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THESIS

In Candidacy for the Degree of
DOCTORATE 3rd CYCLE IN MECHANICAL ENGINEERING
Option: Materials Engineering

Development and characterization of polymeric matrix composite materials reinforced with lignocellulosic wastes

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Presented by :

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Abstract

This research investigates the potential of using date palm leaflet powder (DPLP) as a sustainable reinforcement for polyvinyl chloride (PVC)-based composites. Aiming to reduce environmental impact and promote bio-based alternatives, DPLP was subjected to various chemical treatments, including alkaline (NaOH), acetic acid, and silane coupling agent modifications, to enhance interfacial compatibility with the PVC matrix. The untreated (ULP), alkali-treated (ATLP), alkali–acetic acid treated (ACLP), and silane-treated (STLP) fillers were thoroughly characterized and incorporated at varying concentrations into the PVC matrix.

Fourier-transform infrared spectroscopy (FTIR) confirmed the chemical alterations on the fibre surfaces, especially the successful grafting of silane groups, which improved chemical bonding potential. Mechanical testing revealed that chemical treatments significantly enhanced the composite properties. The STLP-based composites exhibited the highest Young's modulus (221.08 MPa at 25% filler), tensile strength (10.6 MPa at 5% filler), and bending modulus, indicating improved fibre dispersion and interfacial adhesion. ATLP and ACLP composites also showed notable improvements, though slightly lower than STLP. In contrast, untreated composites demonstrated inferior mechanical behavior due to poor fibre–matrix bonding and high moisture absorption.

The findings confirm the effectiveness of surface modification in optimizing DPLP as a reinforcing agent in PVC composites. The silane-treated fibres, in particular, yielded the most balanced performance across mechanical and structural metrics. This study lays the groundwork for further exploration of DPLP-based composites in sustainable engineering applications, offering an eco-friendly alternative for materials used in construction, packaging, and automotive sectors.

Résumé

Cette recherche explore le potentiel de la poudre de folioles de palmier dattier (DPLP) en tant que renfort durable pour les composites à base de polychlorure de vinyle (PVC). Dans une optique de réduction de l'impact environnemental et de valorisation des alternatives biosourcées, la DPLP a été soumise à différents traitements chimiques, notamment un traitement alcalin (NaOH), à l'acide acétique, et à un agent de couplage silane, afin d'améliorer la compatibilité interfaciale avec la matrice PVC. Les charges non traitées (ULP), traitées à l'alcali (ATLP), traitées à l'alcali–acide acétique (ACLP) et traitées au silane (STLP) ont été caractérisées et incorporées dans la matrice à différentes concentrations.

La spectroscopie infrarouge à transformée de Fourier (FTIR) a confirmé les modifications chimiques des surfaces fibreuses, notamment la greffe réussie de groupes silanes qui favorise la liaison chimique. Les essais mécaniques ont montré une amélioration significative des propriétés des composites après traitement chimique. Les composites à base de STLP ont présenté les meilleures performances, avec un module de Young de 221,08 MPa (à 25 % de charge), une résistance à la traction de 10,6 MPa (à 5 %) et un module de flexion élevé, démontrant une meilleure dispersion des fibres et une excellente adhésion interfaciale. Les composites ATLP et ACLP ont également montré des améliorations notables. À l'inverse, les composites non traités présentaient un comportement mécanique médiocre, en raison d'une mauvaise interaction fibre-matrice et d'une absorption élevée de l'humidité.

Ces résultats confirment l'efficacité des traitements de surface pour optimiser l'utilisation de la DPLP comme agent de renfort dans les composites PVC. Les fibres traitées au silane offrent les meilleures performances globales, posant ainsi les bases du développement de matériaux durables pour les secteurs de la construction, de l'emballage et de l'automobile.

الملخص:

تستكشف هذه الدراسة إمكانية استخدام مسحوق فُويصلات سعف النخيل (DPLP) كمادة تدعيم مستدامة في المركبات القائمة على البولي فينيل كلوريد (PVC). وفي إطار السعي لتقليل الأثر البيئي وتعزيز البدائل الحيوية، خضعت الـ DPLP لمجموعة من المعالجات الكيميائية، بما في ذلك المعالجة القلوية (NaOH)، والمعالجة بحمض الأسيتيك، ومعالجة بعامل ربط سيلاني، بهدف تحسين التوافق بين الألياف والمصفوفة البلاستيكية. وقد تم توصيف الحشوات غير المعالجة (ULP)، والمعالجة بالقلوي (ATLP)، والمعالجة بالقلوي وحمض الأسيتيك (ACLP)، والمعالجة بالسيلان (STLP)، ودمجها في مصفوفة الـ PVC بنسب مختلفة.

أكدت أطياف الأشعة تحت الحمراء بتحويل فورييه (FTIR) التغيرات الكيميائية على أسطح الألياف، وخاصة تثبيت مجموعات السيلان بنجاح، مما يُعزز الروابط الكيميائية. وأظهرت الاختبارات الميكانيكية تحسناً ملحوظاً في خصائص المركبات بعد المعالجة الكيميائية، حيث سجلت المركبات المعتمدة على STLP أفضل أداء، مع معامل يونغ قدره 221.08 ميغاباسكال (عند 25% من الحشوة)، ومقاومة شد بلغت 10.6 ميغاباسكال (عند 5%)، ومعامل انحناء مرتفع، مما يدل على توزيع جيد للألياف والتصاق واجهي ممتاز. كما أظهرت المركبات المعتمدة على ATLP وACLP تحسناً كبيراً كذلك. في المقابل، أظهرت المركبات غير المعالجة أداءً ميكانيكياً ضعيفاً، نتيجة ضعف التفاعل بين الألياف والمصفوفة وارتفاع امتصاص الرطوبة.

تؤكد هذه النتائج فعالية المعالجات السطحية في تحسين استخدام DPLP كمادة تدعيم في مركبات الـ PVC. وقد أثبتت الألياف المعالجة بالسيلان أنها توفر أفضل أداء عام، مما يمهد الطريق لتطوير مواد مستدامة تُستخدم في قطاعات البناء والتغليف وصناعة السيارات.

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DEDICATION

To my Parents
Mom, Dad
My dear Sister
My Brothers

To my Dearest Wife

General Introduction

The global quest for sustainable materials and eco-friendly practices has intensified in recent decades, driven by an urgent need to address environmental concerns and minimize the ecological footprint of various industries. One such arena of exploration is the domain of composite materials, where the amalgamation of natural fibres with synthetic matrices has shown promise in achieving a delicate balance between performance, cost-effectiveness, and environmental sustainability. This doctoral thesis embarks on a journey into the uncharted territory of harnessing the inherent strength and eco-friendly attributes of date palm fibres as reinforcement in Polyvinyl Chloride (PVC) matrices.

The use of natural fibres in composite materials has gained traction due to their renewability, biodegradability, and often superior mechanical properties compared to traditional reinforcements. Date palm fibres, derived from the waste generated during date palm cultivation, represent an intriguing prospect for sustainable reinforcement. The choice of PVC as the matrix material is driven by its ubiquity, versatility and extensive applications, especially its use as the main material in the outer sheath of electrical cables. Investigating the compatibility of date palm fibres with PVC opens up new avenues for developing sustainable composite materials that can potentially replace conventional virgin PVC, contributing to a circular and environmentally responsible economy.

This research seeks to address critical gaps in our understanding of the synergy between date palm fibres and PVC matrices. The significance lies not only in the potential development of high-performance, sustainable composites but also in reducing reliance on non-renewable resources. By delving into the mechanical, thermal and morphological properties of these composites, this study aims to provide valuable insights that can inform material scientists, engineers, and industries seeking to adopt more environmentally friendly alternatives.

A comprehensive examination of the existing body of literature reveals a growing interest in natural fibre-reinforced composites. However, the exploration of date palm fibres as reinforcement in PVC matrices is relatively nascent. This thesis builds upon the existing knowledge base by synthesizing information from diverse sources, including studies on natural fibre composites, PVC matrix composites, and the unique characteristics of date palm fibres. By contextualizing the research within the broader scientific discourse, this study aims to contribute to the evolution of sustainable materials engineering.

The primary objectives of this doctoral thesis are threefold:

a) To evaluate the mechanical properties of date palm fibre-reinforced PVC composites, including tensile and flexural strength.
b) To investigate the thermal stability and behaviour of the composites through thermal analysis techniques.
c) To analyse the morphological characteristics and interfacial adhesion between date palm fibres and PVC matrices through microscopy and spectroscopy.

The methodology adopted for this study encompasses a multifaceted approach. Raw date palm fibres will be extracted, processed, and characterized for their physical and chemical properties. PVC composites will be prepared by varying percentages of date palm fibres to PVC matrix. Mechanical testing, thermal analysis, and morphological characterization will be conducted using established laboratory standards and techniques. The systematic approach employed in this research aims to provide robust and reliable data to draw meaningful conclusions.

This doctoral thesis is structured as follows:

I- Chapter I: Literature Review
II- Chapter II: Methodology, Materials and Apparatus;
III- Chapter III: Results and Discussion;
General Conclusion and Future Perspectives.

In conclusion, this work embarks on a pioneering journey into the uncharted territory of using date palm fibres as reinforcement in PVC matrices. Through rigorous experimentation and analysis, this research endeavours to contribute valuable knowledge to the scientific community, offering a sustainable solution to the growing demand for eco-friendly materials. The quest for innovation, sustainability, and a reduced environmental footprint underscores the essence of this study, providing a foundation for future advancements in the realm of composite materials.

CHAPTER I

LITERATURE REVIEW

I.1. Introduction

In recent decades, composite materials have become an integral part of modern engineering and material science due to their ability to offer customized combinations of mechanical, thermal, and chemical properties. This synergy allows composites to outperform conventional materials in a variety of applications, ranging from aerospace and automotive to construction and consumer products. Among the various matrix materials, polyvinyl chloride (PVC) is one of the most widely used thermoplastics. Known for its excellent chemical resistance, thermal stability, and low cost, PVC is extensively utilized in electrical cables, pipes, fittings, and various structural and semi-structural components. The increasing global focus on sustainability and waste reduction has driven interest in the use of natural fibers as reinforcements in polymer composites. Natural fibers are renewable, biodegradable, and generally require less energy to process compared to synthetic alternatives. In this context, agricultural by-products such as date palm (Phoenix dactylifera) leaflets have gained attention due to their widespread availability in arid and semi-arid regions, particularly in the Middle East and North Africa.

This chapter introduces the concept and application of composite materials and explores the role of PVC as a thermoplastic matrix in fiber-reinforced composites. It also presents a comprehensive review of recent research related to the extraction, treatment, and mechanical behavior of date palm leaflet fibers.

I.2. Composite materials

Composite materials are increasingly being used in the industry. Today, they hold a significant position among materials because they possess important advantages compared to traditional materials. They bring numerous functional benefits, including lightweight, mechanical and chemical resistance, reduced maintenance, and freedom of shapes. They contribute to extending the lifespan of certain equipment through their mechanical and chemical properties.

They enhance safety by better withstanding impacts and fire. They offer improved thermal or acoustic insulation, and for some, good electrical insulation. They also broaden design possibilities by allowing for the lightening of structures and the creation of complex forms capable of fulfilling multiple functions. Composite materials find applications in various fields such as transportation, sports and leisure, construction, and medicine. In each application domain, the remarkable performance of composite materials is the origin of innovative technological solutions. [2]

I.2.1. Definition:

A composite material is formed by the assembly of several immiscible materials of different natures, whose complementary qualities result in superior performance compared to each individual compound:[3]

- The matrix, responsible for bonding the assembly and distributing the efforts.

- The reinforcement, forming the structure of the piece and supporting the majority of the stresses.

I.2.2. Characteristics

Composite materials are engineered materials made from two or more constituent materials with significantly different physical or chemical properties. The combination of these materials creates a new material with enhanced performance and specific characteristics, Composite materials have many benefits over traditional materials, such as metals. One of the main benefits is their high strength-to-weight ratio, which means they can be much lighter and stronger than metals. This can improve the performance and efficiency of various applications, such as aircrafts and vehicles. Another benefit is their tailorable properties, which means engineers can customize them by changing the types, amounts, and orientations of the materials that make them up. This can optimize the material for specific applications, such as wind turbine blades and prosthetics. Composites can also have different properties in different directions, which are called anisotropy. This allows designers to adjust the material's performance based to specific loading conditions, such as tension and compression. Composites are also very durable and long-lasting, as they can resist fatigue and repeated loading cycles. This is advantageous in applications where materials are exposed to stress and corrosion. Composites are also suitable for applications where exposure to harsh environmental conditions is a concern, such as marine and oil and gas applications. Composites have low thermal conductivity, which can be useful for insulation or temperature control. This can be beneficial in applications where heat transfer or dissipation is important, such as electronics devises and fire-resistant materials. Composites are also flexible in design, as they can be moulded into complex shapes and create innovative designs. This can enhance the functionality of products. Some composites are also electrically insulating, which makes them suitable for electrical and electronic applications, such as circuit boards and sensors. Composites can also maintain their shape and size under different environmental conditions, such as temperature and humidity changes. This is called dimensional stability. This can improve the reliability and accuracy of the products, as well as reduce the need for maintenance and repair. Composites can be more expensive than traditional materials, but their superior performance can justify the higher cost. Composites can also resist impact, which makes them suitable for applications where they face sudden forces or impacts, such as armors and helmets.[4]

I.2.3. Applications in Various industries:

Composite materials find applications in a wide range of industries due to their unique combination of properties, and we find theme in: [5]

a) ***Aerospace Industry:***

Composites are extensively used in the aerospace sector for their high strength-to-weight ratio. Components such as aircraft fuselage, wings, and interior structures often incorporate composite materials to reduce weight and enhance fuel efficiency.

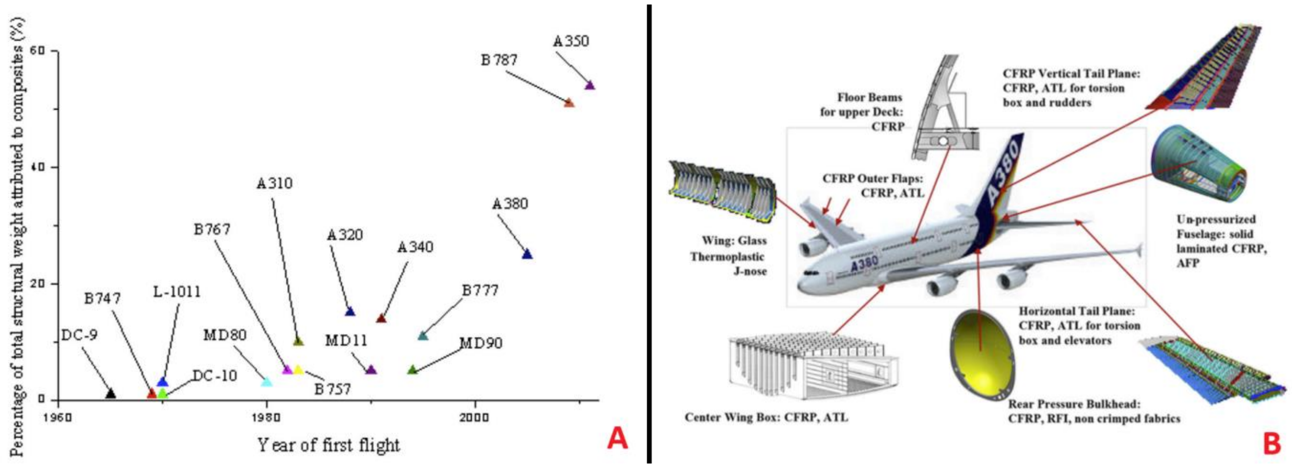

Figure 1 The proportion of the overall structural weight made up by composite materials and their application in the Airbus A380: (a) the share of the total structural weight contributed by composites, and (b) how composites are utilized in the Airbus A380 [6]

b) Automotive Industry:

In Automotive sector, composites are employed to manufacture lightweight components that contribute to improved fuel efficiency and reduced emissions. Examples include body panels, interior components, and structural elements.

Figure 2 Mercedes-Benz A-Class features components manufactured using natural fiber-reinforced composites[7]

c) Marine Industry:

Boats, ships, and marine structures benefit from the corrosion-resistant properties of composites. Hulls decks and other components are often made using composite materials to increase durability and reduce maintenance requirements.

Figure 3 A canoe made from flax/PLA composites was developed as part of the NAVECOMAT project.[8]

d) Wind Energy:

Wind turbine blades are commonly made from composite materials due to their strength, flexibility, and lightweight nature. Composites play a crucial role in the development of efficient and durable wind energy systems.

Figure 4 Manufacturing of wind turbine blades using composite material[9]

e) Construction and Infrastructure:

Composites materials are used in construction for applications such as bridge components, reinforcing materials, and building structures. They offer high strength and durability while potentially reducing overall weight.

