadratic effects are negligible. In addition, in the pure quadratic model, this model does not add much value, as neither the linear nor the quadratic terms for Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) are statistically significant. The results suggest that this model is not appropriate for explaining water absorption.

Ultimately, the linear model provides the best fit for predicting water absorption, with both Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) showing strong statistical significance. The quadratic model offers some additional insight, particularly regarding the interaction between Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ), but quadratic effects are not significant. The pure quadratic model, however, does not improve upon the other models and shows.

### VI.2.2. Models Fit Statistics Results

**Table VI.06.** Evaluates the performance of three different models for predicting water absorption: linear, quadratic, and pure quadratic. The results are given in terms of the following performance metrics: number of observations (n), error degrees of freedom ( $df_{Error}$ ), Root Mean Squared Error (RMSE), R-squared ( $R^2$ ), adjusted R-squared ( $R^2_{Adj}$ ), F-statistic, and p-value.

**Table VI.06. Models Evaluation Statistics for Water Absorption Prediction.**

| Model          | Number of observations<br>(n) | Error degrees of freedom<br>( $df_{Error}$ ) | Root Mean Squared Error<br>(RMSE) | R-squared<br>( $R^2$ ) | Adjusted R-Squared<br>( $R^2_{Adj}$ ) | F-statistic vs. constant<br>model<br>(F) | p-Value<br>(p-value) |
|----------------|-------------------------------|----------------------------------------------|-----------------------------------|------------------------|---------------------------------------|------------------------------------------|----------------------|
| Linear         | 9                             | 6                                            | 0.635                             | 0.967                  | 0.956                                 | 088.10                                   | 3.57E-05             |
| Quadratic      | 9                             | 3                                            | 0.321                             | 0.996                  | 0.989                                 | 142.00                                   | 9.21E-04             |
| Pure Quadratic | 9                             | 4                                            | 0.704                             | 0.973                  | 0.946                                 | 036.10                                   | 2.14E-03             |

The results were as follows:

- 1. Linear Model:** Number of observations ( $n = 9$ ), error degrees of freedom ( $df_{\text{Error}} = 6$ ), and Root Mean Squared Error (RMSE) quantifies the average magnitude of errors in the model's predictions. An Root Mean Squared Error (RMSE = 0.635) indicates that the linear model has moderate error in its predictions, though this can be improved upon by other models. The  $R^2$  ( $R^2 = 0.967$ ) means that the linear model explains 96.7% of the variance in the dependent variable (Water Absorption). This is a very high  $R^2$ , suggesting that the linear model fits the data well. Adjusted  $R^2$  takes into account the number of predictors in the model. A value of ( $R^2_{\text{Adj}} = 0.956$ ) suggests that, after adjusting for the number of predictors, the linear model still explains a large proportion of the variance, making it an effective model. The F-statistic compares the fit of the model to a constant model (No Predictors). An F-statistic of ( $F = 88.10$ ) is quite large, indicating that the linear model significantly improves the fit over the constant model. The p-value associated with the F-statistic is very small (p-value =  $3.57\text{E-}05$ ), far below the typical significance threshold of 0.05. This indicates that the linear model is highly statistically significant and provides a strong relationship between the predictors and water absorption.
- 2. Quadratic Model:** Number of observations ( $n = 9$ ), error degrees of freedom ( $df_{\text{Error}} = 3$ ), and the Root Mean Squared Error (RMSE) of 0.321 for the quadratic model is lower than the Root Mean Squared Error (RMSE) of the linear model (RMSE = 0.635). This suggests that the quadratic model has smaller prediction errors and may fit the data better. The  $R^2$  ( $R^2 = 0.996$ ) means that the quadratic model explains 99.6% of the variance in water absorption. This is an extremely high  $R^2$ , suggesting an excellent fit of the quadratic model to the data. Adjusted R-squared ( $R^2_{\text{Adj}} = 0.989$ ) indicates that, after accounting for the number of predictors, the quadratic model still explains a very high proportion of the variance in the outcome. This demonstrates that the quadratic model is a very good fit. The F-statistic of ( $F = 142.00$ ) is much higher than the linear model's F-statistic ( $F = 88.10$ ), indicating that the quadratic model provides a significantly better fit than the constant model. The p-value is very small (p-value =  $9.21\text{E-}04$ ), well below 0.05, indicating that the quadratic model is highly statistically significant and provides a meaningful improvement over the constant model.

**3. Pure Quadratic Model:** Number of observations ( $n = 9$ ), error degrees of freedom ( $df_{\text{Error}} = 4$ ), the Root Mean Squared Error (RMSE) for the pure quadratic model ( $\text{RMSE} = 0.704$ ) is higher than both the linear ( $\text{RMSE} = 0.635$ ) and quadratic ( $\text{RMSE} = 0.321$ ) models. This indicates that the pure quadratic model performs worse in terms of prediction accuracy compared to the other models. The  $R^2$  ( $R^2 = 0.973$ ) means that the pure quadratic model explains 97.3% of the variance in water absorption. While this is still a very good fit, it is lower than the quadratic model ( $R^2 = 0.996$ ) and the linear model ( $R^2 = 0.967$ ). The adjusted  $R^2$  ( $R^2_{\text{Adj}} = 0.946$ ) indicates that after adjusting for the number of predictors, the pure quadratic model still explains a large proportion of the variance. However, it is again lower than the quadratic model's adjusted  $R^2$  ( $R^2_{\text{Adj}} = 0.989$ ). The F-statistic of ( $F = 36.10$ ) is lower than both the linear ( $F = 88.10$ ) and quadratic ( $F = 142.00$ ) models, indicating that the pure quadratic model does not fit the data as well as the other two models. The p-value of ( $p\text{-value} = 2.14\text{E-}03$ ) is below 0.05, which indicates that the pure quadratic model is statistically significant. However, its significance is less robust compared to the quadratic model, which had a much smaller p-value.

In linear model, the linear model fits the data well, with a high  $R^2$  ( $R^2 = 0.967$ ) and a very small p-value ( $p\text{-value} = 3.57\text{E-}05$ ). However, its Root Mean Squared Error ( $\text{RMSE} = 0.635$ ) is higher than the quadratic model is, suggesting that other models may fit better. In addition, quadratic model, the quadratic model significantly outperforms the linear model, with a very low Root Mean Squared Error ( $\text{RMSE} = 0.321$ ), an  $R^2$  ( $R^2 = 0.996$ ), and a p-value ( $p\text{-value} = 9.21\text{E-}04$ ) indicating that it is highly statistically significant. It provides the best fit for the data. In addition, pure quadratic model, the pure quadratic model performs worse than the quadratic model, with higher Root Mean Squared Error ( $\text{RMSE} = 0.704$ ) and lower  $R^2$  ( $R^2 = 0.973$ ). While still significant p-value ( $p\text{-value} = 2.14\text{E-}03$ ), it is less effective than the quadratic model.

Ultimately, the quadratic model is the best model for predicting water absorption, as it explains the most variance ( $R^2 = 0.996$ ), has the lowest Root Mean Squared Error ( $\text{RMSE} = 0.321$ ), and is highly statistically significant. The linear model is also effective, but its performance is not as strong as the quadratic model. The pure quadratic model is the least effective, providing only marginal improvements over the simpler quadratic model. Therefore, the quadratic model should be preferred for predicting water absorption (WAR) in this case.

### VI.2.3. Analysis of Variance (ANOVA) Results

**Table VI.07.** Shown the Sum of Squares (SumSq), Degrees of Freedom (DF), Mean Squares (MeanSq), F-statistic (F), and p-Value (p-Value) for three models linear, quadratic, and pure quadratic to assess the significance of the predictor's Date Palm Fiber (DPF) size ( $x_1$ ) and Date Palm Fiber (DPF) mass ratio ( $x_2$ ) in modeling Charpy impact strength (G).

**Table VI.07. ANOVA Summary for Models Comparison in Water Absorption Prediction.**

| Model          | -         | Sum of Squares<br>(SumSq) | Degrees of Freedom<br>(DF) | The Mean Squares<br>(MeanSq) | The F-statistic<br>(F) | p-Value<br>(p-value) |
|----------------|-----------|---------------------------|----------------------------|------------------------------|------------------------|----------------------|
| Linear         | Total     | 73.5460                   | 8                          | 09.1933                      | 088.1260               | 3.57E-05             |
|                | Model     | 71.1250                   | 2                          | 35.5630                      |                        |                      |
|                | Residual  | 02.4212                   | 6                          | 00.4035                      |                        |                      |
| Quadratic      | Total     | 73.5460                   | 8                          | 09.1933                      | 142.2300               | 9.21E-04             |
|                | Model     | 73.2370                   | 5                          | 14.6470                      |                        |                      |
|                | Linear    | 71.1250                   | 2                          | 35.5630                      |                        |                      |
|                | Nonlinear | 02.1123                   | 3                          | 00.7041                      |                        |                      |
| Pure Quadratic | Residual  | 00.3090                   | 3                          | 00.1030                      | 006.8370               | 7.43E-02             |
|                | Total     | 73.5460                   | 8                          | 09.1933                      | 36.0920                | 2.14E-03             |
|                | Model     | 71.5630                   | 4                          | 17.8910                      |                        |                      |
|                | Linear    | 71.1250                   | 2                          | 35.5630                      |                        |                      |
|                | Nonlinear | 00.4384                   | 2                          | 00.2192                      |                        |                      |
|                | Residual  | 01.9828                   | 4                          | 00.4957                      |                        |                      |

The results were as follows:

- 1. Linear Model:** Total Sum of Squares (SumSq = 73.5460), representing the total variability in the water absorption (WAR) data. Model Sum of Squares (SumSq = 71.1250), indicating that the linear model explains most of the variation in the data (96.7% of Total Variance). Residual Sum of Squares (SumSq = 2.4212), showing very little unexplained variance, which suggests the linear model fits the data well. Mean squares (MeanSq = 35.5630), and the residual mean square is (MeanSq = 0.4035). F-statistic (F = 88.1260), a very high F-statistic, suggesting that the model explains a significantly greater proportion of the variance than the residual error. The p-value

(p-value = 3.57E-05), which is extremely small and well below the threshold of 0.05. This indicates that the linear model is highly statistically significant, providing strong evidence that the predictors influence water absorption.

**2. Quadratic Model:** Total Sum of Squares (SumSq = 73.5460), same as for the linear model, representing total variability. Model Sum of Squares (SumSq = 73.2370), showing an even higher proportion of explained variance compared to the linear model (99.6% of Total Variance). Residual Sum of Squares (SumSq = 0.3090), indicating that the quadratic model has very low unexplained variance, suggesting it is a very good fit. Mean squares (MeanSq), the quadratic model's mean square is (MeanSq = 14.6470), and the residual mean square is (MeanSq = 0.1030). F-statistic ( $F = 142.2300$ ), which is significantly higher than the F-statistic for the linear model, showing that the quadratic model explains more of the variance. The p-value (p-value = 9.21E-04), which is well below the 0.05 threshold, indicating that the quadratic model is statistically significant.

**3. Pure Quadratic Model:** Total Sum of Squares (SumSq = 73.5460), consistent with the previous models. Model Sum of Squares (SumSq = 71.5630), showing that the pure quadratic model explains most of the variance, though slightly less than the quadratic model (97.3% vs. 99.6%). Residual Sum of Squares (SumSq = 1.9828), which is higher than the quadratic model's residuals (SumSq = 0.3090), indicating slightly more unexplained variance. The mean square (MeanSq) for the pure quadratic model is (MeanSq = 17.8910), and for the residuals, it is (MeanSq = 0.4957). F-statistic ( $F = 36.0920$ ), which is still quite high, but lower than the quadratic model's F-statistic ( $F = 142.2300$ ), suggesting that the pure quadratic model is not an improvement over the quadratic model. The p-value (p-value = 2.14E-03), which is significant, but higher than the quadratic model's p-value (p-value = 9.21E-04).

In linear model, the linear model explains most of the variance in water absorption (WAR) (96.7%) and is highly statistically significant (p-value = 3.57E-05). In addition, the quadratic model, the quadratic model explains nearly all of the variance (99.6%) and provides a better fit than the linear model, with a significantly higher F-statistic ( $F = 142.2300$ ) and a statistically significant p-value (p-value = 9.21E-04). In addition, the pure quadratic model still explains most of the variance (97.3%) but is less effective than the quadratic model (Lower F-statistic and Higher Residuals), with a p-value of (p-value = 2.14E-03) indicating statistical significance.

Ultimately, the quadratic model is the most effective in explaining water absorption, as it provides the highest F-statistic and best overall fit to the data. While the pure quadratic model is statistically significant, it does not offer a notable improvement over the quadratic model, suggesting that the additional complexity may not be justified. Therefore, the quadratic model is the most appropriate for predicting water absorption (WAR) in this case.

#### VI.2.4. The Predicted Results

**Table VI.08.** Shown is the performance of bio-composite materials under various conditions defined by the size of the Date Palm Fiber (DPF) ( $x_1$ ) and mass ratio ( $x_2$ ). The materials were tested to determine their water absorption (WAR) properties, measured in percentage (%). Three different bio-composite materials were tested (GF.I, GF.II, and GF.III), with each having, variations in both Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ), to find three predictive results models (Linear, Quadratic, and Pure-Quadratic).

**Table VI.08. Predicted and Measured Water Absorption for Various Fiber Sizes and Mass Ratios.**

| Bio-composite Material | Test | $x_1$<br>(Size) | $x_2$<br>(Mass Ratio) | Measured [%]       | Linear [%] | Quadratic [%] | Pure-Quadratic [%] |
|------------------------|------|-----------------|-----------------------|--------------------|------------|---------------|--------------------|
| GF.I.05                | 1    | 0.080           | 05                    | $34.878 \pm 3.082$ | 35.462     | 34.904        | 35.575             |
| GF.I.10                | 2    | 0.080           | 10                    | $38.132 \pm 1.893$ | 38.308     | 37.970        | 37.970             |
| GF.I.15                | 3    | 0.080           | 15                    | $41.803 \pm 1.157$ | 41.154     | 41.938        | 41.267             |
| GF.II.05               | 4    | 0.315           | 05                    | $37.957 \pm 3.092$ | 37.625     | 37.910        | 37.859             |
| GF.II.10               | 5    | 0.315           | 10                    | $39.913 \pm 1.596$ | 40.471     | 40.254        | 40.254             |
| GF.II.15               | 6    | 0.315           | 15                    | $43.795 \pm 2.961$ | 43.317     | 43.500        | 43.551             |
| GF.III.05              | 7    | 0.500           | 05                    | $40.030 \pm 2.521$ | 39.328     | 40.051        | 39.431             |
| GF.III.10              | 8    | 0.500           | 10                    | $42.006 \pm 3.224$ | 42.174     | 41.826        | 41.826             |
| GF.III.15              | 9    | 0.500           | 15                    | $44.344 \pm 1.852$ | 45.020     | 44.503        | 45.123             |

**Table VI.08.** Shown as the mass ratio ( $x_2$ ) increases, the bio-composite material has measured water absorption (WAR) that also increases across all series. The bio-composite material (GF.I) series, with a smaller Date Palm Fiber (DPF) size ( $x_1 = 0.080$ ), exhibited an increase in water absorption (WAR) from the bio-composite material (GF.I.05) ( $34.878 \pm 3.082\%$ ) at ( $x_2 = 05$ ) mass ratio to the bio-composite material (GF.I.15) ( $41.803 \pm 1.157\%$ ) at ( $x_2 = 15$ ) mass ratio. In the bio-composite material (GF.II) series, a medium Date Palm Fiber (DPF) size ( $x_1 = 0.315$ ) produced similar trends, with water absorption (WAR) values ranging from the bio-composite material (GF.II.05) ( $37.957 \pm 3.092\%$ ) at ( $x_2 = 05$ ) mass

ratio to the bio-composite material (GF.II.15) ( $43.795 \pm 2.961\%$ ) at ( $x_2 = 15$ ). The bio-composite material (GF.III) series, with the largest Date Palm Fiber (DPF) size ( $x_1 = 0.500$ ), showed the highest water absorption, reaching the bio-composite material (GF.III.05) ( $44.344 \pm 1.852\%$ ) at ( $x_2 = 15$ ) mass ratio. The comparison of measured values with linear, quadratic, and pure-quadratic estimates indicates that the bio-composite materials behavior aligns more closely with the quadratic and pure-quadratic predictions, suggesting a non-linear relationship between size ( $x_1$ ), mass ratio ( $x_2$ ), and material performance (Water Absorption).

Ultimately, these findings highlight the potential for optimizing bio-composite materials based on date palm size and mass ratio to achieve enhanced performance in various applications.

**Figure VI.02.** Shown are the water absorption (WAR) values (Measured in %) for various test conditions, including Date Palm Fiber (DPF) size ( $x_1$ ), mass ratio ( $x_2$ ), and three models (Linear, Quadratic, and Pure-Quadratic) results for prediction.



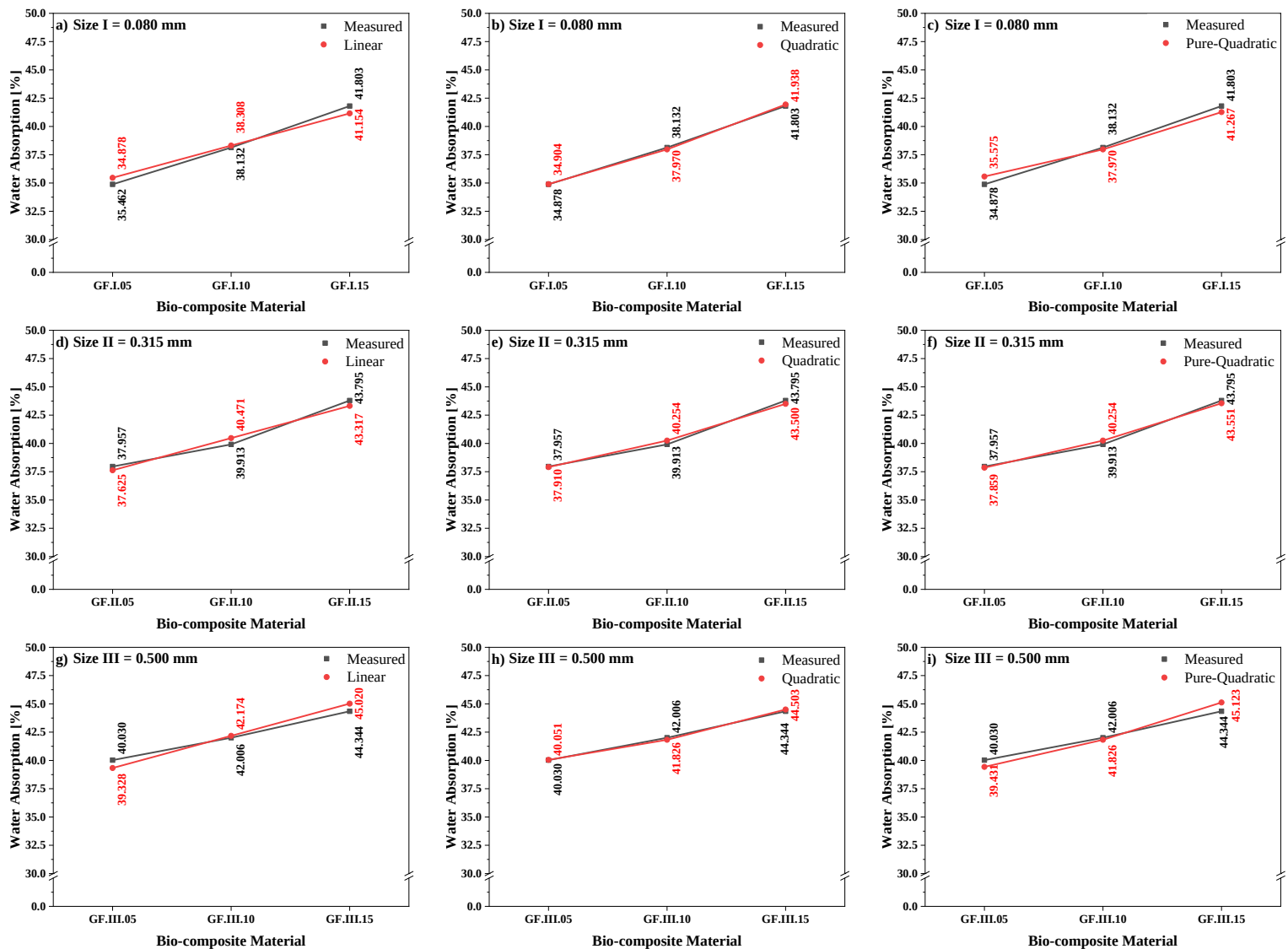


**Figure VI.02. Water Absorption Measurements versus Prediction Models: a, d, and g) Linear; b, e, and h) Quadratic; and c, f, and i) Pure-Quadratic.**