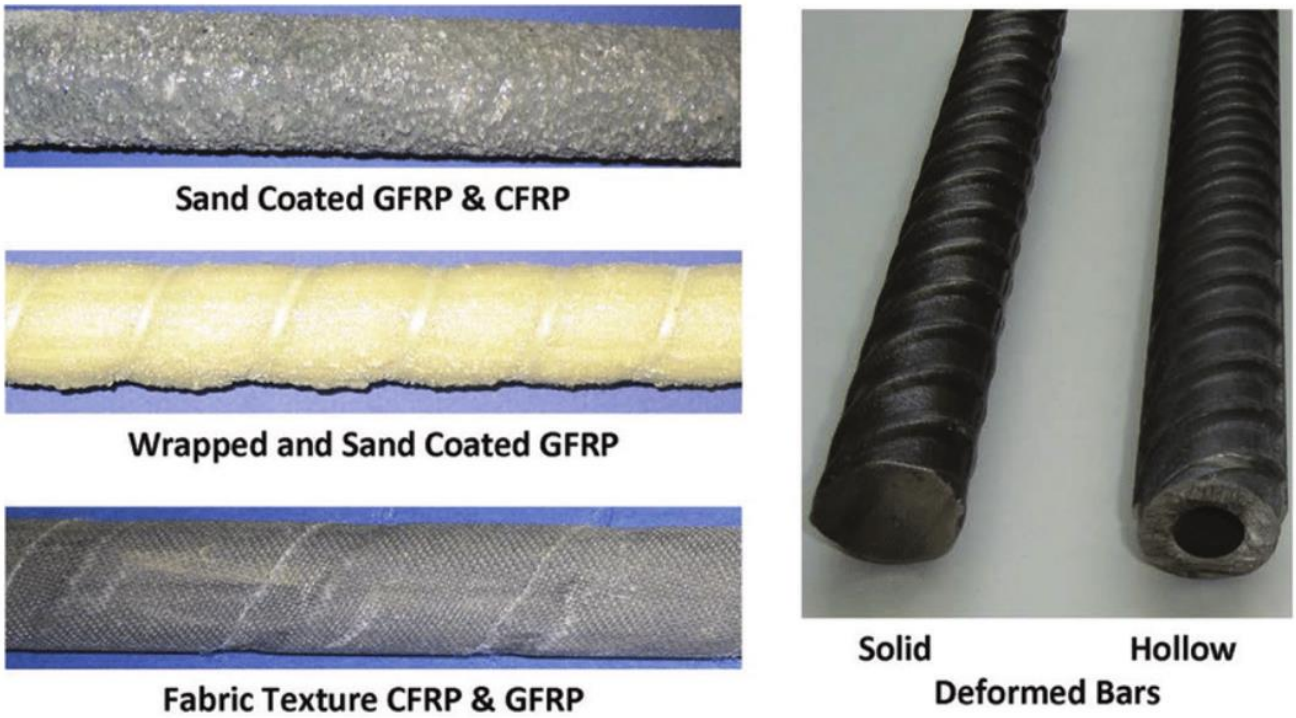

Figure 5 Carbon, glass fibre and steel bars[10]

f) Medical Industry:

Composite are used in the medical field for applications like orthopedic implants, dental materials, and prosthetics. The biocompatibility and tailored mechanical properties of composites makes them suitable for various medical applications.

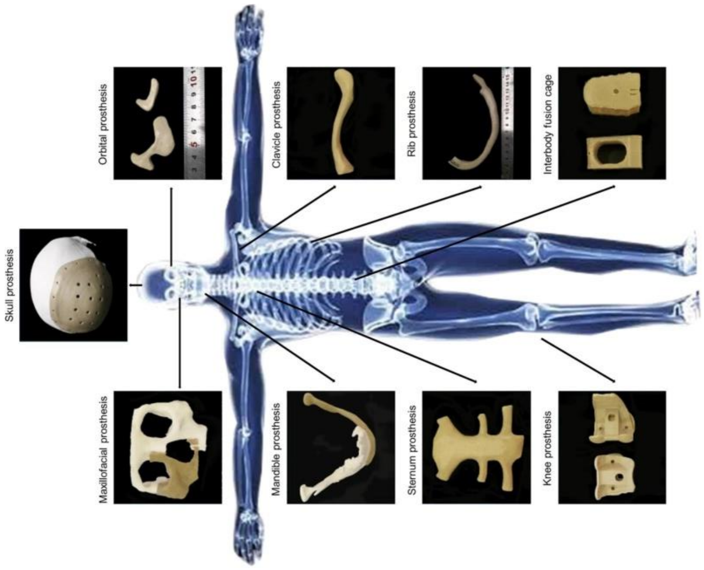

Figure 6 The applications of PEEK implants in orthopedics[11]

g) ***Sports and Recreation:***

Equipment in sports and recreation, such as bicycles, tennis rackets, golf clubs, and helmets, often incorporate composites. These materials provide a balance of strength and flexibility, contributing to improved performance.

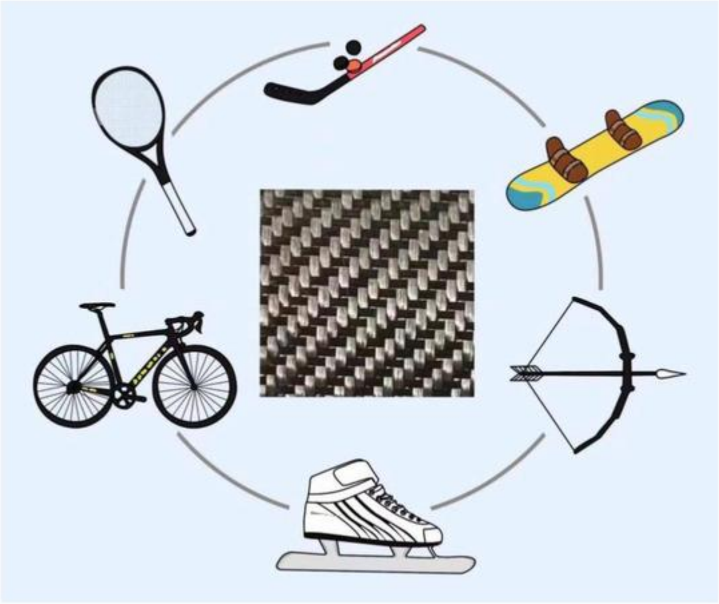

Figure 7 Carbon fibre based sports equipments[12]

h) ***Electronics and Electrical Industry:***

Composite materials are used in electronic devices and electrical insulation due to their electrical insulating properties. They can be utilized in circuit boards, casings, and insulating components.

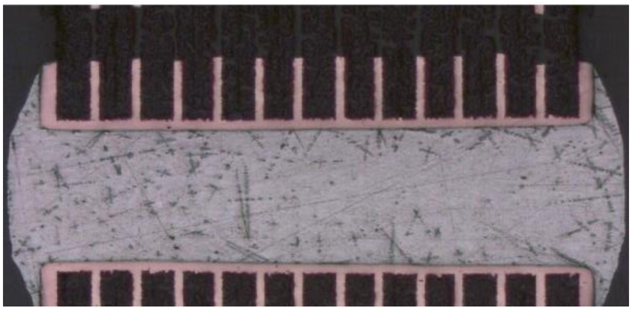

Figure 8 Use of non-woven aramid-polyimide composite materials for printed circuit boards in spacecraft electronic systems. [1]

i) ***Oil and Gas Industry:***

Composites are employed in the oil and gas sector for the construction of pipes, tanks, and other structures. Their resistance to corrosion and chemicals makes them suitable for harsh environments.

Figure 9 Glass Reinforced Epoxy (GRE) Pipes for oil transportation[13]

j) ***Defense and Military Applications:***

Components in defense and military equipment, such as armor, air craft parts, and vehicle structures, often incorporate composite materials. their strength, durability, and resistance to certain environmental conditions make them valuable in these applications.

Figure 10 Composite materials application in military armor

I.3. PVC as a Matrix material:

I.3.1. Definition

Polyvinyl chloride is a synthetic polymer derived from the polymerization of vinyl chloride monomers. It is a thermoplastic material characterized by its excellent chemical resistance, durability, and versatility. PVC can be rigid or flexible, depending on the formulation and processing. [14]

In material science and engineering, the term MATRIX MATERIAL refers to the continuous phase of composite material, which surrounds and binds the reinforcing materials, In the context of polyvinyl chloride (PVC), a widely used thermoplastic polymer, PVC as a matrix material implies the utilization of PVC as the continuous phase in a composite structure.[15]

I.3.2. Properties of PVC:

PVC is favored for many applications due to its high strength-to-weight ratio and good electrical and thermal insulation. What’s more, rigid PVC may be used in temperatures up to 60℃ without melting, and it’s easily bonded, welded, machined, bent, or shaped. In addition, products made from PVC are recyclable, though they may be difficult to recycle due to the high chlorine content and some recycling processes may alert the chemical composition.[16]

PVC is known by its Mechanical Resistance and strength especially when used in rigid forms, also his Chemical Resistance to wide range of chemicals, acids, and basses such as diluted acids, diluted alkalis, and aliphatic hydrocarbons.[16, 17]

PVC is a good Electrical insulator, making it suitable for use in electrical and electronic applications, including cable insulation and wiring for residential buildings, vehicles, it is also known by its Versatility because PVC can be formulated to be rigid or flexible depending on the application requirements.

PVC is easy to process using common techniques, such as extrusion, injection molding, and calendaring. This makes it widely used in different industries. PVC also has a good thermal stability, which means it can keep its properties over a wide range of temperatures. This is important for applications that need to withstand temperature changes. PVC is also water-resistance and does not absorb moister easily. This makes it suitable for applications that need to be water-resistance, such as pipes and fittings. PVC can also made biocompatible, and it is used for medical applications, such as medical tubing and blood bags.

I.3.3. PVC applications:

a) construction materials:

- **window profiles:** Reinforced PVC is used to manufacture durable and energy-efficient window frames.
- **Pipes and Fittings**: Glass fiber-reinforced PVC pipes are widely used in plumping for their strength and corrosion resistance.
- **Decking and Cladding**: PVC composites, reinforced with fibers or particulates, used for outdoor decking and cladding due to their durability and resistance to environmental conditions. [14]

b) Automotive Components:

- **Interior Component**s: PVC composites with reinforcements are employed in the production of automotive interior components such as dashboards and door panels. [18]

c)*Electrical Electronics:*

- **Cable Insulation**: PVC composites are used for cable insulation, ensuring electrical safety and insulation properties.
- **Enclosers and Housings**: Reinforced PVC is used to manufacture enclosures and housings for electrical equipment. [19]

d) Marine Applications:

- **Boat Components**: PVC composites are used in the production if boats components, offering resistance to water and environmental conditions. [20]

e) ***Consumer Goods:***

- **Furniture:** PVC composites are used for furniture applications, including outdoor furniture, where resistance to weathering is essential.
- **Consumer Products**: Reinforced PVC is used in the production of various consumer goods such as sporting equipment, luggage, and toys.[21]

f) ***Industrial Applications:***

- **Chemical Tanks and Containers**: PVC composites with enhanced chemical resistance are used for manufacturing chemical tanks and containers.
- **Industrial Containers**: Reinforced PVC is used in the production of durable and flexible industrial curtains. [22]

g) ***Infrastructure and Civil Engineering:***

- **Panels and Sheets:**PVC composites are used in the construction of panels and sheets for application like signage, barriers, and partitions.
- **Bridge Components:** Reinforced PVC may be used in certain bridge components due to its combination of strength and lightweight properties. [23]

h) ***Medical Applications:***

PVC composites are used for medical tubing due to their biocompatibility and ease of sterilization, also it can be used in: blood and plasma transfusion sets blood bags container for urine continence and ostomy products etc.[24]

I.3.4. Challenges and limitations of using PVC as a matrix material:

PVC have some challenges and limitations as a matrix material, we can mention:

a) ***Environmental impact:***

PVCproduction and disposal can release toxic substances into the environment, such as chlorine, mercury, asbestos, and dioxins.These substances can pose health risks to humans and wildlife, and contribute to global warming and ozone depletion. [25]

b) ***Low toughness and heat-softening temperature:***

PVC haslow facture toughness and impact strength, which limits its applications in high-stress or high-temperature conditions. PVC also softens at temperatures above 80ºC, which can affect its dimensional stability and mechanical properties. [26]

c) ***Compatibility and plasticization issues:***

PVC is not compatible with many other polymers, which can affect the interfacial adhesion and dispersion of the filters in the matrix. PVC also requires the addition of plasticizers to improve its flexibility and processability, but these plasticizers can migrate out of the matrix over time, causing degradation and environmental pollution.[27]

d) Flammability and smoke emission:

PVC cancatch fire easily and release dense, toxic smoke when burned. This can pose fire hazard and health risks in buildings and other applications where PVC is used. [28]

e) Color fading and yellowing:

PVC can lose its color and turn yellow over time, especially when exposed to UV light, heat, or oxygen. This can affect the appearance and aesthetics of PVC products. [29]

f) Biofouling and biodegradation:

PVC can be colonized by microorganisms, such as bacteria, fungi, and algae, which can degrade its surface and affect its properties. This can be a problem in applications where PVC is inContact with water or soil. [30]

g) Recycling difficulties:

PVC can be difficult to recycle, as it requires separation from other plastics, cleaning, and dichlorination. PVC can also contaminate other plastics during recycling, reducing their quality and value.[31]

I.4. Natural fibers in composite materials:

Natural fibers have recently caught the attention of academic and industry researchers due to their ease of availability, environmental friendliness, and biodegradability. Natural fibers derived from plants, such as jute, sisal, banana, flax, hemp, coir, kenaf, and many more, have been extensively researched over the last two decades and are gaining prominence over synthetic fibers. Natural fiber composite materials exhibit excellent strength and stiffness, high fracture resistance, as well as superior thermal and acoustic insulation qualities. Natural fiber reinforced composites have the potential to minimize component cost and material waste in automotive applications. The rising need for lighter, safer, and more fuel-efficient automobiles has led to the increased use of natural fiber composite materials in automotive applications by European automakers over the last decade. This paper investigates recent advancements in the usage of natural fiber-based composite materials in the automobile sector. [7]

I.4.1. Overview of Natural fibers:

Natural fibers are derived from plants, animals, and minerals. Plant-based cellulosic natural fibers are more cost-effective than animal-based fibers. Furthermore, unlike mineral-based fibers, these are not harmful to human's health. As a result, the NFRCs made from plant-based cellulosic fibers are the primary focus while considering the automobile application. [32]

Natural fibers in simple definition are fibers that are not synthetic or manmade. They can be sourced from plants or animals. [33]

I.4.2. Types of natural fibers:

a) *Animal Fibers:*

i. *Silk fiber:*

Silks are protein materials produced by a wide range of insect and spider species. They are used for applications requiring high-performance fibres. Silk is produced by insects and arachnids to make structures such as webs, cocoons and nests. Silk from silkworm cocoons (of the moth Bombyx mori) has been used by mankind to make fabrics, because it has excellent mechanical properties, particularly its high tensile modulus. [34]

The silk of the domesticated silkworm (B. mori) has been used as a suture biomaterial for centuries, and in recent years farmed silkworm silk has also been reprocessed into forms such as films, gels and sponges for medical applications. Spider silks also have outstanding strength, stiffness and toughness that, weight for weight, are unrivalled by synthetic fibres.