**Figure VI.02.** Shown the bio-composite material (GF.I.05), the pure-quadratic model gives the closest prediction to the measured value, while the linear model slightly overestimates the water absorption. The quadratic model is also close. The bio-composite material (GF.I.10), the linear model gives a very close prediction, slightly overestimating the measured value. The quadratic and pure-quadratic models both provide nearly identical predictions and are close to the measured value. The bio-composite material (GF.I.15), the linear model provides the closest prediction, while the quadratic model slightly overestimates the absorption. The pure-quadratic model underestimates it. The bio-composite material (GF.II.05), all models are quite close to the measured value. The pure-quadratic model provides the most accurate prediction, while the linear model slightly underestimates it. The bio-composite material

(GF.II.10), both the quadratic and pure-quadratic models, provide identical predictions, which are very close to the measured value. The linear model slightly overestimates the water absorption. The bio-composite material (GF.II.15), the pure-quadratic model gives the best prediction, being closest to the measured value. The quadratic model is slightly off, and the linear model slightly underestimates. The bio-composite material (GF.III.05), the quadratic model provides the most accurate prediction, followed by the pure-quadratic model. The linear model slightly underestimates the value. The bio-composite material (GF.III.10), the linear model slightly overestimates the water absorption, while the quadratic and pure-quadratic models provide identical predictions, which are close to the measured value. The bio-composite material (GF.III.15), the quadratic model provides the closest prediction, while the linear and pure-quadratic models overestimate the absorption slightly.

Ultimately, the quadratic and pure-quadratic models provide the most accurate predictions of water absorption (WAR) across the different tests, with pure-quadratic being the closest in most cases. The linear model is generally less accurate, particularly when the mass ratio is high, as it often overestimates or underestimates the values. Overall, the models perform well, with the pure-quadratic model being the most reliable for predicting water absorption (WAR) across various sizes and mass ratios.

### VI.3. Thermal Conductivity

The statistical significances were as follows:

#### VI.3.1. Statistical Analysis Results

The statistical analysis results show that the thermal conductivity ( $\lambda$ ) was tested using three distinct types of models: linear, quadratic, and pure quadratic. Every model tries to explain how the predictors, the Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ), are related to the thermal conductivity, which is a feature of a bio-composite material that tells how tough it is. [Table VI.09](#). Shows the estimated coefficients ( $\beta$  or  $p$ ), Standard Errors (SE), t-statistics (tStat), and p-values (p-value) for each model.

Table VI.09. The Statistical Results of Thermal Conductivity.

| Model          |                  | Estimated Coefficients<br>( $\beta$ ) or (p) | Standard Error<br>(SE) | t-Statistic (tStat) | p-Value<br>(p-value) |
|----------------|------------------|----------------------------------------------|------------------------|---------------------|----------------------|
| Linear         | Intercept        | 0.6051                                       | 0.0159                 | 38.1470             | 2.17E-08             |
|                | $x_1$            | -0.2600                                      | 0.0292                 | -8.9135             | 1.11E-04             |
|                | $x_2$            | -1.3800                                      | 0.1228                 | -11.2380            | 2.97E-05             |
| Quadratic      | Intercept        | 0.6519                                       | 0.0451                 | 14.4700             | 7.15E-04             |
|                | $x_1$            | -0.1522                                      | 0.1564                 | -0.9729             | 4.02E-01             |
|                | $x_2$            | -2.6858                                      | 0.8654                 | -3.1035             | 5.32E-02             |
|                | $x_1 \times x_2$ | 0.2652                                       | 0.6982                 | 0.3798              | 7.29E-01             |
|                | $x_1^2$          | -0.2350                                      | 0.2396                 | -0.9806             | 3.99E-01             |
|                | $x_2^2$          | 6.1333                                       | 4.1568                 | 1.4755              | 2.37E-01             |
| Pure Quadratic | Intercept        | 0.6440                                       | 0.0354                 | 18.1830             | 5.38E-05             |
|                | $x_1$            | -0.1256                                      | 0.1241                 | -1.0126             | 3.69E-01             |
|                | $x_2$            | -2.6067                                      | 0.7447                 | -3.5002             | 2.49E-02             |
|                | $x_1^2$          | -0.2350                                      | 0.2124                 | -1.1060             | 3.31E-01             |
|                | $x_2^2$          | 6.1333                                       | 3.6854                 | 1.6642              | 1.71E-01             |

The results were as follows:

- 1. Linear Model:** For the intercept ( $\beta_0 = 0.6051$ ), the p-value is (p-value = 2.17E-08), which is highly significant, meaning the intercept is an important term in predicting thermal conductivity. For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = -0.2600$ ), the p-value is (p-value = 1.11E-04), which is very small, indicating that Date Palm Fiber (DPF) size ( $x_1$ ) has a significant negative effect on thermal conductivity. A negative coefficient suggests that as Date Palm Fiber (DPF) size ( $x_1$ ) increases, thermal conductivity ( $\lambda$ ) decreases. For the fiber mass ratio ( $x_2$ ) coefficient ( $\beta_2 = -1.3800$ ), the p-value is (p-value = 2.97E-05), showing that fiber mass ratio ( $x_2$ ) also has a significant negative effect on thermal conductivity. This suggests that increasing fiber mass ratio ( $x_2$ ) will lead to a decrease in thermal conductivity ( $\lambda$ ) as well.

**2. Quadratic Model:** For the intercept ( $\beta_0 = 0.6519$ ), the p-value is (p-value = 7.15E-04), which indicates a statistically significant intercept, similar to the linear model. For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = -0.1522$ ), the p-value is (p-value = 4.02E-01), which is greater than 0.05, suggesting that Date Palm Fiber (DPF) size ( $x_1$ ) does not have a statistically significant linear effect on thermal conductivity ( $\lambda$ ) in the quadratic model. This contrasts with the linear model where Date Palm Fiber (DPF) size ( $x_1$ ) was significant. For the fiber mass ratio ( $x_2$ ) coefficient ( $\beta_2 = -2.6858$ ), the p-value is (p-value = 5.32E-02), which is slightly above 0.05. This suggests that fiber mass ratio ( $x_2$ ) has a marginally significant negative effect on thermal conductivity ( $\lambda$ ) in the quadratic model.

For the interaction term ( $\beta_3 = 0.2652$ ), the p-value is (p-value = 7.29E-01), which is very high, indicating that the interaction between Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) does not significantly affect thermal conductivity. For the ( $x_1^2$ ) coefficient ( $\beta_4 = -0.2350$ ), the p-value is (p-value = 3.99E-01), indicating that the quadratic effect of Date Palm Fiber (DPF) size ( $x_1$ ) is not statistically significant. For the ( $x_2^2$ ) coefficient ( $\beta_5 = 6.1333$ ), the p-value is (p-value = 2.37E-01), which is above 0.05, suggesting that the quadratic effect of fiber mass ratio ( $x_2$ ) is also not statistically significant.

**3. Pure Quadratic Model:** For the intercept ( $\beta_0 = 0.6440$ ), the p-value is (p-value = 5.38E-05), suggesting that the intercept is highly significant in the pure quadratic model as well. For the Date Palm Fiber (DPF) size ( $x_1$ ) coefficient ( $\beta_1 = -0.1256$ ), the p-value is (p-value = 3.69E-01), which is greater than 0.05, indicating that Date Palm Fiber (DPF) size ( $x_1$ ) is not significantly related to thermal conductivity ( $\lambda$ ) in this model. For the fiber mass ratio ( $x_2$ ) coefficient ( $\beta_2 = -2.6067$ ), the p-value is (p-value = 2.49E-02), which is less than 0.05, suggesting that fiber mass ratio ( $x_2$ ) has a significant negative effect on thermal conductivity ( $\lambda$ ) in the pure quadratic model. For the ( $x_1^2$ ) coefficient ( $\beta_3 = -0.2350$ ), the p-value is (p-value = 3.31E-01), which is greater than 0.05, suggesting that the quadratic effect of Date Palm Fiber (DPF) size ( $x_1$ ) is not statistically significant in the pure quadratic model. For the ( $x_2^2$ ) coefficient ( $\beta_4 = 6.1333$ ), the p-value is (p-value = 1.71E-01), which is greater than 0.05, indicating that the quadratic effect of fiber mass ratio ( $x_2$ ) is not significant either.

In the linear model, both Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) are highly significant predictors of thermal conductivity, with negative coefficients indicating that increases in these variables

lead to decreases in thermal conductivity. The model is well-supported statistically, with all p-values being less than 0.05. In addition, the quadratic model, the quadratic model shows that while the intercept and fiber mass ratio ( $x_2$ ) are significant, the linear term for Date Palm Fiber (DPF) size ( $x_1$ ) and the interaction and quadratic terms for both Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) are not. This suggests that adding quadratic terms does not significantly improve the model for predicting thermal conductivity. In addition, the pure quadratic model, in this model fiber mass ratio ( $x_2$ ) has a significant negative linear effect on thermal conductivity, but none of the other terms (Including Date Palm Fiber (DPF) size ( $x_1$ ) and the Quadratic Terms) are significant. This model also does not provide any major improvements over the linear model.

Ultimately, the linear model provides the best explanation for thermal conductivity, with both Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) showing strong, statistically significant effects. The quadratic model does not substantially improve upon the linear model, as most of the additional terms (Such as Interactions and Quadratic Effects) are not significant. Similarly, the pure quadratic model does not offer significant improvements, with fiber mass ratio ( $x_2$ ) being the only relevant predictor in the model. Therefore, the linear model is the most appropriate model for predicting thermal conductivity ( $\lambda$ ) in this case.

### VI.3.2. Models Fit Statistics Results

**Table VI.10.** Evaluates the performance of three different models for predicting thermal conductivity: linear, quadratic, and pure quadratic. The results are given in terms of the following performance metrics: number of observations (n), error degrees of freedom ( $df_{\text{Error}}$ ), Root Mean Squared Error (RMSE), R-squared ( $R^2$ ), adjusted R-squared ( $R^2_{\text{Adj}}$ ), F-statistic, and p-value.

**Table VI.10. Model Evaluation Statistics for Thermal Conductivity Prediction.**

| Model                 | Number of observations<br>(n) | Error degrees of freedom<br>(df <sub>Error</sub> ) | Root Mean Squared Error<br>(RMSE) | R-squared<br>(R <sup>2</sup> ) | Adjusted R-Squared<br>(R <sup>2</sup> <sub>Adj</sub> ) | F-statistic vs. constant model<br>(F) | p-Value<br>(p-value) |
|-----------------------|-------------------------------|----------------------------------------------------|-----------------------------------|--------------------------------|--------------------------------------------------------|---------------------------------------|----------------------|
| <b>Linear</b>         | 9                             | 6                                                  | 0.015                             | 0.972                          | 0.962                                                  | 103.00                                | 2.27E-05             |
| <b>Quadratic</b>      | 6                             | 3                                                  | 0.014                             | 0.986                          | 0.964                                                  | 043.70                                | 5.28E-03             |
| <b>Pure Quadratic</b> | 9                             | 4                                                  | 0.013                             | 0.986                          | 0.972                                                  | 069.50                                | 5.98E-04             |

The results were as follows:

- 1. Linear Model:** Number of observations ( $n = 9$ ), error degrees of freedom ( $df_{\text{Error}} = 6$ ), and the Root Mean Squared Error ( $RMSE = 0.015$ ) indicate that the linear model has very small prediction errors, suggesting a high level of accuracy in its predictions for thermal conductivity. The  $R^2$  ( $R^2 = 0.972$ ) means that the linear model explains 97.2% of the variance in thermal conductivity. This is an extremely high  $R^2$ , suggesting that the linear model fits the data very well. Adjusted  $R^2$  ( $R^2_{\text{Adj}} = 0.962$ ) accounts for the number of predictors in the model, and this value still indicates a very good fit, suggesting that the model explains a substantial portion of the variability in thermal conductivity ( $\lambda$ ) after adjusting for the predictors. The F-statistic of ( $F = 103.00$ ) is quite large, indicating that the linear model is significantly better than a constant model (A Model with no Predictors). The p-value ( $p\text{-value} = 27\text{E-}05$ ) is very small, far below the typical threshold of 0.05. This indicates that the linear model is statistically significant, providing strong evidence that the predictors have a meaningful relationship with the thermal conductivity.
- 2. Quadratic Model:** Number of observations ( $n = 6$ ), error degrees of freedom ( $df_{\text{Error}} = 3$ ), and the Root Mean Squared Error ( $RMSE$ ) for the quadratic model ( $RMSE = 0.014$ ) is slightly lower than that of the linear model (0.015), indicating that the quadratic model has marginally smaller prediction errors. The  $R^2$  ( $R^2 = 0.986$ ) means that the quadratic model explains 98.6% of the variance in thermal conductivity, which is a slight improvement over the linear model ( $R^2 = 0.972$ ). This indicates that the quadratic model offers a better fit to the data than the linear

model. Adjusted  $R^2$  of ( $R^2_{Adj} = 0.964$ ) is very high and shows that, after adjusting for the number of predictors, the quadratic model still explains a substantial portion of the variance in thermal conductivity. This suggests that the model is a good fit, but not drastically better than the linear model ( $R^2_{Adj} = 0.962$ ). The F-statistic of ( $F = 43.70$ ) indicates that the quadratic model fits the data significantly better than a constant model, though it is lower than the linear model's F-statistic ( $F = 103.00$ ). This suggests that the quadratic model offers a good improvement over the constant model, but not as strong as the linear model. The p-value of ( $p\text{-value} = 5.28E-03$ ) is small and below the typical significance threshold of 0.05, indicating that the quadratic model is statistically significant and provides a meaningful improvement over the constant model.

**3. Pure Quadratic Model:** Number of observations ( $n = 9$ ), error degrees of freedom ( $df_{Error} = 4$ ), and the Root Mean Squared Error (RMSE) for the pure quadratic model ( $RMSE = 0.013$ ) is the lowest among the three models, indicating that it provides the smallest prediction errors and is the most accurate in its predictions. The  $R^2$  ( $R^2 = 0.986$ ) for the pure quadratic model is identical to that of the quadratic model. This suggests that the pure quadratic model explains the same proportion of variance in thermal conductivity ( $\lambda$ ) as the quadratic model. Adjusted  $R^2$  of ( $R^2_{Adj} = 0.972$ ) is slightly higher than that of the quadratic model ( $R^2_{Adj} = 0.964$ ), suggesting that after adjusting for the number of predictors, the pure quadratic model explains a slightly better portion of the variance in the data. The F-statistic ( $F = 69.500$ ) indicates that the pure quadratic model provides a significantly better fit than the constant model. This value is higher than the quadratic model's F-statistic ( $F = 43.70$ ), suggesting that the pure quadratic model might be a better fit. The p-value ( $p\text{-value} = 5.98E-04$ ) is very small, indicating that the pure quadratic model is highly statistically significant and provides a strong relationship between the predictors and thermal conductivity.

In the linear model, the linear model is very effective, with an  $R^2$  ( $R^2 = 0.972$ ) and a low Root Mean Squared Error (RSME = 0.015). It has a statistically significant p-value ( $p\text{-value} = 2.27E-05$ ) and explains a large portion of the variance in thermal conductivity. In addition, the quadratic model, the quadratic model improves slightly over the linear model, with an  $R^2$  ( $R^2 = 0.986$ ) and a lower Root Mean Squared Error ( $RMSE = 0.014$ ). It is statistically significant p-value ( $p\text{-value} = 5.28E-03$ ) and provides a better fit than the linear model, though the improvement is modest. In addition, the pure quadratic model has the lowest Root Mean Squared Error ( $RMSE = 0.013$ ) and an identical  $R^2$  ( $R^2 = 0.986$ ) to the quadratic

model. It has the highest adjusted  $R^2$  ( $R^2_{Adj} = 0.972$ ) and a higher F-statistic ( $F = 69.50$ ), suggesting that it is a better fit than the quadratic model. The p-value (p-value =  $5.98E-04$ ) indicates that it is highly statistically significant.

Ultimately, the pure quadratic model offers the best fit for thermal conductivity, with the lowest Root Mean Squared Error (RMSE), the highest adjusted  $R^2$ , and a significantly higher F-statistic compared to the other models. Although the quadratic model is almost as effective, the linear model is still a strong contender, with very high  $R^2$  and low Root Mean Squared Error (RMSE), making it a viable model for predicting thermal conductivity. However, the pure quadratic model should be preferred for its better predictive accuracy and statistical significance.

### VI.3.3. Analysis of Variance (ANOVA) Results

**Table VI.11.** Shown the Sum of Squares (SumSq), degrees of freedom (DF), Mean squares (MeanSq), F-statistic (F), and p-Value (p-value) for three models linear, quadratic, and pure quadratic to assess the significance of the predictor's Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) in modeling Charpy impact strength (G).



Table VI.11. ANOVA Summary for Model Comparison in Thermal Conductivity Prediction.

| Model          | -         | Sum of Squares<br>(SumSq) | Degrees of Freedom<br>(DF) | The Mean Squares<br>(MeanSq) | The F-statistic<br>(F)        | p-Value<br>(p-value)             |
|----------------|-----------|---------------------------|----------------------------|------------------------------|-------------------------------|----------------------------------|
| Linear         | Total     | 0.0479                    | 8                          | 0.0060                       | 102.8800                      | 2.27E-05                         |
|                | Model     | 0.0465                    | 2                          | 0.0233                       |                               |                                  |
|                | Residual  | 0.0014                    | 6                          | 0.0002                       |                               |                                  |
| Quadratic      | Total     | 0.0479                    | 8                          | 0.0060                       | 43.7480<br>107.7300<br>1.0943 | 5.28E-03<br>1.61E-03<br>4.71E-01 |
|                | Model     | 0.0472                    | 5                          | 0.0094                       |                               |                                  |
|                | Linear    | 0.0465                    | 2                          | 0.0233                       |                               |                                  |
|                | Nonlinear | 0.0007                    | 3                          | 0.0002                       |                               |                                  |
|                | Residual  | 0.0006                    | 3                          | 0.0002                       |                               |                                  |
| Pure Quadratic | Total     | 0.0479                    | 8                          | 0.0060                       | 69.5220<br>137.0500<br>1.9964 | 5.98E-04<br>2.07E-04<br>2.50E-01 |
|                | Model     | 0.0472                    | 4                          | 0.0118                       |                               |                                  |
|                | Linear    | 0.0465                    | 2                          | 0.0233                       |                               |                                  |
|                | Nonlinear | 0.0007                    | 2                          | 0.0003                       |                               |                                  |
|                | Residual  | 0.0007                    | 4                          | 0.0002                       |                               |                                  |