Structural proteins are commonly fibrous proteins such as keratin, collagen and elastin. Skin, bone, hair and silk all depend on such proteins for their structural properties. The structures (several types have been recorded) all consist of silk based on anti-parallel sheets of the fibrous protein fibroin. Long stretches of the polypeptide chain consist of sequences (Glycine- Sericin- Glycine-Alanine-Glycine- Alanine), where the symbols indicate different amino acids. The Gly chains extend from one surface of the-sheets and the Ser and Ala from the other, forming an alternating layered structure. The orientation of the chains along the sheet under- pins the tensile strength of silk, while the weak forces between sheets ensure that silk fibres are flexible. Silk fibres have a complex hierarchical structure, in which a fibroin core is surrounded by a skin of the protein sericin. Within the core, termed bave, there are crystalline regions containing layered sheets and amorphous regions that may contain isolated sheets.[35]

ii. *Agriculturally Derived Proteins:*

Other than animals, agricultural materials can also be considered as an ideal source of protein and are prospective materials for the preparation of fibers. Fibers of regenerated protein were produced commercially in between 1930 and 1950, and by today’s standards, they would be considered natural, sustainable, renewable, and biodegradable. Casein from milk was used by M/s Courtaulds Ltd. to make Fibrolane and by M/s Snia to make Lanital; groundnut (peanut) protein was used by M/s ICI to make Ardil; Vicara was made by the M/s Virginia–Carolina Chemical Corporation from zein (corn protein); and soybean protein fibre was developed by the Ford Motor Company. [36]

The regenerated fibres had several qualities typical of the main protein fibres, wool and silk; they were soft, with excellent drape and high moisture absorbency. They could be processed on conventional textile machinery and coloured with conventional dyes. Superior to wool in some regards, they did not prickle, pill or shrink. They could be produced as staple or filament, crimped or straight, with control over diameter, and dope-dyed if required. Regen- erated protein fibres are potentially environmentally sustainable, renewable and biodegradable. Two protein sources, feather keratin and wheat gluten, have been considered for their suitability to make an eco-friendly regenerated fibre. Both appear to be viable, although low wet strength may make it problematic. The inclusion of nanoparticles and use of cross-linking technologies offer the potential to improve mechanical strength to make them fit for use in apparel or technical textile applications. Wool is similar to feather in some regards, both keratins being highly cross-linked, although wool proteins are heterogeneous with a generally higher molecular weight (10–55 kDa) and higher cysteine content. [37]

b) Plant fibers:

i. _Leaves fibres:_

The fibers are made by overlapping bundles that run along the leaves to reinforce them, these fibres are hard and rigid.[38]

ii. _Stem Fibers:_

Stem fibers are obtained from the stems of dicotyledonous plants. Their role is to provide good rigidity to plant stems. The most commonly used stem fibers are those of flax, kenaf, and hemp.[38, 39]

iii. _Wood fibers:_

Wood fibers are obtained from the grinding of trees as bamboo.[40]

iv. _Surfacefibers:_

Surface fibers are generally surrounding the surface of stems, fruits, or grains. Surface fibers of grains constitute the most important group in this family of fibers. Among others, we mention cotton and coconut (coir). Coconut fibers have yielded good results for the flexural strength of fiber cement.[41]

I.4.3. Date palms:

The date palm is typically cultivated in desert Oases. The one originating from North Africa is extensively cultivated from Arabia to the Persian Gulf, where it forms the characteristic vegetation of oases.

It is a cold-sensitive plant that grows on any type of soil, provided it is fertile and well-drained, primarily used as an ornamental plant for its slender appearance and foliage. [42]

I.4.4. The palm:

The palm is a pinnate leaf adorned with regularly arranged leaflets positioned obliquely along the upper part of the rachis. The lower segments are transformed into thorns, more or less numerous, and of varying lengths. The number of palms varies between 30 and 150, arranged spirally with a length reaching 350 to 450 mm. The palm tree produces 10 to 20 palms per year depending on the varieties and the cultivation method. These last ones live and remain green for 3 to 7 years before they become dry and inclined, after they would be removed by pruning.

Figure 11 Date palm from Biskra Region

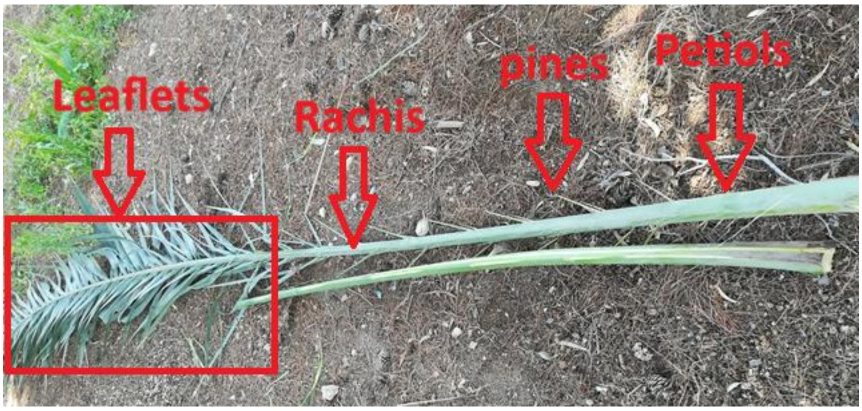

Figure 12 Diagram of the palm structure

I.4.5. Leaflets:

Leaflets or SAÅF in Arabic, are the small leaves scattered on both sides of the palm. They have an epidermis covered with a waxy coating and pointed shape at the ends. The attachment to the palm occurs at various angles depending on the variety of palm tree. The portion occupied by the leaflets represents 65% of the total length of the palm; It contains individual leaflets and leaflets grouped in twos, threes, fours, or fives. The length and width of the leaflets depend on the variety of the palm tree, the maintenance it has undergone, and the environmental conditions. Leaflets can have a length ranging from 15 to 85 cm and a width between 1 and 6 cm. However, the number of leaflets on palm varies between 120 and 240 depending on the size and maturity of the palm. [43]

I.5. Literature Review

The study of (K. Almi, 2018)[43], focused on the valorization of date palm wood waste, for the production of low-cost, bio-based composite materials. Eight types of wood from the Deglet Nour variety were characterized and showed similar properties, allowing them to be treated as a single raw material.The palm wood exhibited low density (<600 kg/m³), high porosity (>80%), high water absorption (>100%), and a composition of ~40% cellulose, ~20% hemicellulose, and ~28% lignin. Mechanical tests showed moderate performance (MOE: 3–8 GPa; MOR: 86–213 MPa), comparable to coconut wood.Composite panels were developed using PVA glue and polyester resin under various processing conditions. Optimized results showed promising density (950–1070 kg/m³), mechanical strength (MOR > 20 MPa), and acceptable thermal conductivity (~0.2 W/m·°C). These findings support the use of date palm waste as a viable material for rigid, lightweight composites in dry applications.

(T. Masri, 2018) [44] developed two innovative wood–plastic composites (WPCs) using date palm waste.The first, a thermoplastic composite called LPC (Leaflet Polystyrene Composite), combines fibers from date palm leaflets with recycled polystyrene dissolved in solvent. Various fiber sizes (0.1–1 mm) and reinforcement ratios (70–80%) were tested. LPC showed wood-like density, thermal insulation properties comparable to MDF and plywood, and moderate mechanical performance. While its bending modulus is lower than MDF, its insulation is ~50% better than similar composites with wood waste. Small fiber sizes improved fiber–matrix adhesion, confirmed by SEM, though mechanical strength remains limited—suggesting the need for protocol improvements or hybrid reinforcement strategies.The second composite, a thermosetting material, uses ground date seeds as filler in an epoxy matrix. Composites with up to 40% seed content were prepared. Mechanical tests showed enhanced flexural strength at ≥30% reinforcement, although brittleness increased beyond this point. The epoxy's mechanical properties were in line with those in existing literature.Overall, date palm waste shows strong potential for creating low-cost, recyclable composites with thermal insulation and acceptable mechanical properties, especially in dry environments.

The work of (F. Touloum, 2018) [45]aimed to evaluate the potential of date palm wood waste (Phoenix Dactylifera L.) as reinforcement in cement-based materials. Mortars and cement pastes were formulated with varying wood content (up to 4% and 10% respectively), and their thermal and mechanical properties were analyzed.Date palm wood showed low thermal conductivity (0.061 ± 0.006 W/m·K) and a density of 1500 ± 1 kg/m³. Morphological analysis revealed two fiber types differing in structure and size, contributing to the wood's low bulk density.***Mortar Study:***Thermal conductivity decreased with increasing wood content, reaching 0.64 W/m·K at 4%, comparable to other bio-composites (e.g., coconut fiber mortar). Compressive strength decreased with wood addition, but at 3%, the material retained good strength (30.51 MPa), outperforming similar composites from literature. Flexural strength improved up to 3% wood, indicating efficient stress transfer due to particle size and compatibility with Portland cement. ***Cement Paste Study:***Thermal conductivity further dropped to 0.24 W/m·K at 10%, aligning with ASTM standards for lightweight concrete. Unexpectedly, compressive strength remained stable up to 8% wood content, beyond which a decline was observed. The 8% composite met structural criteria for wall covering applications. Flexural tests confirmed these trends, showing enhanced mechanical performance at 8%, likely due to improved interfacial adhesion.

The study of (T. Guettaf Temmam, 2018) [46]investigated three date palm varieties from Biskra—Deglet Nour, Mech Degla, and El Ghers—focusing on their structural, physical, and mechanical properties to assess their suitability in sustainable material applications. Optical and microstructural analyses revealed a dual-layer wood structure: an outer "skin" rich in fine fibers and an inner "core" with fewer but thicker fibers, with both layers showing increased fiber density along the longitudinal axis. This configuration supports the fruit load and resists harsh desert conditions. Water absorption tests showed El Ghers had the highest rate, followed by Mech Degla and Deglet Nour, forming a baseline for technical classification. Moisture content varied similarly, with El Ghers categorized as soft, Deglet Nour as semi-soft, and Mech Degla as dry. Tensile testing across different drying durations (2 to 288 hours) demonstrated a progressive increase in Young's modulus, ranging from 7036 to 16161 MPa, with the outer layer consistently exhibiting superior stiffness compared to the core.

The paper of (H. Benchouia, 2024) [47]explores the development of an eco-friendly gypsum plaster hybrid biocomposite by incorporating two abundant Algerian wastes—date palm petiole fibers (DPP) and expanded polystyrene (EPS)—aiming to create a material that matches or surpasses the properties of neat gypsum plaster (NGP). Handcrafted samples were prepared with varying DPP loadings (0, 5, 10, and 15%) and a fixed EPS content (0.3%), both individually and combined. Comprehensive morphological, mechanical, and thermophysical tests were conducted. FTIR analysis confirmed the presence of characteristic functional groups from gypsum, EPS, and DPP, while XRD revealed crystallinity levels of 71.58% for NGP, 29.91% for EPS, and 52.93% for DPP. Mechanical testing indicated that adding EPS, DPP, or both decreased compression strength, flexural strength, and Young's modulus. However, the hybrid biocomposites showed improved thermal insulation, with reduced thermal conductivity values (0.265–0.414 W/m·K) and lower bulk densities (852–925 kg/m³) compared to NGP (0.425 W/m·K and 977 kg/m³, respectively). These findings suggest that incorporating DPP and EPS into gypsum plaster presents a promising, sustainable alternative for thermal insulation in construction.

His other paper [48], This study aims to reduce both the environmental impact and the cost associated with petroleum-based polystyrene by incorporating waste date palm leaves into polystyrene/date palm fiber composites (PS-DPF), targeting mechanical and thermal insulation properties comparable to or better than virgin polystyrene (VPS) or other insulating materials. Three types of fibers were used—untreated (UDPF), alkalinized (ADPF), and benzoylated (BDPF)—at filler loadings of 10%, 20%, and 30% by weight. The composites were fabricated using melt mixing followed by hot compression molding. Morphological, mechanical, and thermophysical characterizations were conducted. SEM images revealed improved filler dispersion and matrix interaction, attributed to reduced hydroxyl content, as confirmed by FTIR. XRD results showed that ADPF exhibited the highest crystallinity. The composites demonstrated tensile strength of 14–27 MPa, flexural strength of 31–44 MPa, and moduli ranging from 2.9 to 5.9 GPa. Both alkaline and benzoyl treatments enhanced tensile and flexural strength while slightly lowering modulus values, showing similar effects overall. TGA analysis confirmed that PS-DPF samples with 30% fiber content offered higher thermal stability than VPS, with untreated fibers slightly outperforming the treated ones in residue retention. The composites also showed low thermal conductivity (0.118–0.141 W/m·K at 18°C) and a bulk density ranging from 860 to 980 kg/m³, which decreased as fiber content increased. Replacing one-third of conventional building material compositions with PS-DPF composites led to a thermal conductivity reduction of up to 50%, highlighting their effectiveness as thermal insulators and demonstrating the potential of using agricultural waste in construction applications, especially in date-producing countries.

In terms of PVC-date palm composites, according to the authors’ knowledge no investigations has been established before, except the study of (H. Boussahel, 2024) [49]which has been recently published where it explores the potential of date palm fibers as reinforcement for polyvinyl chloride (PVC) composites, emphasizing the impact of benzoyl chloride surface treatment on their mechanical and thermal performance. The treatment significantly enhanced the adhesion between fibers and the PVC matrix, as confirmed by FTIR spectroscopy and SEM surface morphology analysis. As a result, the tensile strength of the treated composites (PVC-BTF) improved from $11.3 \pm 0.48\,\mathrm{MPa}$ in untreated composites (PVC-UTF) to $12.8 \pm 1.12\,\mathrm{MPa}$, while the tensile modulus increased markedly from $112.2 \pm 11.06\,\mathrm{MPa}$ to $304.5 \pm 7.26\,\mathrm{MPa}$, indicating a substantial reinforcement effect. Thermal analysis further confirmed improved thermal stability after treatment. Although hydrothermal aging tests revealed a 21.68% reduction in tensile strength for untreated composites due to water absorption and fiber-matrix interface degradation, the treated composites exhibited reduced water uptake and better preservation of mechanical properties. These results suggest that benzoyl chloride-treated date palm fibers can serve as an effective and eco-friendly reinforcement in PVC composites, enhancing strength, stiffness, and thermal resistance, and making them well-suited for moderate-performance applications in environments with mild moisture exposure.

1.6. Conclusion

This chapter highlighted the growing importance of composite materials in modern engineering, emphasizing their superior mechanical, thermal, and chemical properties compared to traditional materials. Among these, PVC stands out as a versatile thermoplastic matrix widely used across multiple industries. At the same time, the shift toward sustainable solutions has led to increased interest in natural fiber reinforcements. Date palm leaflets, as an abundant agricultural by-product, offer a promising eco-friendly alternative. The integration of such natural fibers into PVC composites opens the door to developing materials that are both high-performing and environmentally responsible.

In a broader socio-economic context, the present study aims to explore and evaluate the physical, thermal, and mechanical properties of polyvinyl chloride (PVC) composites reinforced with date palm leaflet powder, with the ultimate goal of promoting the industrial utilization of this abundant and sustainable bio-reinforcement. To achieve this, a comprehensive experimental approach has been adopted. The chemical characterization of the composites is conducted using Fourier Transform Infrared Spectroscopy (FTIR). The mechanical behavior is assessed through tensile and flexural (bending) tests to determine the strength and flexibility of the composites, alongside Shore Hardness testing to measure surface resistance. Thermal stability analysis is also performed to evaluate the composite’s performance under varying temperature conditions, which is crucial for practical industrial applications. In this context, three surface treatments—mercerization, acetic acid, and silane coupling agent—were selected for fiber modification, chosen for their cost-effectiveness and proven efficiency in enhancing fiber-matrix adhesion and overall composite performance.

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CHAPTER II

METHODOLOGY MATERIALS AND APPARATUS

II.1. Introduction

The methodology outlined in this chapter encompasses a series of systematic steps, ensuring the reproducibility and reliability of the results. It begins with the extraction and preparation of date palm leaflets followed by the compounding and moulding processes involved in creating the composite materials, each stage is meticulously detailed, with a particular focus on the physical and mechanical tests used to evaluate the final product. These include Fourier Transform Infrared Spectroscopy (FTIR) for chemical characterization. The mechanical properties of the composites are examined through tensile and bending tests to determine strength and flexibility, while Shore Hardness testing evaluates surface hardness. Additionally, thermal stability is assessed to understand the composite's behaviour under temperature variations. Moreover, the chapter provides insights into the apparatus employed for these processes, elucidating the role of each instrument in achieving the desired composite properties.

II.2. Materials

II.2.1Date Palm Leaflet

Figure 13 Date palm leaflet

The date palm (Phoenix dactylifera L.) is a highly valuable plant, particularly in theTolga region of Biskra, Algeria, where the Deglet-Nour variety is cultivated. The palm plants in this study were moderately mature, approximately 10 years old. Date palm leaflets are a by-product of this plant known for their fibrous nature, which makes them a suitable candidate for composite material production.

The preparation of the date palm leaflets followed a systematic methodology to enhance their properties and facilitate their use in composites. The process began with harvesting the leaflets, which were then cut into pieces measuring between 1 and 2 cm. These pieces were washed twice, first in cold distilled water and then in hot distilled water to remove any remaining impurities. Figure II-1 illustrates this process.

Figure 14 Date palm leaflet preparation process

II.2.2. Chemical treatments

Three types of chemical treatments were employed to modify the surface microstructure of the leaflets in order to enhance fiber-matrix bonds.

a) ***NaOH Treatment***

A 2% wt. Solution of sodium hydroxide (NaOH) from CLCM Labs (Laghouat-Algeria) was prepared, and the leaflets were immersed in this solution for 1h. Afterward, the leaflets were rinsed with distilled water containing 0.01 mol/L of acetic acid to neutralize the soda effect. Finally, a simple rinse using distilled water was carried out to remove any residual chemicals.The obtained leaflets were divided into three parts: the first one remained treated with NaOH, the second part was treated with acetic acid, and finally, the third part was treated with silane.

a) ***Acetic acid treatment***

A 1% wt. Solution of acetic acid was dissolved in distilled water for 15 min. The NaOH treated fibers were added to the acetic acid solution at room temperature for 1H. After treatment, the leaflets were rinsed with distilled water until a PH of 7 was achieved.

a) ***Silane Coupling Agent treatment***

The third treatment involved the use of [3-(Methacryloyloxy) propyl] trimethoxysilane (Silane A174). To prevent the polymerization of the Silane into polysiloxane, a 1% wt. Solution of Silane A174 was prepared by dissolving it in distilled water, followed by the addition of acetic acid until a pH of 2.9-4 was achieved. After 15 minutes of hydrolysis, the NaOH treated leaflets were added to the silane mixture at room temperature for 1 hour. The treated leaflets were then rinsed with distilled water until pH of 7 was reached.