The results were as follows:

- 1. Linear Model:** Total Sum of Squares (SumSq = 0.0479), representing the total variability in the thermal conductivity ( $\lambda$ ) data. Model Sum of Squares (SumSq = 0.0465), indicating that the linear model explains a significant portion of the variation (Approximately 97% of the Total Variance). residual Sum of Squares (SumSq = 0.0014), showing a small amount of unexplained variability, which means the model fits the data very well. The model mean square is (MeanSq = 0.0233), and the residual mean square is (MeanSq = 0.0002). F-statistic (F = 102.8800), a very high F-statistic, indicating that the model explains a large proportion of the variance compared to the residual error. The p-value (p-value = 2.27E-05), which is significantly lower than 0.05, meaning that the linear model is statistically significant and strongly explains thermal conductivity.
- 2. Quadratic Model:** Total Sum of Squares (SumSq = 0.0479), the same as the linear model, representing total variability. Model Sum of Squares (SumSq = 0.0472), showing that the

quadratic model explains slightly more of the variation than the linear model (Approximately 98.5% of the Total Variance). Residual Sum of Squares (SumSq = 0.0006), which is even smaller than the residual Sum of Squares for the linear model, indicating better fit. The model mean square is (MeanSq = 0.0094), and the residual mean square is (MeanSq = 0.0002). F-statistic ( $F = 43.7480$ ), which is high but lower than the linear model's F-statistic ( $F = 102.8800$ ). This suggests that the quadratic model explains a significant proportion of variance but not as effectively as the linear model. The p-value (p-value =  $5.28E-03$ ), which is less than 0.05, indicating that the quadratic model is statistically significant.

- 3. Pure Quadratic Model:** Total Sum of Squares (SumSq = 0.0479), consistent across models. Model Sum of Squares (SumSq = 0.0472), which is similar to the quadratic model, showing that the pure quadratic model explains nearly all the variance (98.5% of Total Variance). Residual Sum of Squares (SumSq = 0.0007), which is slightly higher than the quadratic model's residuals (SumSq = 0.0006), indicating slightly more unexplained variance. The model mean square is (MeanSq = 0.0118), and the residual mean square is (MeanSq = 0.0002). F-statistic ( $F = 69.5220$ ), which is higher than the quadratic model's F-statistic ( $F = 43.7480$ ), suggesting that the pure quadratic model may be a better fit than the quadratic model. The p-value (p-value =  $5.98E-04$ ), which is less than 0.05, indicating that the pure quadratic model is statistically significant.

In linear model, explains the majority of the variance in thermal conductivity ( $\lambda$ ) (Approximately 97%), with a very high F-statistic ( $F = 102.8800$ ) and a statistically significant p-value (p-value =  $2.27E-05$ ). In addition, quadratic model, explains slightly more variance (98.5%) than the linear model but with a lower F-statistic ( $F = 43.7480$ ), indicating that the quadratic model does not improve much over the linear model. The model is still statistically significant (p-value = 0.0053). In addition, pure quadratic model, explains 98.5% of the variance and has a higher F-statistic ( $F = 69.5220$ ) compared to the quadratic model. It is statistically significant (p-value =  $5.98E-04$ ), but the improvement over the quadratic model is minimal.

Ultimately, the linear model offers the best fit for thermal conductivity, with the highest F-statistic and significance. However, the pure quadratic model shows a slightly higher F-statistic and offers a marginal improvement in fitting the data. Therefore, the linear model is the most effective, though the pure quadratic model may be useful for capturing slight nonlinearities in the data.

### VI.3.4. The Predicted Results

**Table VI.12.** Shown is the performance of bio-composite materials under various conditions defined by the size of the Date Palm Fiber (DPF) ( $x_1$ ) and mass ratio ( $x_2$ ). The bio-composite materials were tested to determine their thermal conductivity ( $\lambda$ ) properties, measured in percentage (W/m.k). Three different bio-composite materials were tested (GF.I.m, GF.II.m, and GF.III.m), with each having, variations in both Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ), to find three predictive results models (Linear, Quadratic, and Pure-Quadratic).

**Table VI.12. Predicted and Measured Thermal Conductivity for Various Fiber Sizes and Mass Ratios.**

| Bio-composite Material | Test | $x_1$ (Size) | $x_2$ (Mass Ratio) | Measured [W/m.k]  | Linear [W/m.k] | Quadratic [W/m.k] | Pure-Quadratic [W/m.k] |
|------------------------|------|--------------|--------------------|-------------------|----------------|-------------------|------------------------|
| GF.I.05                | 1    | 0.080        | 05                 | $0.521 \pm 0.002$ | 0.515          | 0.520             | 0.517                  |
| GF.I.10                | 2    | 0.080        | 10                 | $0.436 \pm 0.003$ | 0.446          | 0.433             | 0.433                  |
| GF.I.15                | 3    | 0.080        | 15                 | $0.373 \pm 0.002$ | 0.377          | 0.377             | 0.379                  |
| GF.II.05               | 4    | 0.315        | 05                 | $0.455 \pm 0.002$ | 0.454          | 0.466             | 0.466                  |
| GF.II.10               | 5    | 0.315        | 10                 | $0.394 \pm 0.002$ | 0.385          | 0.382             | 0.382                  |
| GF.II.15               | 6    | 0.315        | 15                 | $0.327 \pm 0.002$ | 0.316          | 0.328             | 0.328                  |
| GF.III.05              | 7    | 0.500        | 05                 | $0.415 \pm 0.002$ | 0.406          | 0.405             | 0.407                  |
| GF.III.10              | 8    | 0.500        | 10                 | $0.308 \pm 0.003$ | 0.337          | 0.323             | 0.323                  |
| GF.III.15              | 9    | 0.500        | 15                 | $0.277 \pm 0.003$ | 0.268          | 0.272             | 0.269                  |

**Table VI.12.** Shown the thermal conductivity, measured in watts per meter-kelvin (W/m·K), showed a consistent decrease as the mass ratio increased across all bio-composite materials. For instance, in the bio-composite material (GF.I) series, with a Date Palm Fiber (DPF) size of ( $x_1 = 0.080$ ), thermal conductivity ( $\lambda$ ) decreased from bio-composite material (GF.I.05) ( $0.521 \pm 0.002$  W/m·K) at a ( $x_2 = 05$ ) mass ratio to bio-composite material (GF.I.15) ( $0.373 \pm 0.002$  W/m·K) at a ( $x_2 = 15$ ) mass ratio. The bio-composite material (GF.II) series, with a Date Palm Fiber (DPF) of ( $x_1 = 0.315$ ), exhibited similar trends, with thermal conductivity ( $\lambda$ ) values ranging from bio-composite material (GF.II.05) ( $0.455 \pm 0.002$  W/m·K) at ( $x_2 = 05$ ) to the bio-composite material (GF.II.15) ( $0.327 \pm 0.002$  W/m·K) at ( $x_2 = 15$ ). The bio-composite material (GF.III) series, with the larger Date Palm Fiber (DPF) ( $x_1 = 0.500$ ), showed the lowest thermal conductivity ( $\lambda$ ) values, with bio-composite material (GF.III.05) ( $0.415 \pm 0.002$  W/m·K) at ( $x_1 = 05$ ) and decreasing to bio-composite material (GF.III.15) ( $0.277 \pm 0.003$  W/m·K) at ( $x_2 = 15$ ). The comparison between the measured thermal conductivity ( $\lambda$ )

values and those predicted by linear, quadratic, and pure-quadratic models showed good agreement, suggesting that the relationship between Date Palm Fiber (DPF) size ( $x_1$ ), mass ratio ( $x_2$ ), and thermal conductivity ( $\lambda$ ) is well-represented by quadratic models.

Ultimately, these findings highlight the influence of both date palm size and mass ratio on the thermal conductivity ( $\lambda$ ) of bio-composites, which could guide the development of materials with tailored thermal properties for specific applications.

**Figure VI.03.** Shown are the thermal conductivity ( $\lambda$ ) values (Measured in W/m.k) for various test conditions, including Date Palm Fiber (DPF) size ( $x_1$ ), mass ratio ( $x_2$ ), and three models (Linear, Quadratic, and Pure-Quadratic) results for prediction.

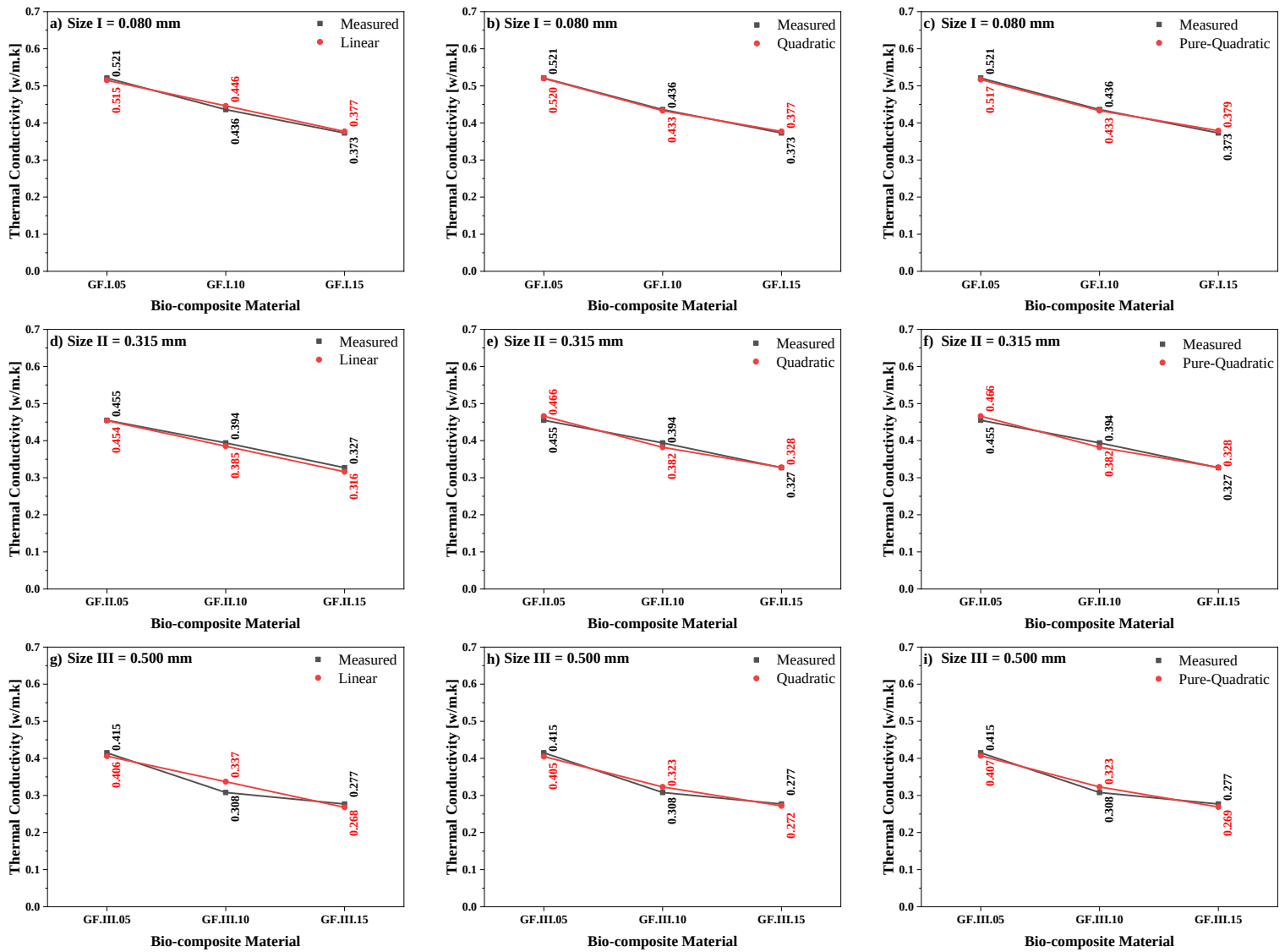


**Figure VI.03. Thermal Conductivity Measurements versus Prediction Models: a, d, and g) Linear; b, e, and h) Quadratic; and c, f, and i) Pure-Quadratic.**

**Figure VI.03.** Shown the bio-composite material (GF.I.05), all models provide very close predictions to the measured value. The quadratic and pure-quadratic models are slightly more accurate than the linear model, which slightly underestimates the value. The bio-composite material (GF.I.10), the quadratic and pure-quadratic models are very close to the measured value, with the linear model slightly overestimating the thermal conductivity. The bio-composite material (GF.I.15), the linear and quadratic models provide almost identical predictions, with the pure-quadratic model slightly overestimating the thermal conductivity. The bio-composite material (GF.II.05), the linear model is very close to the measured value, while the quadratic and pure-quadratic models slightly overestimate the thermal conductivity. The bio-composite material (GF.II.10), all models slightly underestimate the measured value, with the linear

model being closest to the measured thermal conductivity. The bio-composite material (GF.II.15), the quadratic and pure-quadratic models provide the most accurate predictions, closely matching the measured value, while the linear model slightly underestimates the thermal conductivity. The bio-composite material (GF.III.05), all models are fairly close to the measured value, with the pure-quadratic model being slightly more accurate than the other two models. The bio-composite material (GF.III.10), both the quadratic and pure-quadratic models provide predictions closer to the measured value, with the linear model overestimating the thermal conductivity. The bio-composite material (GF.III.15), the quadratic model gives the closest prediction, while the linear model slightly underestimates the thermal conductivity. The pure-quadratic model also provides a close prediction.

Ultimately, the quadratic and pure-quadratic models generally provide the most accurate predictions of thermal conductivity ( $\lambda$ ) across the tests, with the pure-quadratic model often being slightly more accurate. The linear model tends to either overestimate or underestimate the thermal conductivity, particularly at higher mass ratios. Overall, the quadratic and pure-quadratic models are more reliable than the linear model for predicting thermal conductivity, especially for larger sizes and mass ratios.

## VI.4. Multi-Objective Optimization Results

The Multi-Objective Optimization (MOO) results were as follows:

### VI.4.1. Optimization Objective

In optimization objectives, a Multi-Objective Optimization (MOO) problem was formulated to achieve three goals, as shown in Equation (VI.01):

$$\left\{ \begin{array}{l} \text{Min} \quad (-G), \text{WAR}, \lambda, \\ \text{Where } 0.08 \leq x_1 \leq 0.5 \\ \quad \quad 0.05 \leq x_2 \leq 0.15 \\ \text{Subject} \quad f_m \geq \overline{f_m} \end{array} \right. \quad (\text{VI.01})$$

Where:

- **G**: The Charpy Impact Strength [ $\text{KJ/m}^2$ ];
- **WAR**: The Water Absorption [%];
- **$\lambda$** : The Thermal Conductivity [ $\text{w/m.k}$ ];

- $x_1$ : The Date Palm Fiber (DPF) Size (Decision Variable) [mm];
- $x_2$ : The Fiber Mass Ratio (Decision Variable) [%];
- $f_m$ : The Gypsum Compressive Strength (Constraint) [MPa];
- $\bar{f}_m$ : The Average Compressive Strength of Gypsum [MPa].

Equation (VI.01) represented the minimize thermal conductivity ( $\lambda$ ), the minimum water absorption (WAR), and the maximum Charpy impact strength (G).

The optimization variables considered in the study were the Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ). The decision variables in Equations (VI.02 and 03), Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ), were constrained within the following bounds:

$$0.08 \leq x_1 \leq 0.50 \quad (\text{VI.02})$$

$$0.05 \leq x_2 \leq 0.15 \quad (\text{VI.03})$$

Additionally, a constraint was added for the gypsum compressive strength, as shown in Equation (VI.04), ensuring that the optimized bio-composite material had adequate mechanical strength.

$$f_m \geq \bar{f}_m = 6.998 \text{ MPa; Gypsum Compressive Strength} \quad (\text{VI.04})$$

Where:

- $f_m$ : The Gypsum Compressive Strength (Constraint) [MPa];
- $\bar{f}_m$ : The Average Compressive Strength of Gypsum [MPa].

#### VI.4.2. Pareto Optimal Solutions

**Figure VI.04.** Shown 2052 Pareto optimal solutions resulting from the Multi-Objective Optimization (MOO) problem. These solutions represent trade-offs between the three objectives and are crucial for identifying the best possible compromises in the bio-composite material design.

The Pareto front is a set of solutions where no objective can be improved without worsening another objective. This means that the solutions along this front are efficient and cannot be dominated by any other solution.

**Figure VI.04.** Shown 3D scatter plot that visually represents the relationship between three key properties of the bio-composite material: water absorption, thermal conductivity, and Charpy impact strength (G). These properties are influenced by the decision variables, Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ). These data points of the optimization study, where:

- 1. Water Absorption [%] (x-axis):** water absorption (WAR) is a crucial property for bio-composite materials used in environments exposed to moisture. A higher water absorption (WAR) percentage often correlates with poorer mechanical properties in bio-composite materials, such as reduced impact strength.
- 2. Thermal Conductivity [W/m·K] (y-axis):** Thermal conductivity ( $\lambda$ ) reflects the bio-composite material's ability to conduct heat. A lower thermal conductivity ( $\lambda$ ) is typically desired in bio-composite materials used for insulation or temperature-sensitive applications.
- 3. Impact Strength [kJ/m<sup>2</sup>] (z-axis):** Impact strength is a measure of the bio-composite material's resistance to sudden forces or impacts. Higher impact strength is often desirable for bio-composite materials in applications requiring durability and resistance to mechanical stress.

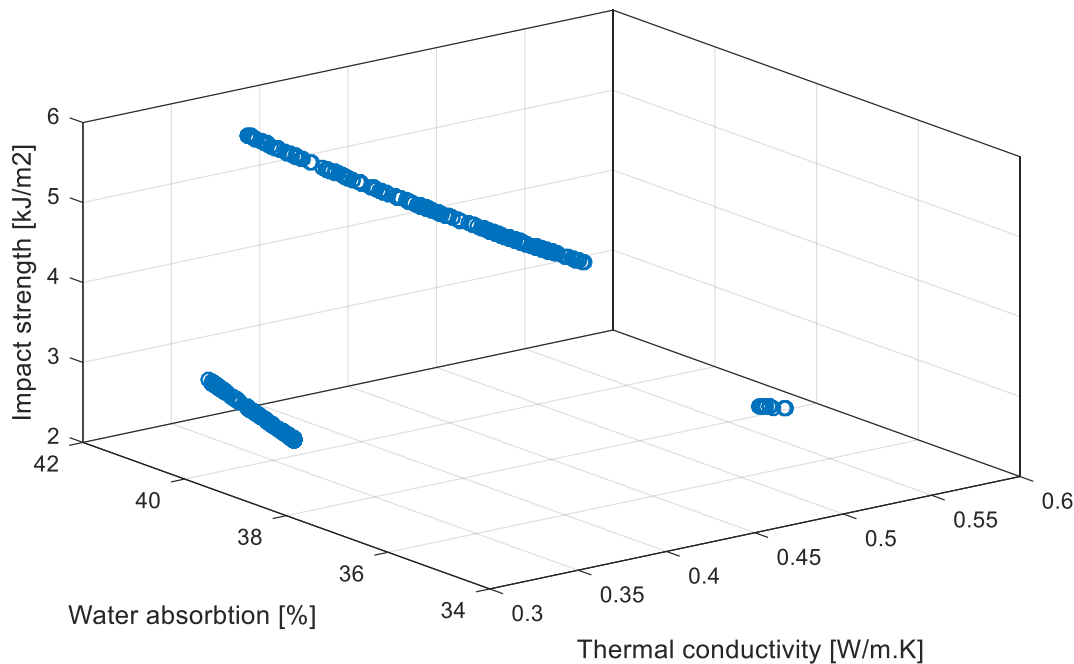


**Figure VI.04. The Obtained Pareto Optimal Solutions.**



Each data point reflects a specific configuration of Date Palm Fiber (DPF) size (0.080, 0.315, and 0.500 mm) and mass ratio (05, 10, and 15%), which results in varying values for these three bio-composite materials properties (Water Absorption, Thermal Conductivity, and Charpy Impact Strength (G)).