Figure 15 NaOH Treatment process

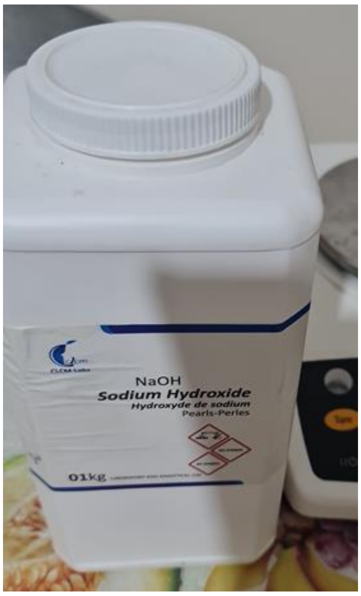

Figure 16 Sodium Hydroxide (NaOH) used in this study

Figure 17 Acetic acid treatment process

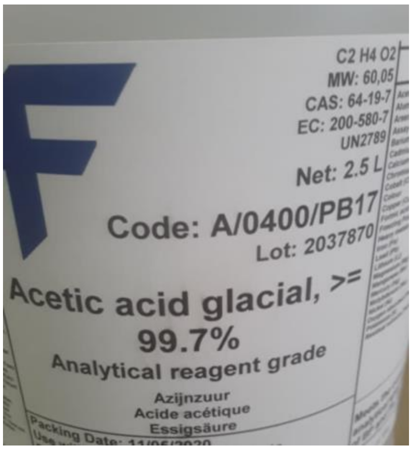

Figure 18Acetic Acid

Figure 19 [3-(Methacryloyloxy) propyl] trimethoxysilane (Silane A174)

For all chemical treatments, the leaflets were subsequently dried in an oven at 80°C for 24 hours to remove any remaining moisture before being processed further.

Once dried, the leaflets were ground into a fine powder. The grinding was carried out using BOMANN KSW 6504 CB. The obtained powders were sieved using an AS-200 sieve shaker from RETSCH. The AS-200 is an advanced apparatus designed for the precise classification of particle sizes in a wide range of materials. It features a high-frequency sieve motion that ensures efficient separation and accurate results. The leaflets were classified into two distinct particle sizes for NaOH and acetic acid powders: 63µm and 125µm. For the Silane treated leaflets, regarding the insufficiency of the prepared quantity we had only one size of the powder: 63µm.

Figure 20 Date palm leaflets at the drying process

The resulted treated and untreated powders are given the following codes:

- **U**ntreated **L**eaflet **P**owder: **ULF**
- **A**lkaline **T**reated **L**eaflet **P**owder: **ATLP**
- **A**cetic **Ac**id Treated **L**eaflet **P**owder: **ACLP**
- **S**ilane **T**reated **L**eaflet **P**owder: **STLP**

Figure 21 BOMANN KSW 6504 CB

Figure 22 The 63µm and 125µm sieves

Figure 23 RETSCH AS-200 sieve shaker

II.3. Composites Preparation

The preparation of the PVC-based composite materials with date palm leaflet powder (DPLP) as a filler was carried out in the ENICAB-Biskra, Algeria laboratory using the two-roll mill calendering process, utilizing the SCHWABENTHAN “Polymix 200 P” model. The standard calendering process for PVC typically operates at temperatures between 160°C-170°C, ensuring that the material is soft enough for processing but not subjected to excessive thermal degradation. The addition of the filler was carried out carefully to maintain the integrity of the PVC matrix and achieve optimal dispersion within the composite.

The standard parameters for calendaring process were followed:

- The matrix material used was Polyvinyl Chloride (PVC with Jayflex DIDP plasticizer) and the DPLP as a filler, the calendar rolls were set to the optimal processing temperature range of 160°C-165°C, which is suitable for PVC fusion. The temperature was carefully controlled to avoid degradation of the polymer while ensuring proper mixing of the PVC with the filler material.
- PVC resin was initially loaded into the calendar, and mixing began using the two-roll mill. The mixture was continuously processed for duration of 10 minutes to achieve complete polymer fusion. During this stage, the PVC matrix was softened and prepared for filler incorporation.
- After achieving polymer fusion, the date palm leaflet powder was gradually introduced into the PVC. The filler was evenly distributed by continuous milling between the calendar rolls.

The filler content was adjusted based on the desired composite properties, ensuring homogeneity.

- The compounded PVC and DPLP mixture was passed through the rolls, where it was further processed to a uniform thickness. The calendaring process also helped in improving the dispersion of the filler within the PVC matrix, ensuring that the composite material exhibited consistent mechanical properties.
- After achieving the desired thickness and uniformity, the composite sheets were cooled and removed from the calendar rolls. The final product was then cut into small square pieces of 1-2cm for further processing.

After the calendering process, the obtained small pieces were further processed using moulding to achieve the final shape and thickness. The moulding procedure was carried out as follows:

- The composite material, in the form of a sheet from the calendaring stage, was placed into the moulding cavity of the “SchwabenthanPolystat 300S” hydraulic press. The moulding was carried out at a temperature range of 160°C-165°C, which corresponds to the optimal processing conditions for PVC, ensuring the material remained pliable without risk of thermal degradation.
- A preheating step was carried out to prepare the composite material and remove any trapped air. This was crucial to avoid the formation of air bubbles within the final product, which could affect the mechanical properties of the composites. During the preheating phase, the material was heated to the moulding temperature, and any gases or volatiles were allowed to escape.
- After preheating and degassing, the material was subjected to a pressure of 300 bars for a duration of 10 minutes. The pressure ensured proper consolidation of the composites, facilitating uniform distribution of the filler and enhancing the bonding between the PVC matrix and the DPLP filler.
- Once the pressing time was completed, the composite was allowed to cool under pressure to solidify the material and maintain its final shape. After cooling, the moulded composite plate was carefully removed from the mould.
- The resulting moulded composites were a 2 mm thick plate, which exhibited uniform properties and consistent filler dispersion. The plates were then cut into the required specimen shapes for testing or further processing.

Table 01 presents the datasheet of Jayflex DIDP Plasticizer.

Table 1 JayFlex DIDP Plasticizer datasheet

Properties	Minimum	Maximum	Unit	Test Method
Color, Pt-Co	-	30	-	ASTM D5386
Viscosity at 20°C	110	125	mPa.s	ASTM D445
Density at 20°C	0.064	0.968	g/cm3	ASTM D4052
Water Content	-	0.05	Wt%	ISO 12937
Acid Value	-	0.07	mgKOH/g	ASTM D1045
Ester Content	99.7	-	Wt%	RPP 201
Refractive index	1.484	1.486	-	ASTM D1218

The table 2 identifies the prepared PVC composite materials composition as follows:

Table 2 PVC composite materials composition

No.	Designation	PVC content (%)	DPLP Content (%)	DPLP size (µm)
1	PVC	100		
2	5-ULP-0.063	95	5	63
3	10-ULP-0.063	90	10	
4	15-ULP-0.063	85	15	
5	20-ULP-0.063	80	20	
6	5-ULP-0.125	95	5	125
7	10-ULP-0.125	90	10	
8	15-ULP-0.125	85	15	
9	20-ULP-0.125	80	20	
10	5-ATLP-0.063	95	5	63
11	10-ATLP-0.063	90	10	
12	15-ATLP-0.063	85	15	
13	20-ATLP-0.063	80	20	
14	5-ATLP-0.125	95	5	125
15	10-ATLP-0.125	90	10	
16	15-ATLP-0.125	85	15	
17	20-ATLP-0.125	80	20	
18	5-ACLP-0.063	95	5	63
19	10-ACLP-0.063	90	10	
20	15-ACLP-0.063	85	15	
21	20-ACLP-0.063	80	20	
22	5-ACLP-0.125	95	5	125
23	10-ACLP-0.125	90	10	
24	15-ACLP-0.125	85	15	
25	20-ACLP-0.125	80	20	
26	5-STLP-0.063	95	5	63
27	25-STLP-0.063	75	25	

Figure 24 SCHWABENTHAN "Polymix 200 P" model

Figure 25 A picture during the calendering process

Figure 26 A specimen of the resulting material after the calendering process

Figure 27 Schwabenthan Polystat 300S

Figure 28 A specimen of the resulting material after the moulding process

II.4. Physical analysis

II.4.1. FTIR Analysis

The FTIR analysis was performed following the guidelines of **ASTM E1252-98 (2021)**, using two different FTIR instruments: the “SCHIMADZU FTIR-8400S” for STLP and “AGILENT CARY 630 FTIR” for other materials. The methodology described as follows:

- A small amount of the sample (0.001g), either treated or untreated, was mixed with 0.2g of Potassium bromide (KBr) to form pellet. The KBr serves as a matrix to disperse the sample uniformly.
- The sample-KBr mixture was thoroughly ground to ensure homogeneity and then compressed into a thin pellet using manual hydraulic press. The resulting pellet was used for FTIR analysis.
- The spectra were acquired in the mid-infrared range of 400 to 4000 cm^{-1}, covering the characteristic absorption bands of cellulosic and other organic materials.

- The resolution of the scans was set to 2 cm^{-1} to provide sufficient detail for spectral analysis, ensuring the detection of important functional groups.
- The FTIR spectra were processed using appropriate software (Origin Pro) for baseline correction, normalization, and peak identification (e.g., O-H stretching, C-H bending, C-O stretching) based on the established absorption bands for cellulosic fibers and other materials.

Figure 29 AGILENT CARY 630 FTIR

Figure 30 Potassium bromide (KBr)

II.4.2. SEM Analysis

The scanning electron microscopy (SEM) analysis of date palm leaflet fiberswasconducted using **Thermo Scientific Prisma E Scanning Electron Microscope**. This apparatus use the ESEM technique which is a specialized mode of SEM that allows for imaging of samples in their natural state or in a controlled environment without the need for extensive sample preparation, such as coating with conductive materials. ESEM enables the observation of specimens in varying gaseous environments (such as air, water vapour, or other gases) at relatively low vacuum levels, which is particularly useful for biological, hydrated, and non conductive samples.

Figure 31Thermo Scientific Prisma E Scanning Electron Microscope

II.4.3. Bulk density

The bulk density test using KERN Analytical Balance (ALS-A / ALJ-A) involves determining the mass and volume of a composite sample to calculate its bulk density. The mass (mc) is measured using the precise and accurate KERN balance, which provides high-resolution reading, up to four digits. The volume (Vc) of the sample was determined using a two digits precision calliper. Once both mass and volume are obtained, the bulk density (ρ_b) is calculated using equation 1:

$$\rho_b = m_c / V_c \tag{1}$$

Three samples were measured for each composite, the mean value was calculated after.

II.5. Thermal characterization

II.5.1. Thermal Stability Test

When exposed to high temperatures, such as those encountered in an indirect fire scenario, PVC releases hydrochloric acid (HCl), which accelerates its degradation. The thermal stability test helps assess the durability of these plastics.

Tests are performed at least 16 hours after the compounds are prepared, following IEC 60811-1-2 international standard. The test method uses glass test tubes that are 110 mm in length, with an external diameter of about 5 mm and an internal diameter of (4.0 ± 0.5) mm, universal pH paper (with a range from 1 to 10), and thermostat-controlled heating system to achieve the required temperature based on the specific cable type.

Three samples, each weighing (50 ± 5) mg, are taken from the composite materials. These samples consist of two or three small strips measuring 20 to 30 mm. The samples are placed in a glass tube with a strip of dry pH paper measuring about 15 mm by 3 mm. The pH paper should extend about 5 mm above the tube's top. These samples are then placed in the thermal heating equipement, which is pre-heated to the required temperature, and subjected to the test for the specified duration.

The thermal stability test evaluates the time it takes for the pH paper to shift from a pH value of 5 to a range from 2 to 3. The color change point is identified when the pH paper turns red, corresponding to a pH of 3.

To pass the thermal stability test, the average result of the three samples must meet or exceed the value specified in the relevant standard for that particular type of cable (90 minutes in our case).

Figure 32 Glass test tube for thermal stability test

Figure 33 Thermal stability Test

II.6. Mechanical characterization

II.6.1. Shore Hardness Test

The Shore Hardness of PVC composites is determined following the **ASTM D2240** standard, which involves measuring the resistance of the material to indentation. The test requires a Shore D durometer calibrated using a standard reference block to ensure accurate readings. The PVC composites samples are prepared as a flat plate with a thickness of 6 mm. Before testing, the sample should equilibrate at room temperatures for at least 1 hour. The Shore durometer is then applied perpendicularly to the material's surface, pressing the indenter until it makes full contact. The hardness value is read after 1-3 seconds of stabilization, five readings are taken at different points on the sample to ensure reliability.

The final result is the average of these measurements, which are reported along with any relevant observations.

Figure 34 Zwick/ Roell HPE

II.6.2. Tensile Test

The tensile test of the samples was performed in accordance with ISO 527 standard to determine their mechanical properties, including tensile strength, elongation at break, and modulus of elasticity. The test was conducted using a tensile testing machine INSTRON 5969 and Zwick/Roell Z050. The procedure involved placing the "H2" specimen into the jaws of the dynamometer, ensuring that the sample was aligned properly to avoid any misalignment during testing.

The deformation rate was controlled at 2 mm/min to maintain consistent strain rates during the test. A total of five samples from each composite material were tested. The testing was carried out until the samples were tested ruptured, and the mechanical properties of each sample were recorded for analysis.

II.6.3. Bending Test

The bending test was conducted in accordance with ASTM D790 to evaluate the flexural properties of the composite materials. The test was performed using an INSTRON 5969 universal testing machine. The specimens, with a thickness of 2 mm, were prepared in accordance with ASTM D790 standard, with dimensions of 127 mm in length, 12.7 mm in width, and 2 mm in thickness.

Five samples were tested for each material variety to ensure reliable and consistent results. The bending was applied at a constant rate, and the force required to produce a specified deflection was measured.

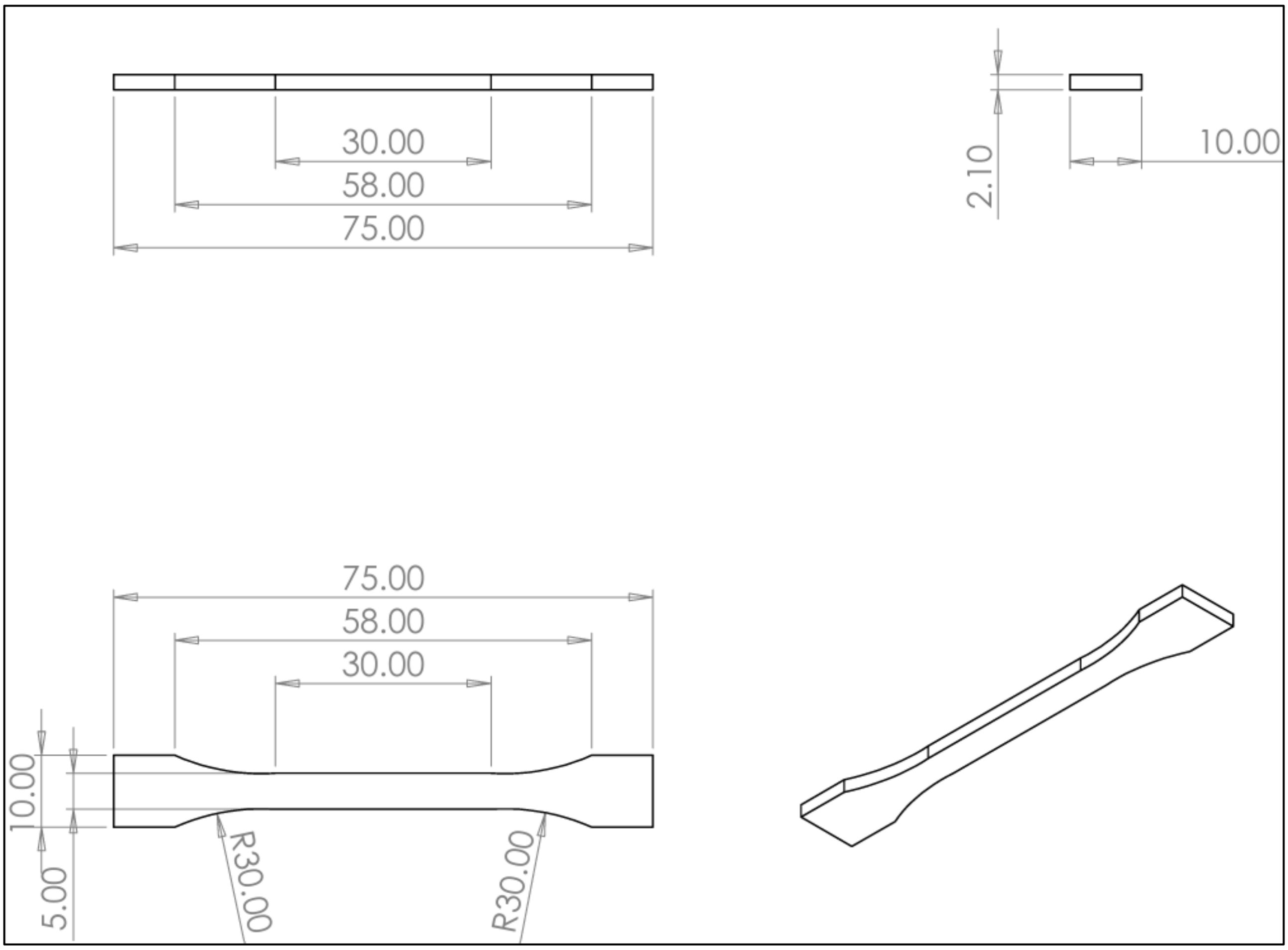

Figure 35 Tensile test H2 specimen dimensions

Figure 36 Testing machines INSTRON 5969 and Zwick/Roell Z050

Conclusion

This chapter has provided a comprehensive methodology for the development and characterization of PVC-based composites reinforced with date palm leaflet powder (DPLP). The process began with the extraction and preparation of date palm leaflets, followed by their chemical treatment (NaOH, acetic acid, and silane) to enhance fiber-matrix adhesion. The treated and untreated fibers were then incorporated into a PVC matrix through a carefully controlled calendering and moulding process, ensuring uniform dispersion and optimal mechanical properties.