A cluster of data points appears to be situated at the lower thermal conductivity ( $\lambda$ ) and water absorption (WAR) values, with high impact strength. This suggests that certain combinations of Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) lead to a more favorable balance of these properties. These combinations likely represent the Pareto-optimal solutions, where the best trade-offs are found.

As the water absorption (WAR) increases (Moving Towards the Right Along the X-axis), the impact strength tends to decrease. This indicates an inverse relationship between water absorption (WAR) and impact strength, a typical trend in the bio-composite materials. Higher water absorption (WAR) may reduce the bio-composite material's mechanical integrity, which in turn reduces its resistance to impact. A similar trend is observed between thermal conductivity ( $\lambda$ ) and impact strength. Higher thermal conductivity ( $\lambda$ ) seems to be associated with lower impact strength, implying that bio-composite materials with better insulating properties may be less resistant to mechanical stress, possibly due to lower structural integrity.

**Figure VI.04.** Highlights the trade-offs between minimizing thermal conductivity ( $\lambda$ ) and water absorption, while maximizing impact strength. In a Multi-Objective Optimization (MOO) problem, the goal is to find the Pareto front, which represents the set of optimal solutions where no objective can be improved without worsening another. The plot likely contains these Pareto-optimal points, helping to identify the most balanced combinations of the decision variables (Date Palm Fiber (DPF) Size ( $x_1$ ), and Mass Ratio ( $x_2$ )) that result in desirable bio-composite material properties.

Ultimately, this scatter plot provides valuable insights into how Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) influence the mechanical and thermal properties of the bio-composite material. The observed trends can inform the design of bio-composite materials with optimized performance characteristics based on specific application needs. For example, suppose a bio-composite material with high impact strength and low thermal conductivity ( $\lambda$ ) is required. In that case, the plot can guide the selection of Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) to achieve this optimal performance. The data may also serve as a foundation for further refinement of the optimization process, incorporating more complex modeling or additional constraints to enhance bio-composite material design.

### VI.4.3. Limiting Solutions

**Table VI.13.** Shown the results of a Multi-Objective Optimization (MOO) problem, where the goal is to optimize three bio-composite material properties: impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ). The **Table VI.13.** Shows the values of the decision variables (Date Palm Fiber (DPF) Size ( $x_1$ ) and Mass Ratio ( $x_2$ )) for three different optimization strategies:

**Table VI.13. The Results of a Multi-Objective Optimization (MOO) Problem.**

| Solution                | Variables |        | Objectives        |          |                        |
|-------------------------|-----------|--------|-------------------|----------|------------------------|
|                         | $x_1$     | $x_2$  | $\lambda$ [W/m.K] | WAR [%]  | G [KJ/m <sup>2</sup> ] |
| Maximize Only G         | 0.315     | 0.1132 | 0.36468           | 41.02398 | 5.76569                |
| Minimize Only WAR       | 0.125     | 0.0502 | 0.51144           | 35.51649 | 2.92247                |
| Minimize Only $\lambda$ | 0.500     | 0.0835 | 0.34659           | 41.14225 | 2.75429                |
| Best Compromise         | 0.315     | 0.1132 | 0.36468           | 41.02398 | 5.76569                |

To better understand the optimization results, three limiting solutions were identified:

- 1. Maximizing Impact Strength (G):** This solution focuses on achieving the highest impact strength possible while adjusting the thermal conductivity ( $\lambda$ ) and water absorption (WAR). The corresponding solution variables are:
  - a. **Fiber Size:**  $x_1 = 0.315$ ;
  - b. **Fiber Mass Ratio:**  $x_2 = 0.1132$ ;
  - c. **Thermal Conductivity:**  $\lambda = 0.36468$  W/m.k;
  - d. **Water Absorption:** WAR = 41.02398 %;
  - e. **Impact Strength:** G = 5.76569 kJ/m<sup>2</sup>.

Ultimately, this solution provides the highest impact strength, while both thermal conductivity ( $\lambda$ ) and water absorption (WAR) are relatively balanced. However, this solution still presents a compromise where water absorption (WAR) and thermal conductivity ( $\lambda$ ) are not minimized to their fullest potential.

- 2. Minimizing Water Absorption (WAR):** The goal here is to minimize water absorption (WAR) while adjusting the other objectives accordingly. The corresponding solution variables are:

- a. **Fiber Size:**  $x_1 = 0.125$ ;
- b. **Fiber Mass Ratio:**  $x_2 = 0.0502$ ;
- c. **Thermal Conductivity:**  $\lambda = 0.51144$  W/m.k;
- d. **Water Absorption:** WAR = 35.51649 %;
- e. **Impact Strength:**  $G = 2.92247$  kJ/m<sup>2</sup>.

Ultimately, although water absorption (WAR) is minimized, this solution results in a relatively higher thermal conductivity ( $\lambda$ ) and lower impact strength, indicating a compromise in bio-composite material performance.

**3. Minimizing Thermal Conductivity ( $\lambda$ ):** This solution focuses on minimizing thermal conductivity ( $\lambda$ ) while allowing the other objectives to vary. The corresponding solution variables are:

- a. **Fiber Size:**  $x_1 = 0.500$ ;
- b. **Fiber Mass Ratio:**  $x_2 = 0.0835$ ;
- c. **Thermal Conductivity:**  $\lambda = 0.34659$  W/m.k;
- d. **Water Absorption:** WAR = 41.14225 %;
- e. **Impact Strength:**  $G = 2.75429$  kJ/m<sup>2</sup>.

Ultimately, in this case, the thermal conductivity ( $\lambda$ ) is minimized, but both water absorption (WAR) and impact strength are less optimized. This reflects the trade-off between these properties, where minimizing thermal conductivity ( $\lambda$ ) sacrifices impact strength and increases water absorption.

In conclusion, this solution represents a balanced approach where the three objectives are considered together, resulting in an optimal combination of  $x_1$ ,  $x_2$ ,  $G$ , WAR, and  $\lambda$ .

#### VI.4.4. Best Compromise Solution

The best compromise solution is identified as:

- a. **Fiber Size:**  $x_1 = 0.315$ ;
- b. **Fiber Mass Ratio:**  $x_2 = 0.1132$ ;
- c. **Thermal Conductivity:**  $\lambda = 0.36468$  W/m.k;
- d. **Water Absorption:** WAR = 41.02398 %;

e. **Impact Strength:**  $G = 5.76569 \text{ kJ/m}^2$ .

Ultimately, this solution represents an optimal trade-off between the three objectives, balancing all of them to provide a material with high impact strength, relatively low thermal conductivity, and acceptable water absorption.

## Conclusion

In conclusion, this sixth chapter presents a comprehensive analysis of the influence of Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) on the performance of bio-composite materials, specifically focusing on Charpy impact strength ( $G$ ), water absorption (WAR), and thermal conductivity ( $\lambda$ ). The results indicate that varying the Date Palm Fiber (DPF) size ( $x_1$ ) and fiber mass ratio ( $x_2$ ) significantly impacts the bio-composite material's thermal and mechanical properties. While the linear and quadratic models provided valuable insights into the relationships between these variables, the Pareto front analysis offered a robust framework for optimizing the design of bio-composite materials with superior performance characteristics. The best compromise solution, which achieved an optimal balance between the desired objectives, offers promising potential for the development of advanced bio-composite materials that can meet the specific requirements of various industrial applications.

Through the application of Multi-Objective Optimization (MOO) techniques, a set of Pareto-optimal solutions was identified, balancing the conflicting objectives of minimizing thermal conductivity ( $\lambda$ ) and water absorption (WAR) while maximizing Charpy impact strength ( $G$ ).

The Multi-Objective Optimization (MOO) approach has highlighted the challenges of balancing thermal conductivity, water absorption, and impact strength in the design of bio-composite materials. The best compromise solution provides a viable approach for achieving a material with optimized properties for practical applications, particularly when high impact strength is desired. However, further exploration of additional factors or alternative optimization strategies could lead to improvements in achieving even better balances among the three objectives.

- 1. Impact Strength (G):** The solution that maximizes impact strength shows that this property can be optimized, but at the expense of both water absorption (WAR) and thermal conductivity. The results suggest that achieving an optimal balance in impact strength, while also controlling thermal conductivity ( $\lambda$ ) and water absorption, requires more intricate adjustments of the Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ).

2. **Water Absorption (WAR):** Similarly, minimizing water absorption (WAR) often leads to higher thermal conductivity, indicating that reducing the material's water absorption (WAR) capacity is inversely related to its thermal properties. Thus, solutions that minimize one property often result in poorer performance for the other properties.
3. **Thermal Conductivity ( $\lambda$ ):** The optimization results show that there is a clear trade-off between minimizing thermal conductivity ( $\lambda$ ) and improving impact strength or reducing water absorption. The highest impact strength solutions tend to result in higher thermal conductivity ( $\lambda$ ) values, suggesting a balance is necessary to maintain material performance across multiple properties.

Ultimately, overall, the findings of this research contribute to the broader understanding of how bio-composite material properties can be engineered through Date Palm Fiber (DPF) reinforcement in bio-composite material, and they provide a foundation for future studies aimed at further improving the performance of sustainable bio-composite materials. The use of Multi-Objective Optimization (MOO) proves to be a valuable tool in guiding the design and manufacturing of materials with tailored properties, which are crucial for meeting the evolving demands of modern engineering applications.

## Conclusion

In conclusion, the third part of this thesis, which encompasses the fifth and sixth chapters, provides a thorough exploration of the methodologies and analyses used to evaluate the performance of bio-composite materials and the relationships between key variables.

The fifth chapter has systematically outlined the process of data collection, regression modeling, and statistical evaluation. Various regression models, including linear, quadratic and pure quadratic approaches, have been employed to assess the relationships between the variables, while Multi-Objective Optimization (MOO) techniques were utilized to evaluate trade-offs and identify optimal solutions for real-world scenarios. The use of statistical measures such as t-statistics, p-values, and ANOVA, along with the application of MATLAB, has enhanced the robustness of the findings and ensured the reproducibility of the methodology.

The sixth chapter, building upon this analytical foundation, has provided an in-depth examination of the influence of Date Palm Fiber (DPF) size ( $x_1$ ) and mass ratio ( $x_2$ ) on bio-composite material properties. The results highlight the significant impact of these variables on Charpy impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ) of the bio-composite materials. The chapter's focus on Multi-Objective Optimization (MOO) has further emphasized the challenges and trade-offs involved in designing bio-composites with optimized performance across multiple properties. By identifying the best compromise solution, this chapter has laid the groundwork for the development of advanced bio-composite materials tailored to meet specific industrial requirements.

Together, the methodologies and findings of these two chapters offer valuable insights into the performance optimization of bio-composite materials. The application of regression models and Multi-Objective Optimization (MOO) not only enhances the understanding of material behavior but also provides a framework for future studies and practical applications in sustainable material design.

Ultimately, the third part of these findings contributes to the broader field of materials engineering, with the potential to advance the development of bio-composites that balance performance, sustainability, and cost-effectiveness.

## **General Conclusion**

## **General Conclusion**

This doctoral thesis has presented a comprehensive exploration of bio-composite materials, specifically focusing on the use of Date Palm Fibers (DPFs) and gypsum in creating sustainable and high-performance bio-composites. Through an in-depth investigation across multiple chapters, this thesis has examined the potential and challenges of integrating natural fibers into bio-composite systems, assessing both the materials' mechanical, physical, thermal, chemical, and morphological properties.

The first chapter laid the foundational understanding of composite materials, highlighting the role of bio-composites in advancing sustainable material science. Emphasis was placed on the potential of natural fibers, such as Date Palm Fibers (DPFs), to serve as effective reinforcements in composite formulations. The benefits of using these fibers, including their biodegradability, cost-effectiveness, and environmental sustainability, were contrasted with challenges related to their processing and durability. These discussions set the stage for the following chapters, which explored specific applications and performance assessments.

In the second chapter, the integration of Date Palm Fibers (DPFs) and gypsum was explored in detail. The favorable mechanical properties of Date Palm Fibers (DPFs) and the versatile applications of gypsum were showcased, along with the various methods of fiber extraction and processing. This second chapter identified key gaps in existing research, opening the door for future studies aimed at enhancing the performance and processing techniques of these bio-composite materials.

The third chapter focused on the preparation and evaluation of bio-composite materials (GF.s.m), discussing the impact of varying fiber mass ratios (m: 05-20%) and fiber sizes (s: I-III) on the bio-composite material's properties. A systematic approach was employed to assess the bio-composites through rigorous mechanical, physical, thermal, chemical, and morphological testing. These tests provided valuable insights into the performance of bio-composites, especially for construction and insulation applications, highlighting their strengths and weaknesses across different environmental conditions.

In the fourth chapter, the results of the bio-composite materials' (GF.s.m) performance were analyzed in-depth, revealing significant variations in mechanical properties, bulk density, water absorption,



## General Conclusion

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thickness swelling, moisture content, and thermal conductivity ( $\lambda$ ) based on the fiber size (s: I-III) and mass ratio (m: 05-20%). These findings highlighted the importance of balancing the mechanical, physical, and thermal properties of the bio-composites (GF.s.m) for optimal performance in real-world applications. Despite challenges, such as lower mechanical strength compared to gypsum, bio-composites (GF.s.m) with Date Palm Fibers (DPFs) proved to be promising candidates for energy-efficient and sustainable construction.

The fifth chapter provided a robust statistical framework, employing linear, quadratic, and pure quadratic regression models and Multi-Objective Optimization (MOO) to analyze the relationships between variables and their influence on the bio-composite materials' (GF.s.m) performance. In addition, the use of Multi-Objective Optimization (MOO) techniques helped identify optimal solutions for balancing conflicting material properties, providing a valuable tool for future material design and development.

The sixth and final chapter demonstrated the practical application of a robust statistical framework, employing linear, quadratic, and pure quadratic regression models and Multi-Objective Optimization (MOO) in fine-tuning the properties of bio-composite materials (GF.s.m). The optimization results revealed trade-offs between impact strength (G), water absorption (WAR), and thermal conductivity ( $\lambda$ ), offering insights into how to balance these properties for specific industrial applications. This fifth chapter not only enhanced the understanding of bio-composite material (GF.s.m) design but also provided a foundation for future research aimed at further improving the bio-composite materials' (GF.s.m) properties.

Overall, this thesis has made significant contributions to the field of bio-composite materials (GF.s.m), particularly in the context of utilizing Date Palm Fibers (DPFs) and gypsum. It has provided a thorough evaluation of their properties, manufacturing techniques, and potential applications. The findings of this thesis offer valuable insights for developing sustainable bio-composite materials (GF.s.m) with tailored properties for a wide range of industrial applications, especially in the construction and manufacturing sectors.

Ultimately, this thesis also lays the groundwork for future research on enhancing the durability, performance, and scalability of bio-composite materials (GF.s.m), addressing existing challenges, and paving the way for more eco-friendly solutions in material science.

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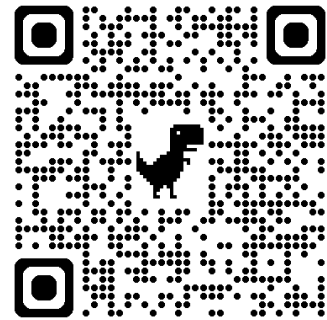
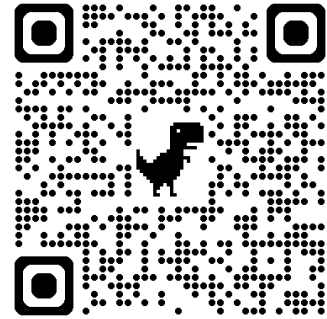
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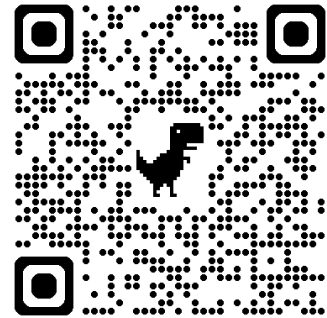
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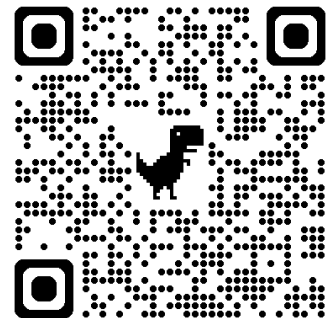
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## 1. International Scientific Articles

- **Abderrahmane SAHRAOUI**, Tarek DJOUDI, Mabrouk HECINI (2024), Investigating the Impact of Date Palm Fiber Waste on the Properties of a Novel Gypsum-Based Bio-Composite, Construction and Building Materials. 457, 139419. <https://doi.org/10.1016/j.conbuildmat.2024.139419>.

## 2. International and National Conferences

### a. International Conferences

- **Abderrahmane SAHRAOUI**, Mabrouk HECINI, Tarek DJOUDI, Wassila GRINE. Simulation of the Effect of the Length of Palm Fiber Reinforced on Two Different Composite Materials (Epoxy Resin / Palm Fibers and Polyester / Palm Fibers). Presented at The 1<sup>ST</sup> International Seminar on Chemical Process and Environment 1<sup>ST</sup> ISCPE 2022, Biskra, Algeria, March 14-16, 2023. <https://iscpe2022.univ-biskra.dz/html/iscpe2022.html>.
- **Abderrahmane SAHRAOUI**, Tarek DJOUDI, Mabrouk HECINI. Effect of Ratio of Natural Fibers on the Physical, Mechanical and Thermal Properties of a Bio-Composite Insulating Material (Date Palm Fibers/ Gypsum). Presented at The Second Arab Conference on Mechanics and Engineering, ARCME'23, Biskra, Algeria, December 10-12, 2023. <https://arcme.univ-biskra.dz/10-2/>.
- **Abderrahmane SAHRAOUI**, Tarek DJOUDI, Mabrouk HECINI, Zakaria LAKHAL. Mechanical and Thermal Performances of Gypsum Composite Materials Reinforced with Date Palm Tree Waste. Presented at The 8th International Conference on Mechanics and Energy, ICME'2023, Sousse, Tunisia, December 20-22, 2023. <https://icme.aicme.net>.

### b. National Conferences

- **Abderrahmane SAHRAOUI**, Lakhdar SEDIRA, Mabrouk HECINI, Tarek DJOUDI, Anfel LARIBI. The use of Digimat Software for the Modeling of the Creep Phenomena in Composite Materials with a Viscoelastic Behavior. Presented at The First National Conference on Mechanical Engineering, NCME'23, Batna, Algeria, May 10, 2023. <https://www.ncme23-univ-batna2.dz/>.
- **Abderrahmane SAHRAOUI**, Tarek DJOUDI, Mabrouk HECINI, Hossam Eddine BECHA. Effects of the Size of Date Palm Tree Fibers on the Physical, Mechanical and Thermal Properties of a New Bio-composite based on a Gypsum Matrix. Presented at The 1<sup>ST</sup> National Conference on Physics and it's Applications, Ncpa 2023, Bousaada, Algeria, December 02, 2023. <https://www.ens-bousaada.dz/ar/wp-content/uploads/2023/10/%D9%85%D9%84%D8%AA%D9%82%D9%89-%D9%88%D8%B7%D9%86%D9%8A1.pdf>.