A series of physical and mechanical tests were conducted to evaluate the composite materials. FTIR spectroscopy analysis was employed to assess chemical interactions, while SEM provided insights into the morphological changes induced by chemical treatments. Bulk density and thermal stability tests were performed to determine the material’s physical and thermal properties. Additionally, mechanical performance was evaluated through tensile, bending, and Shore hardness tests, providing a thorough understanding of the composite’s strength, flexibility, and durability.

The findings from these tests will be analyzed and discussed in the next chapter.

CHAPTER III

RESULTS AND DISCUSSION

III.1. Introduction

In this chapter, the results of the tests conducted on the composite materials and DPLP are presented and analyzed in detail. The data gathered from the FTIR, SEM, mechanical testing, and thermal stability assessments are examined to evaluate the performance of the composites. This chapter attempts to thoroughly understand these findings by emphasizing how the several elements—chemical treatments, and mechanical characteristics—help to determine the general composite performance. The results are reviewed in light of their possible uses and the pragmatic consequences of including date palm leaflets in the production of composite materials.

III.2. Acetic acid treated Results:

III.2.1. FTIR Analysis

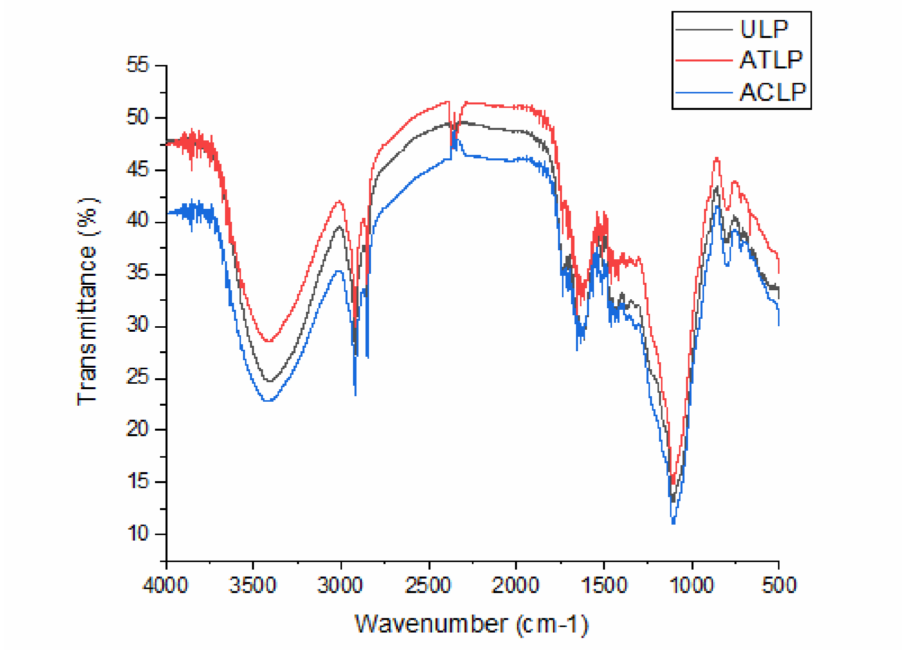

Figure 37 FTIR spectra of ULP, ATLP and ACLP powder

Presenting the FTIR data for untreated (ULP), alkaline-treated (ATLP), and acetic acid-treated (ACLP) leaflet powder in (Figure 35). The results expose notable chemical alterations brought about by the treatments, therefore offering information on structural and compositional changes. The ULP spectrum shows regular peaks connected to the inherent composition of plant fibers. A broad peak at [3400 – 3300] cm^{-1} corresponds to O-H stretching vibrations, primarily from cellulose and hemicelluloses, and absorbed moisture, indicating strong hydrogen bonding. The peak at 2900 cm^{-1} represents C-H stretching vibrations in cellulose and hemicelluloses, typical of organic materials. A distinct peak at 1730 cm^{-1} is attributed to C=O stretching vibrations in ester groups, likely from hemicelluloses and lignin, while the peaks at 1630 cm^{-1} are associated with absorbed water (H-O-H bending) and possibly C=C stretching in lignin. Additionally, the peak at 1240 cm^{-1} corresponds to C-O stretching vibrations in lignin and hemicelluloses, and the peak at 1030 cm-1 is attributed to C-

O-C stretching vibrations in cellulose and hemicelluloses, indicating the presence of polysaccharides. Together, these peaks draw non-cellulosic elements like hemicelluloses and lignin, which add to the stiffness and natural roughness of the fiber.

On the contrary, the ATLP shows notable changes in their FTIR spectrum that represent eliminating non-cellulosic components. The O-H stretching peak at [3400 – 3300] cm^{-1} becomes slightly narrower and less intense, indicating the reduction of hydrogen bonding due to the removal of hemicelluloses and lignin. The C=O stretching peak at 1730 cm^{-1} diminishes or disappears entirely, confirming the removal of ester groups from hemicelluloses and lignin. Furthermore, the C-O stretching peak at 1240 cm^{-1} decreases in intensity, further supporting that lignin and hemicelluloses have been removed. The C-O stretching peak at 1030 cm^{-1} becomes pronounced, suggesting a relatively higher cellulose concentration following the treatment. The above changes indicate that NaOH treatment effectively removed amorphous components, leading to cellulose-rich fiber with improved surface properties and lesser hydrogen bonding. Thus making them suitable for applications that require strong interfacial adhesion, like composite materials.

The ACLP shows a spectrum between the ULP and ATLP spectrums, reflecting a more moderate level of refinement. The O-H stretching peak at [3400 – 3300] cm^{-1} remains broad but may show slight narrowing compared to ULP, indicating partial removal of hemicelluloses and lignin. The C=O stretching peak at 1730 cm^{-1} is reduced but not eliminated, suggesting that some ester groups remain after the acetic acid treatment. The C-O stretching peak at 1240 cm^{-1} is also reduced but still present, confirming that some lignin and hemicelluloses remain in the fibers. The C-O-C stretching peak at 1030 cm^{-1} remains strong, indicating that cellulose is still the dominant component. These results suggest that the acetic acid treatment partially removes non-cellulosic materials while preserving more of the fiber's natural chemical structure than the NaOH treatment. Due to the above, acetic acid-treated fibers can be viewed as a balanced option because of the moderate refinement without considerable over-processing, which, in turn, may also be a favorable aspect for many applications where some natural fiber characteristics must be preserved.

III.2.2. SEM analysis

The results of SEM Leaflet fibres untreated imagery are presented in the following figures:

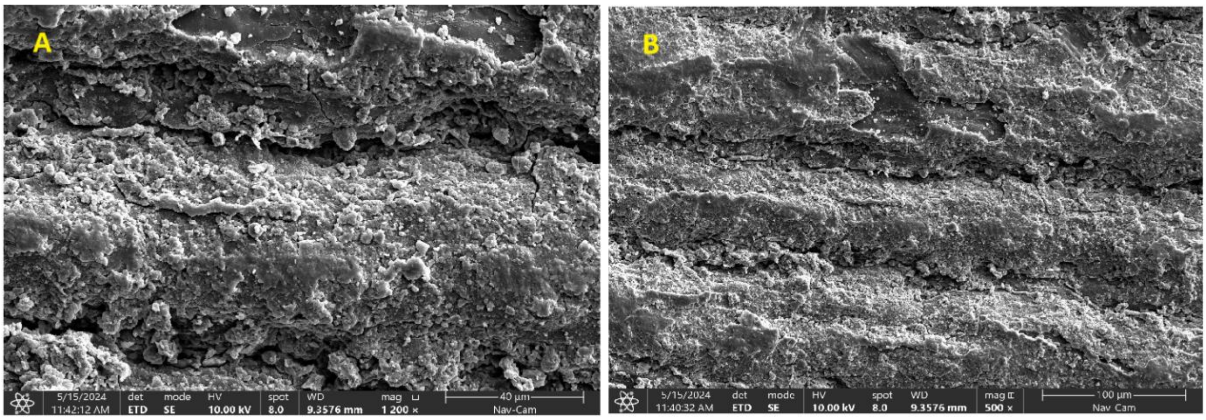

Figure 38 SEM longitudinal images for ULP: A: ×1200, B: ×500

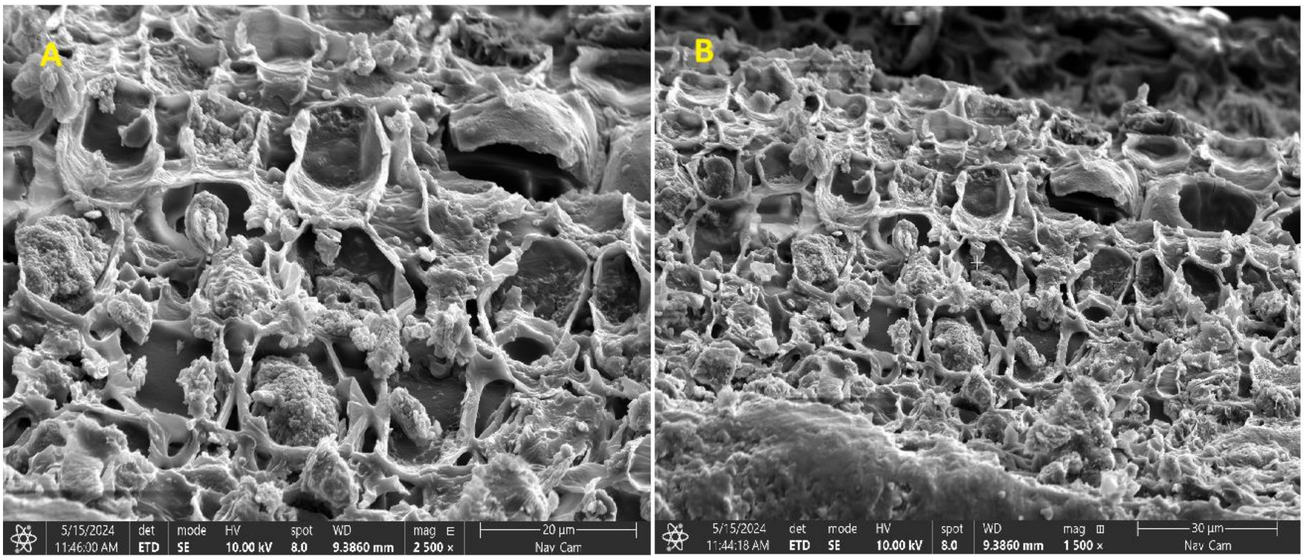

Figure 39 SEM transversal images for ULP: A: ×2500, B: ×1500

The untreated date palm fibers exhibit a natural, unrefined structure characterized by a rough and uneven surface in both longitudinal and transversal views, with visible natural impurities such as waxes, lignin, and hemicelluloses coating the fibers. Fibers in longitudinal view, bundled and agglomerated, with a twisted or ribbon-like organization parallel to the fiber axis, adorned with striations, ridges, and perhaps cracks or grooves due to environmental exposure. In cross-sections, however, fibers appear to have nonuniform shapes and irregular edges, variable wall thicknesses, and pores or hollowness, which adds more evidence of fiber imperfections of a natural kind. Overall, the untreated fibers display limited separation, poor visibility of micro-fibrils, and a textured, porous morphology consistent with non-cellulosic materials and the absence of chemical or mechanical refinement.

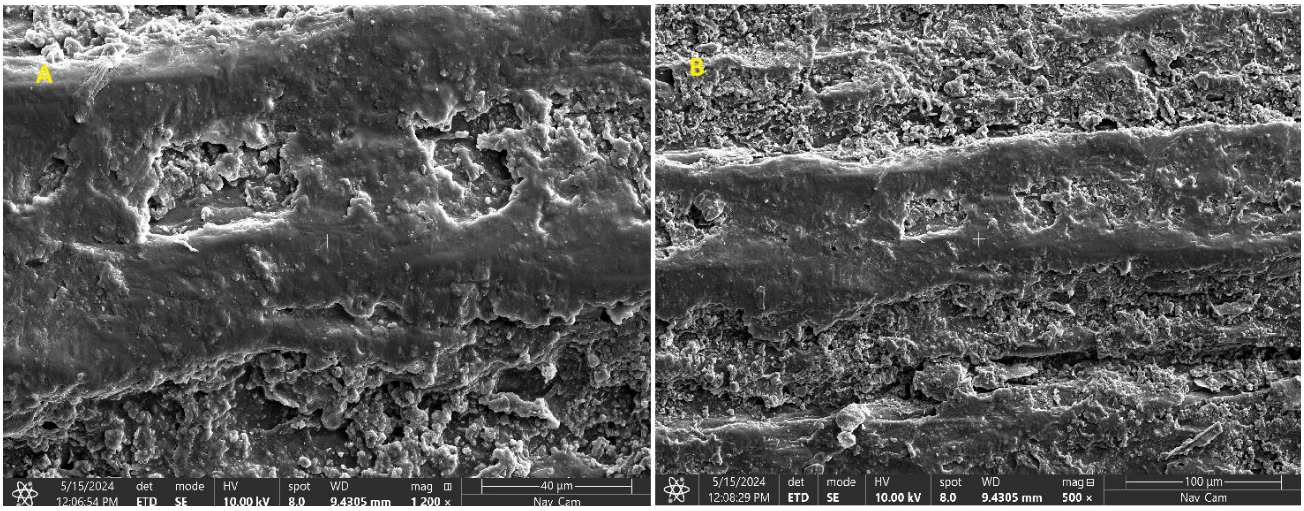

Figure 40 SEM longitudinal images for ATLP: A: ×1200, B: ×500

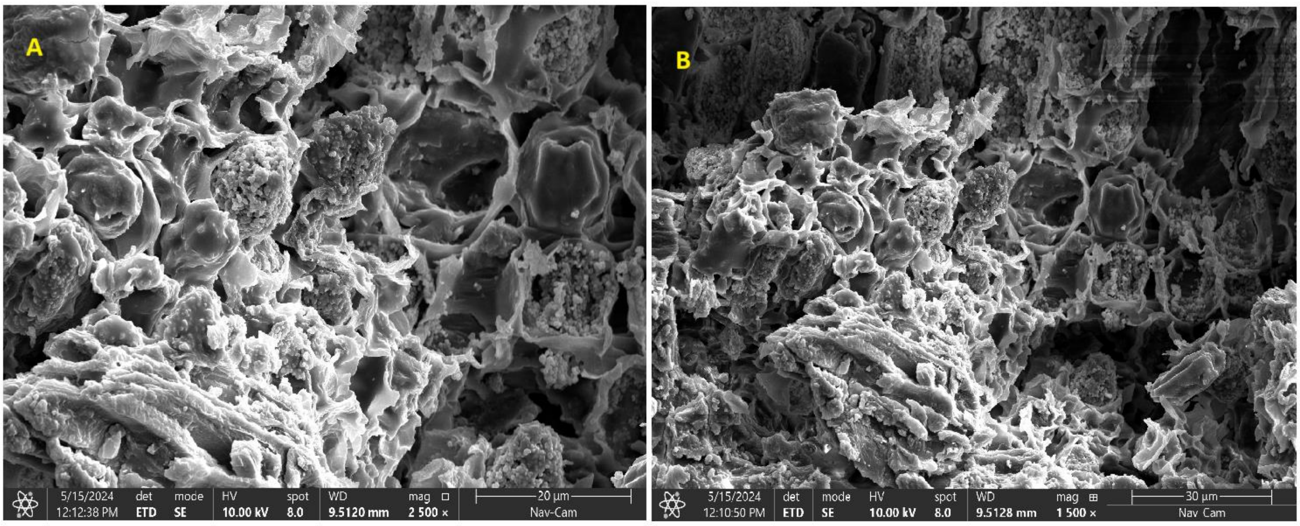

Figure 41 SEM transversal images for ATLP: A: ×2500, B: ×1500

The NaOH-treated date palm fibers exhibit significant morphological and structural changes compared to the untreated fibers, as observed in both longitudinal and transversal SEM images. In the longitudinal view, the fibers display a cleaner and smoother surface due to removing non-cellulosic materials such as waxes, lignin, and hemicelluloses during the NaOH treatment. The surface roughness is notably reduced, and the fibers appear more separated and less bundled, with increased visibility of micro-fibrils and a more defined fibrous structure. The treatment also provided the fiber surface with pores or grooves originating from the dissolution of impurities. The fiber surface area increased, permitting a more significant interfacial area for adhesion in the composites. The cross-section of fibers treated with NaOH displayed a more well-defined and compact structural framework, with fewer irregularities and less porosity, than the untreated fibers. The fiber walls look denser and more uniformly thick, suggesting that the NaOH treatment has mostly removed inner impurities, extremely benefiting the fibre's internal architecture. The NaOH treatment efficiently removed the amorphous hemicelluloses and lignin, acting upon them and thus further enhancing the fibre's rigidity and brittleness. Removing such components provides a cellulose-rich structure, thus augmenting flexibility and mechanical properties.

The treatment also exposes more hydroxyl (-OH) groups on the fiber surface, enhancing its hydrophilicity and potential for chemical bonding with matrices in composite materials. Compared to the untreated fibers, which exhibited a rough, irregular surface with visible cracks, defects, and natural impurities, the NaOH-treated fibers show a more refined and homogeneous morphology. The untreated fibers were characterized by bundled aggregates, limited microfibril visibility, and a porous, nonuniform cross-section.

On the other hand, the NaOH-treated fibers showed superior separation, cleaner surfaces, and a more organized internal structure. These changes demonstrate the efficacy of NaOH treatment in modifying the surface and internal morphology of fiber to suit advanced applications in material engineering better. In general, NaOH treatment helps convert palm date fiber into a uniform and cellulose-rich product with much better surface areas and structural integrity, thus facilitating its use to produce high-performance composites.

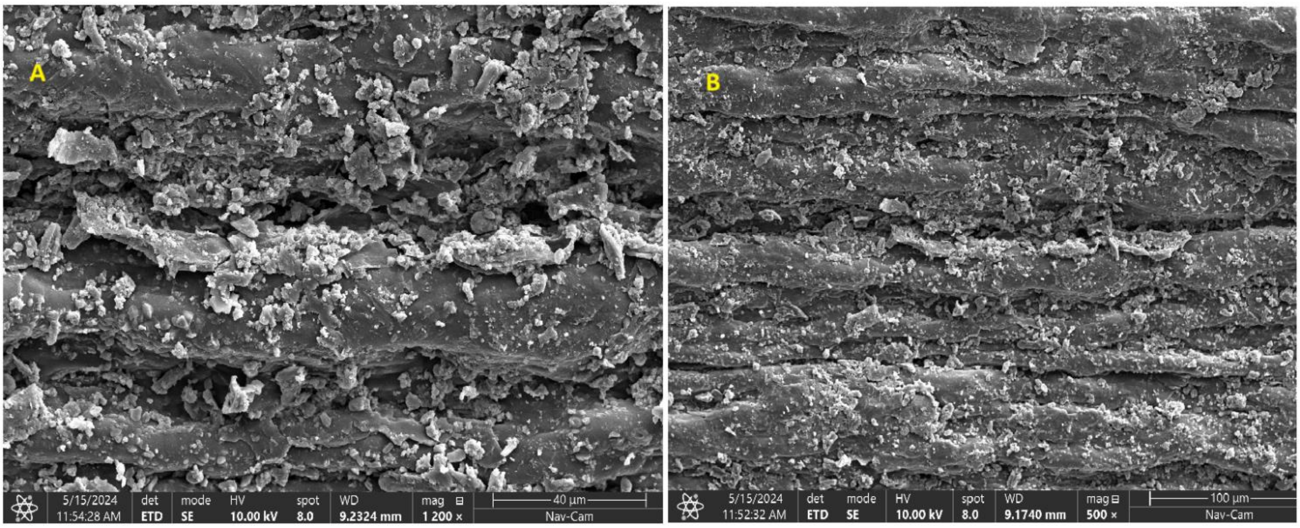

Figure 42 SEM longitudinal images for ACLP: A: ×1200, B: ×500

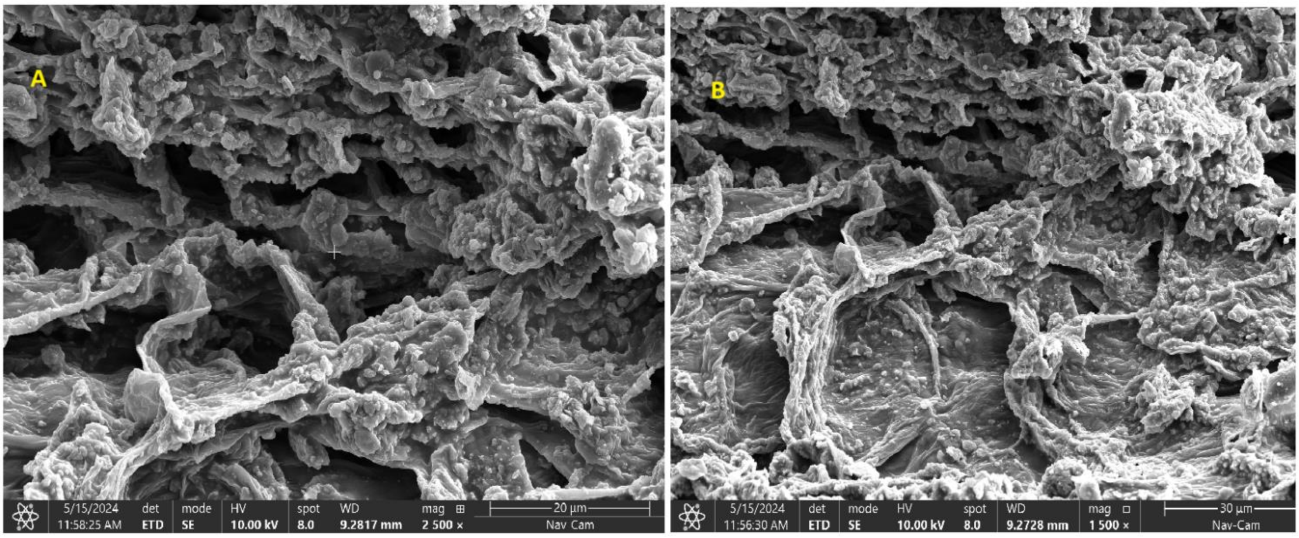

Figure 43 SEM transversal images for ACLP: A: ×2500, B: ×1500

The acetic acid-treated fibers exhibit distinct morphological and structural changes compared to untreated and NaOH-treated fibers, as observed in the longitudinal and transversal SEM images. In the longitudinal view, the fibers display a smoother and cleaner surface than the untreated fibers, though not as refined as the NaOH-treated ones. The acetic acid treatment has effectively neutralized any residual NaOH and removed some surface impurities, resulting in a surface that is less rough but retains some natural texture. The fibers appear more separated and less bundled than the untreated fibers, with moderate visibility of micro-fibrils and a more organized surface structure. However, the surface does not show the pronounced pores or grooves in the NaOH-treated fibers, indicating a gentler chemical action. In the transversal view, the cross-section of the fibers reveals a more uniform and less porous structure than the untreated fibers, though it is not as dense or compact as the NaOH-treated fibers. The thickness of the fiber walls shows slight variation; less deviation suggests that the treatment with acetic acid makes the internal structure of acetic acid more homogeneous without relevant damage or over-etching. Chemically, the treatment with acetic acid has neutralized the alkaline environment driven by the NaOH treatment run earlier, which prevented further degradation of the fiber structure. The fibers achieve a clean surface with this step while retaining mechanical integrity. The treatment has also contributed to the partial removal of residual non-cellulosic materials, though not as extensively as the NaOH treatment. Compared to the untreated fibers, which exhibited a rough, irregular surface with visible cracks, defects, and natural impurities, the acetic acid-treated fibers show a more refined morphology with improved surface smoothness and separation. Nonetheless, the fibers treated with NaOH do not exhibit a pronounced exposure of microfibrils and surface porosity. The untreated fibers comprised bundles with minimal microfibril visibility and a porous/nonuniform cross-section. In contrast, the acetic acid-treated fibers showed moderate levels of refinement with a combination of surface cleanliness and subsistence of structure.

The acetic acid-treated fibers show a less aggressive surface and internal structure modification than the NaOH-treated fiber. The NaOH-treated fibers exhibited a highly cleaned surface with visible pores, grooves, and a dense, compact cross-section due to the extensive removal of non-cellulosic materials.

In contrast, the fibers treated with acetic acid maintain more natural characteristics, resulting in a smoother surface than non-treated fibers and ess altered than fibers exposed to NaOH treatment. Here, acetic acid-treated fibers are the intermediary between fibers treated with NaOH and non-treated fibers, training a confluence between surface refinement and structure retention. In the bottom line, for the acetic acid treatment, the surface attributes and internal structure of the fiber improved without over-processing; hence, it can be used where moderate levels of refinement are desired with the retention of natural integrity for the fiber.

III.2.3. Bulk density

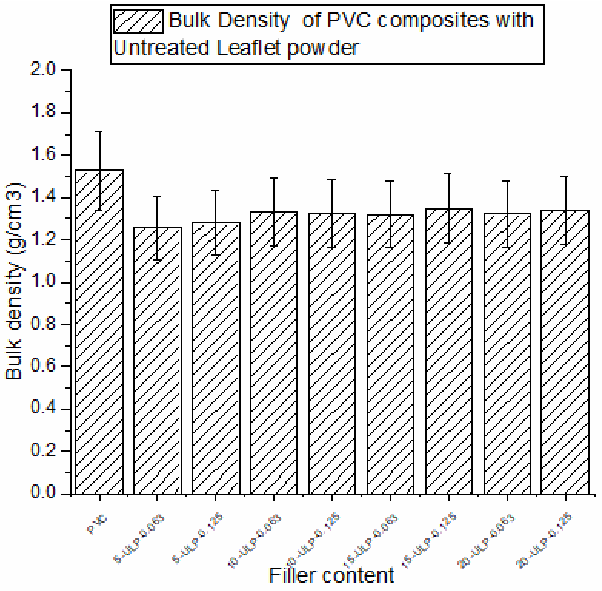

Figure 44 Bulk density results of PVC composites with untreated leflet powder in function of filler content and filler particle size

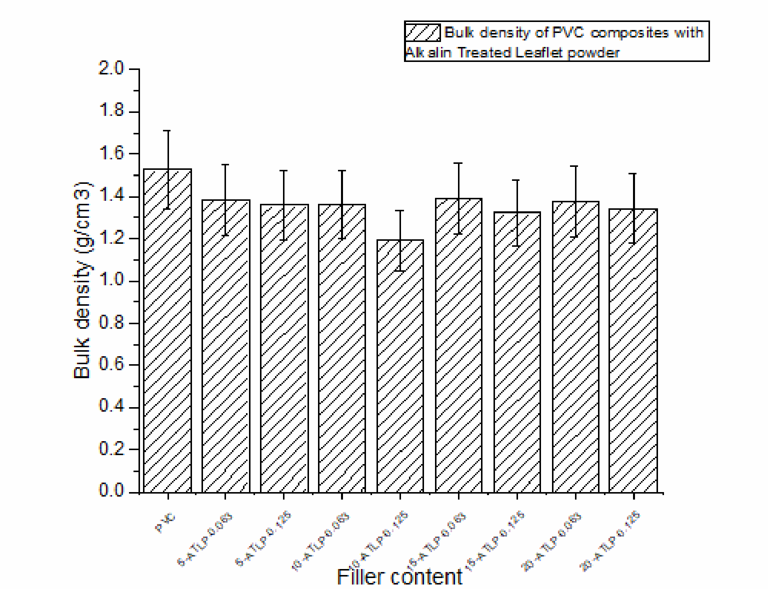

Figure 45 Bulk density results of PVC composites with ATLP in function of filler content and filler particle size

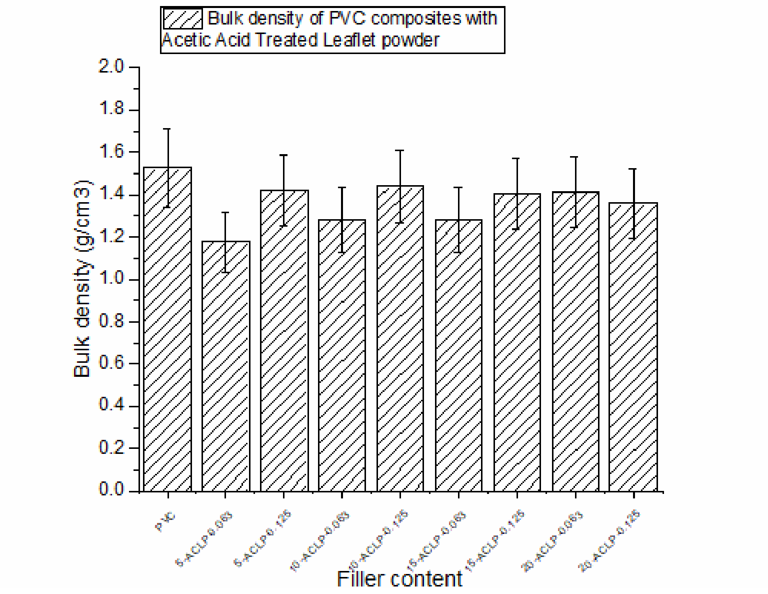

Figure 46 Bulk density results of PVC composites with ACLP in function of filler content and filler particle size

The bulk density result of the composites provides an overview of how the addition of DPLP (both treated and untreated) affects the density of a PVC matrix, where neat PVC bulk density of 1.5265 g/cm3 is the baseline for comparison. This relatively high density is due to the fact that the structure of the polymer is rather homogeneous and dense. When DPLP is added to the PVC matrix, the bulk density changes depending on the type of treatment, particle size, and filler content, reflecting variations in filler-matrix interaction, porosity, and compaction. For ULP composites, the bulk density decreases compared to neat PVC, ranging from 1.2571 to 1.3493 g/cm3, representing a decrease of 11.6% to 17.6%. This reduction is primarily due to the lower density of the natural fibers and the introduction of porosity caused by poor filler-matrix interaction. The untreated powder, which retains its natural impurities such as waxes, lignin, and hemicelluloses, does not bond effectively with the PVC matrix, forming voids and air pockets. The particle size of the DPLP also plays a role in determining the bulk density, with 0.063 mm particles generally exhibiting slightly lower bulk density than 0.125 mm particles, as the finer particles create more voids within the matrix. As the percentage of ULP increases from 5% to 20%, the bulk density slightly increases. In spite of this, however, it remains lower than neat PVC. This probably means that the effect of increased filler content works in compacting the structure; nonetheless, due to the properties of untreated fibers, it has much lower density on the whole.

In contrast, ATLP composites exhibit a different, with bulk density values ranging from 1.1931 to 1.3935 g/cm3, representing a decrease of 8.7% to 21.8% for lower-density composites and an increase of UP to 8.7% for higher-density composites. The alkaline treatment removes non-cellulosic materials such as lignin and hemicelluloses and improves the compatibility between the fibers and the PVC matrix. This enhanced interaction leads to better packing and higher bulk density, particularly at lower filler content. For example, 5% and 10% ATLP composites show bulk densities closer to or slightly higher than neat PVC, indicating effective filler-matrix integration. However, at higher percentages (15% and 20%), the bulk density decreases slightly, likely due to the increased filler content introducing some porosity. The particle size also influences the bulk density of ATLP composites, with 0.063 mm particles generally resulting in higher density than 0.125 mm particles, as the ATLP finer particles integrate more effectively into the PVC matrix, reducing the formation of voids and improving overall compaction.

The acetic acid-treated leaflet powder (ACLP) composites show a more variable trend, with bulk density values ranging from 1.1783 to 1.4387 g/cm3, representing a decrease of 5.8% to 22.8% for lower-density composites and an increase of up to 5.8% for higher-density composites. The acetic acid treatment, which neutralizes residual alkali and partially removes non-cellulosic materials, results in moderate improvements in filler-matrix interaction. At lower filler percentages (5% and 10%), the bulk density of ACLP composites is generally lower than that of neat PVC, suggesting that the treatment does not significantly enhance the integration of the powder into the matrix. However, at higher percentages (15% and 20%), the bulk density increases and approaches or exceeds that of neat PVC, indicating better compaction and interaction at higher filler loadings. The particle size also plays a role, with 0.125 mm particles generally leading to higher bulk density than 0.063 mm particles.

To better understand the impact of treatment type, we compare the bulk density of composites with the same filler content but different treatments. For 5% filler content, the bulk density of 5-ULP-0.063 is 1.2571 g/cm3 (↓ 17.6% from neat PVC), while 5-ATLP-0.063 is 1.3830 g/cm3 (↓ 9.4% from neat PVC), and 5-ACLP-0.063 is 1.1783 (↓ 22.8% from neat PVC). This comparison shows that alkaline treatment significantly improves bulk density due to incomplete removal of impurities. For 10% filler content, the bulk density of 10-ULP-0.063 is 1.3314 g/cm3 (↓ 12.8% from neat PVC), while 10-ATLP-0.063 is 1.3611 g/cm3 (↓ 10.8% from neat PVC), and 10-ACLP-0.063 is 1.2821 g/cm3 (↓ 16.0% from neat PVC). Here, alkaline treatment again shows the highest density, while acetic acid treatment performs better than ULP but still lags behind alkaline treatment. For 20% filler content, the bulk density of 20-ULP-0.125 is 1.3418 g/cm3 (↓ 12.1% from neat PVC), while 20-

ATLP-0.125 is 1.3433 g/cm3 (↓ 12.0% from neat PVC), and 20-ACLP-0.125 is 1.3583 g/cm3 (↓ 11.0% from neat PVC). At higher filler content, the differences between treatments diminish, with all composites showing similar bulk density.

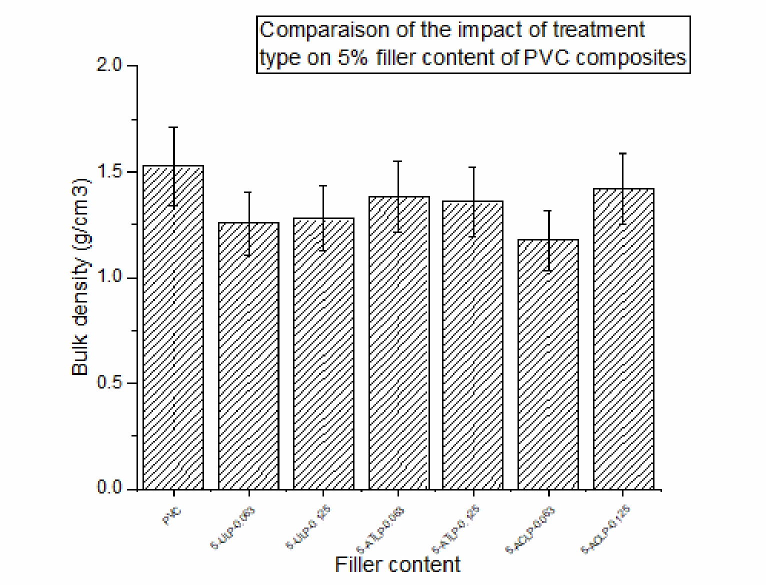

Figure 47 Comparison of the impact of treatment type on 5% filler content of PVC composites Bulk Density

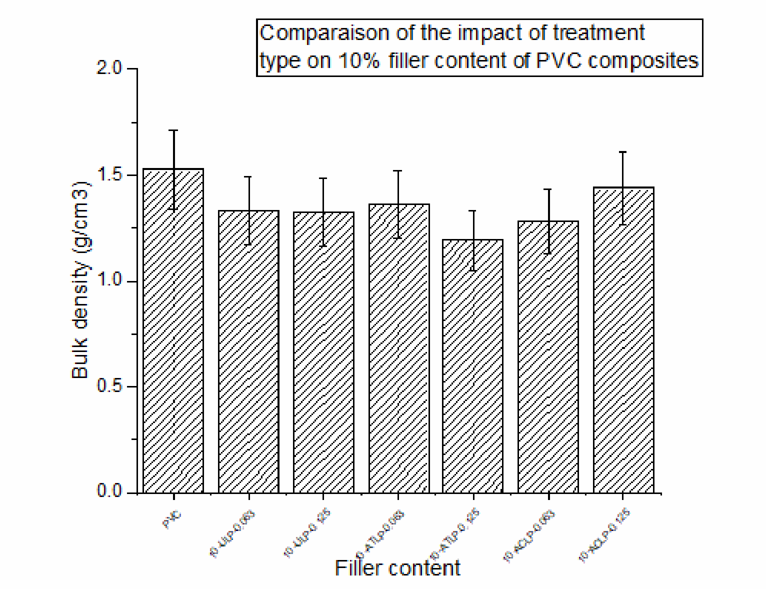

Figure 48 Comparison of the impact of treatment type on 10% filler content of PVC composites Bulk Density

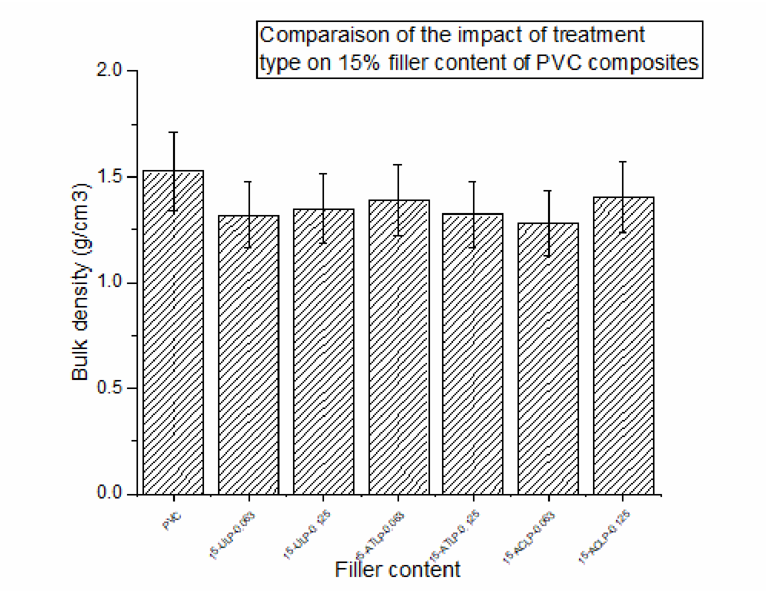

Figure 49 Comparison of the impact of treatment type on 15% filler content of PVC composites Bulk Density

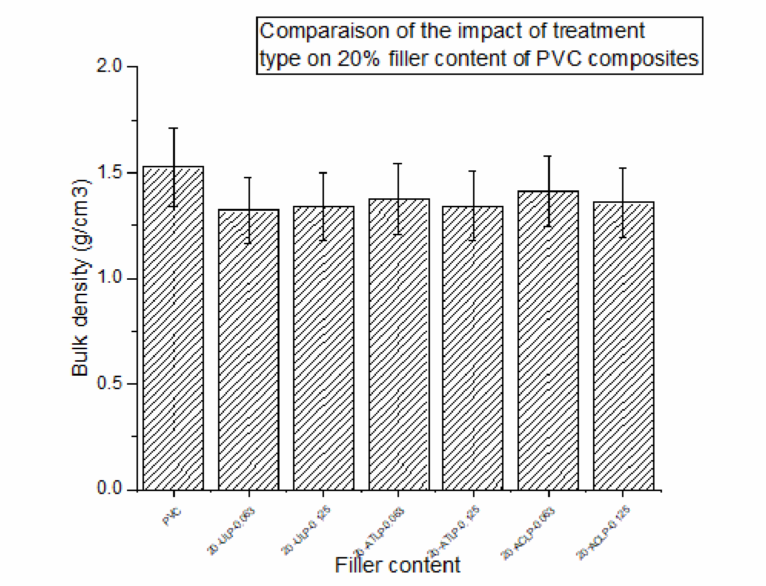

Figure 50 Comparison of the impact of treatment type on 20% filler content of PVC composites Bulk Density

III.2.4. Thermal stability test

The thermal stability results, measured in minutes for neat PVC and its composites, give valuable information about how DPLP addition, along with its treatment, influences the thermal stability properties of the PVC matrix. The thermal stability of neat PVC is taken as the baseline for comparison at 183 minutes or 3 Hours and 03 minutes. When DPLP is added to the PVC matrix, the thermal stability changes depending on the type of treatment, particle size, and filler content, reflecting variations in filler-matrix interaction and thermal properties of the DPLP. For ULP composites, the thermal stability ranges from 120 minutes to 201 minutes (2 Hours to 3 Hours and 21 minutes), showing decreases and increases compared to neat PVC. At lower percentages (5% and 10%), the thermal stability is slightly higher than neat PVC, with 5-ULP-0.125 showing thermal stability of 198 minutes (3 hours and 18 minutes, ↑ 8.2% from neat PVC), and 10-ULP-0.063 showing 186 minutes (3 Hours and 6 minutes, ↑ 1.6% from neat PVC). This suggests that adding ULP can enhance thermal stability at lower percentages due to their natural thermal resistance. However, at higher percentages (15% and 20%), the thermal stability decreases, with 20-ULP-0.063 showing 129 minutes (2 Hours and 9 minutes, ↓ 29.5% from neat PVC), and 20-ULP-0.125 showing 143 minutes (2 Hours and 23 minutes, ↓ 21.9% from neat PVC). Such reduction is primarily due to the use of a filler that has paved the way for thermal degradation. Also, the particle sizes have a considerable effect on this since 0.125mm particles generally have better thermal stability than 0.063mm particles.

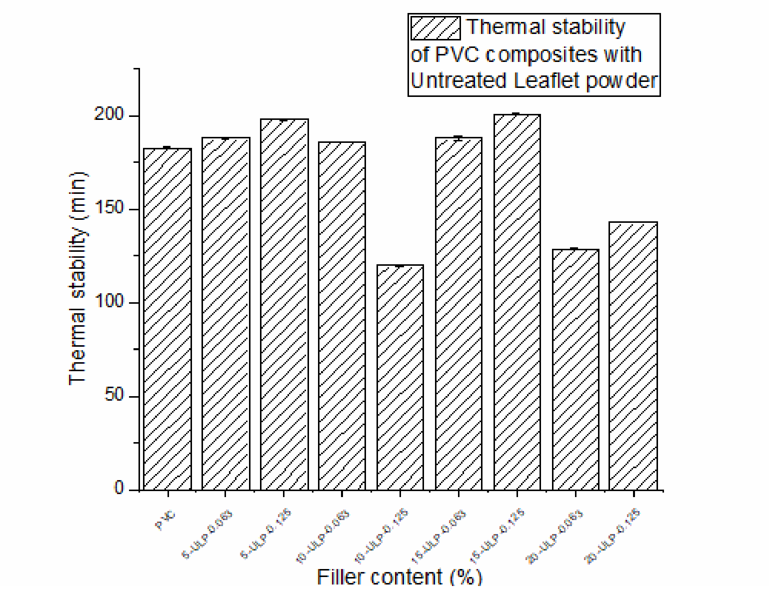

Figure 51 Thermal stability results of PVC composites with ULP in function of filler content and filler particle size

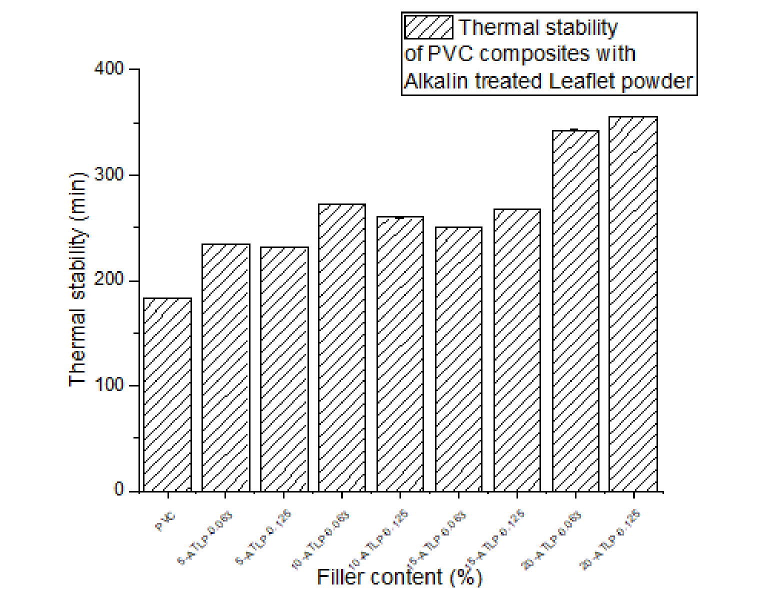

Figure 52 Thermal stability results of PVC composites with ATLP in function of filler content and filler particle size

In contrast, ATLP composites exhibit significant improvements in thermal stability, ranging from 231 minutes to 335 minutes (3 Hours and 51 minutes to 5 Hours and 55 minutes), representing an increase of 26.2% to 94.0% compared to neat PVC. The alkaline treatment removes noncellulosic materials, improving the compatibility of the powder with the PVC matrix. For example, 5-ATLP-0.063 shows a thermal stability of 234 minutes (3 Hours and 54 minutes, ↑ 27.9% from neat PVC), while 20-ATLP-0.125 shows 355 minutes (5 Hours and 55 minutes, ↑ 94.0% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the thermal stability increases significantly, indicating that the alkaline treatment enhances the thermal stability of the composites, particularly at higher filler content. Removing noncellulosic materials like lignin, hemicelluloses, and waxes exposes more of the cellulose of the powder; these exposed cellulose have hydroxyl groups (-OH), which can form hydrogen bonds with the PVC matrix. This increases the fiber and matrix interaction, leading to improved adhesion. The particle size also influences the thermal stability, with 0.125 mm particles generally providing better thermal stability than 0.063 mm particles.

The ACLP composites show the highest thermal stability, ranging from 254 minutes (2 Hours and 14 minutes) to 470 minutes (7 Hours and 50 minutes), representing an increase of 38.8% to 156.8% compared to neat PVC. The acetic acid treatment partially removes noncellulosic materials, improving the thermal stability of the powder and its compatibility with the PVC matrix. For example, 5-ACLP-0.063 shows a thermal stability of 305 minutes (5 Hours and 5 minutes, ↑ 66.7% from neat PVC), while 15-ACLP-0.125 shows 470 minutes (7 Hours and 50 minutes, ↑ 156.8% from neat PVC). As the filler content of ACLP increases (from 5% to 20%), the thermal stability increases significantly, indicating that the acetic acid treatment enhances the thermal stability of the composites, particularly at higher filler percentages. The particle size also plays a role, with 0.125 mm particles generally providing better thermal stability than 0.063mm particles.

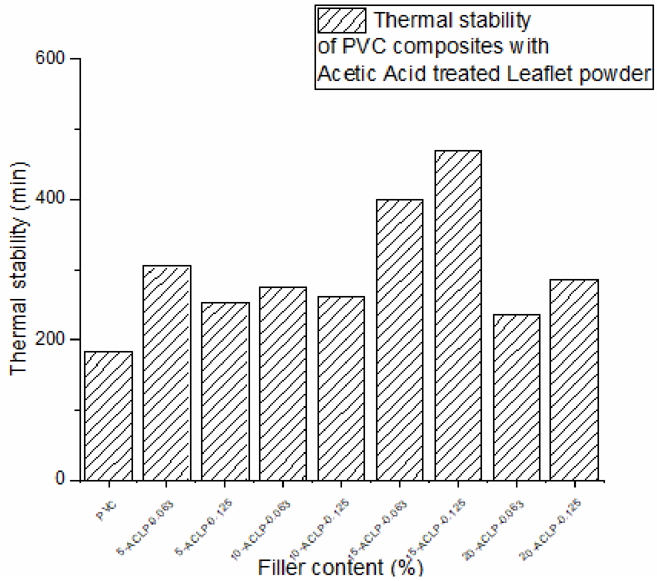

Figure 53 Thermal stability results of PVC composites with ACLP in function of filler content and filler particle size

Thermal stability is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment used. ULP retain their natural impurities, such as waxes, lignin, and hemicelluloses, which hinder adequate bonding with the PVC matrix. This results in poor filler-matrix interaction, forming voids and thermal degradation pathways, particularly at higher filler percentages. The slight improvement in thermal stability at lower percentages can be attributed to the natural thermal resistance of the DPLP. However, this effect diminishes as the filler content increases due to the introduction of defects and weak interfacial bonding.

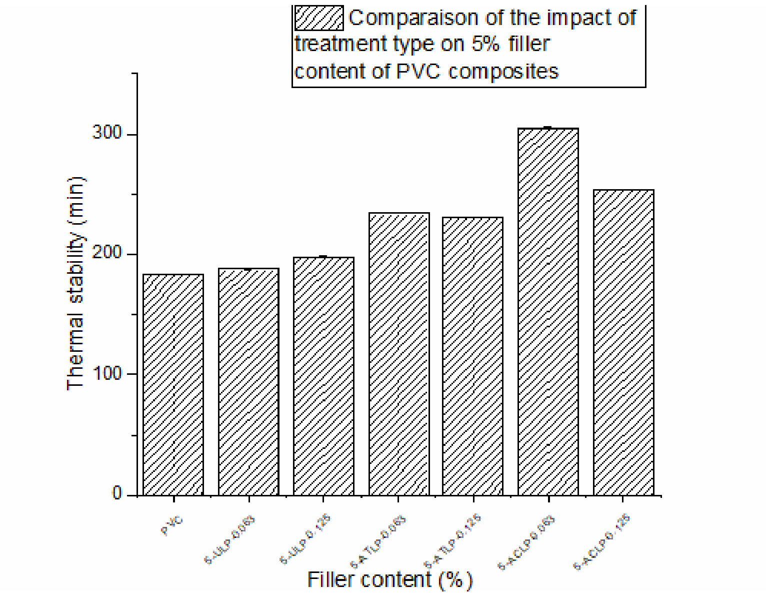

Figure 54 Comparison of the impact of treatment type on 5% filler content of PVC composites Thermal stability

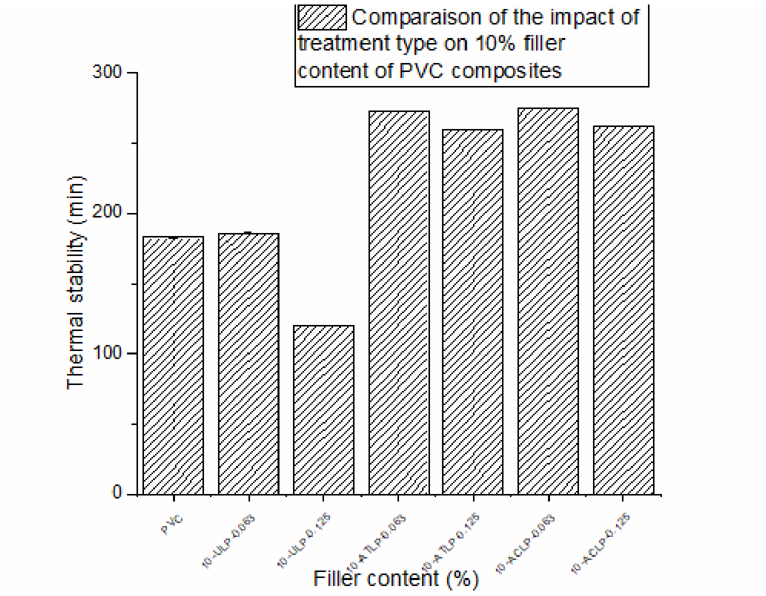

Figure 55 Comparison of the impact of treatment type on 10% filler content of PVC composites Thermal stability

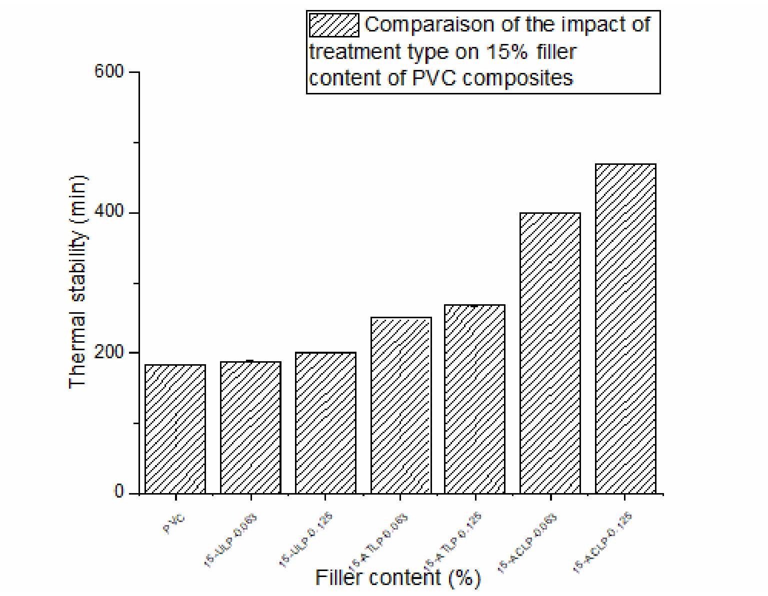

Figure 56 Comparison of the impact of treatment type on 15% filler content of PVC composites Thermal stability

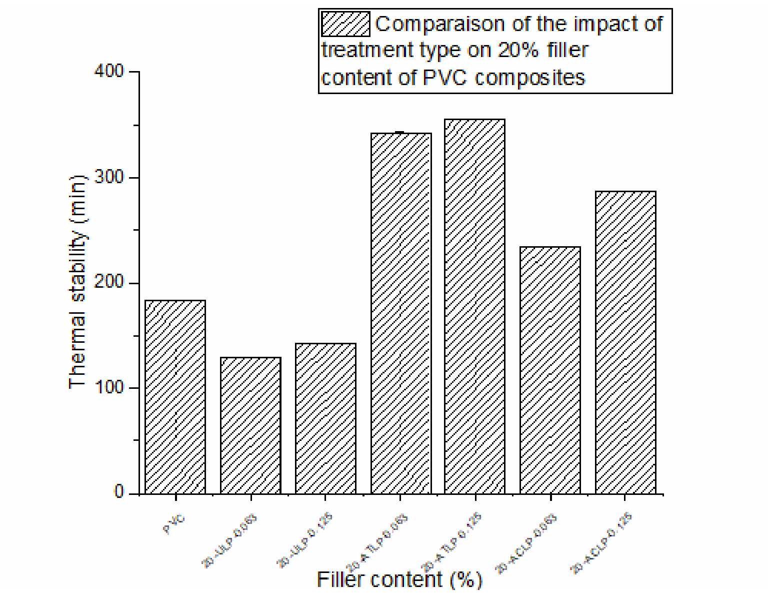

Figure 57 Comparison of the impact of treatment type on 20% filler content of PVC composites Thermal stability

The alkaline treatment enhances considerably the compatibility between the DPLP and the PVC matrix by removing the noncellulosic materials, exposing more cellulose that possesses higher thermal stability and forms much stronger bonds with the PVC matrix. The reduced impurities also minimize void formation thus maximizing the packing of the composite and improving the thermal stability. The increase in thermal stability is particularly pronounced at higher filler percentages, as the ATLP integrates more effectively into the matrix, reducing thermal degradation pathways.

Acetic acid treatment also improves the compatibility between the DPLP and the PVC matrix but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes noncellulosic materials and neutralizes residual alkali, resulting in a cleaner fiber surface with improved thermal stability. However, the effect is less aggressive than alkaline treatment, leading to higher variability in thermal stability. The ACLP shows the highest thermal stability at higher filler content, as the partial removal of impurities and improved filler-matrix interaction reduce thermal degradation pathways.

III.2.5. Shore hardness test

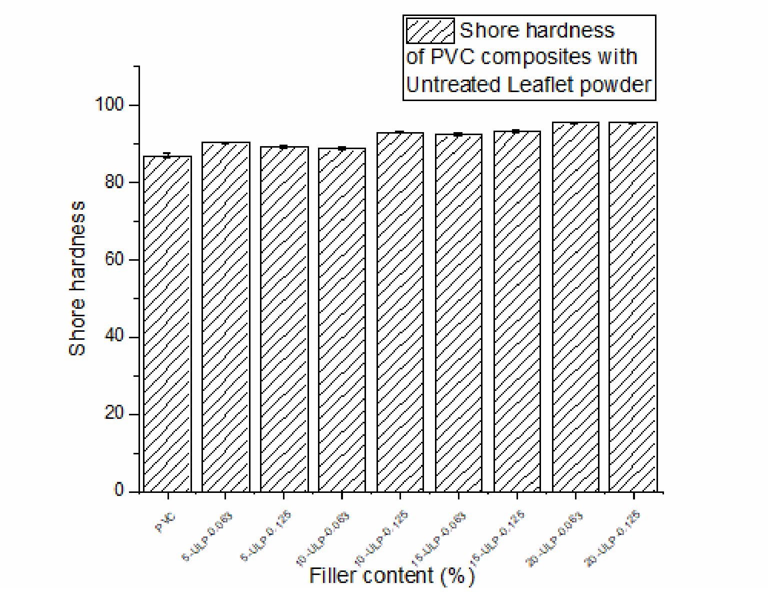

Figure 58 Shore hardness results of PVC composites with ULP in function of filler content and filler particle size

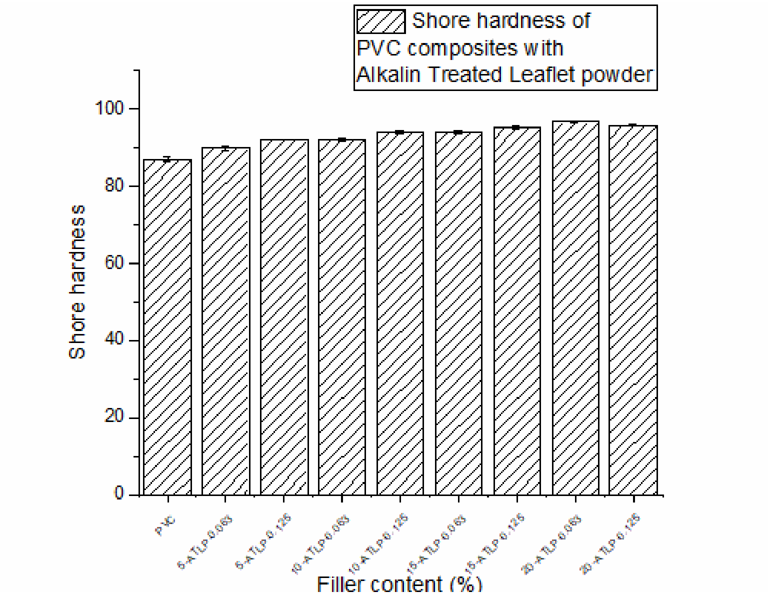

Figure 59 Thermal stability results of PVC composites with ATLP in function of filler content and filler particle size

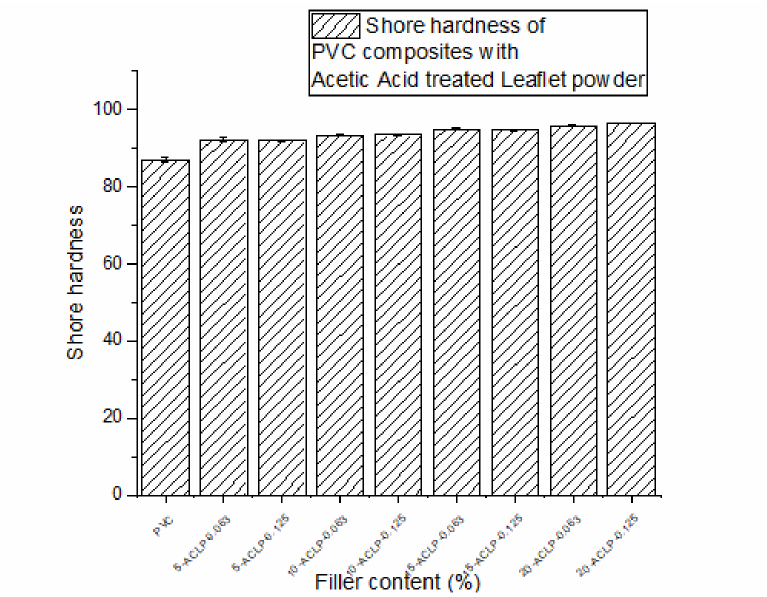

Figure 60 Thermal stability results of PVC composites with ACLP in function of filler content and filler particle size

The shore hardness result, which concerns neat PVC and its composites, elucidates how the inclusion of DLPL and the treatments it has undergone has a bearing on the hardness of the PVC matrix. The shore hardness of neat PVC, which acts as the reference for comparison, is 87.08. When DPLP is added to the PVC matrix, the shore hardness changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction and the mechanical properties of the DPLP.

For ULP composites, the shore hardness ranges from 88.8 to 95.62, showing an increase of 2.0% to 9.8% compared to neat PVC. At lower percentages (5% and 10%), the shore hardness is slightly higher than PVC, with 5-ULP-0.063 showing a shore hardness of 90.36 (↑ 3.8% from neat PVC) and 10-ULP-0.125 showing 93.06 (↑ 6.9% from neat PVC). This suggests that adding ULP can enhance the hardness of the composites due to their natural rigidity. As the percentage of ULP increases (from 15% to 20%), the shore hardness further increases, with 20-ULP-0.063 showing 95.62 (↑ 9.8% from neat PVC) and 20-ULP-0.125 showing 95.54 (↑ 9.7% from neat PVC). This increase is likely due to the higher filler content providing additional reinforcement to the PVC matrix. Likewise, the size of the particles may contribute, whereby 0.125 mm particles usually developed slightly higher hardness than those from the 0.063 mm size range. On the contrary, ATLP composites show considerable increment in shore hardness with values from 90.0 to 96.74, which represents an increment of 3.4% to 11.1% over neat PVC. The alkaline treatment removes non-cellulosic materials, improving the DPLP and PVC matrix compatibility. For example, 5-ATLP-0.063 shows a shore hardness of 90.0 (↑ 3.4% from neat PVC), while 20-ATLP-0.063 shows 96.74 (↑ 11.1% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the shore hardness increases significantly, indicating that the alkaline treatment enhances the hardness of the composites, particularly at higher filler percentages. The particle size also influences the shore hardness, with 0.125 mm particles generally providing slightly higher hardness than 0.063mm particles.

The ACLP composites show the highest shore hardness, ranging from 92.0 to 96.54, representing an increase of 5.6% to 10.9% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the DPLP and PVC matrix compatibility. For example, 5-ACLP-0.063 shows a shore hardness of 92.26 (↑ 6.0% from neat PVC), while 20-ACLP-0.125 shows 96.54 (↑ 10.9% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the shore hardness increases significantly, indicating that the acetic acid treatment enhances the hardness of the composites, particularly at higher filler percentages. The particle size also plays a role, with 0.125 mm particles generally providing slightly higher hardness than 0.063mm particles.

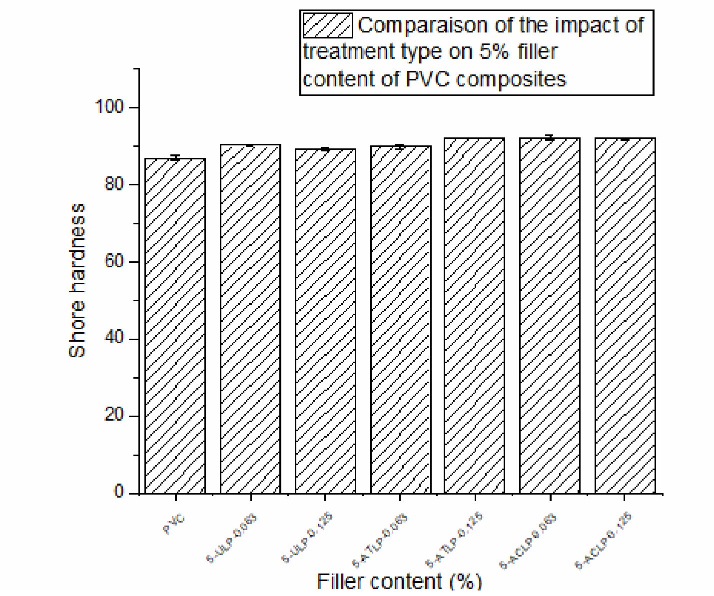

Figure 61 Comparison of the impact of treatment type on 5% filler content of PVC composites Shore hardness

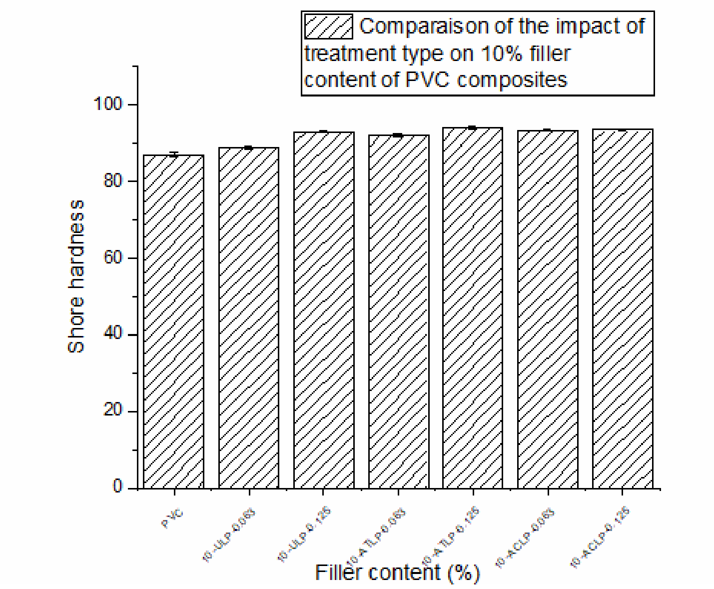

Figure 62 Comparison of the impact of treatment type on 10% filler content of PVC composites Shore hardness

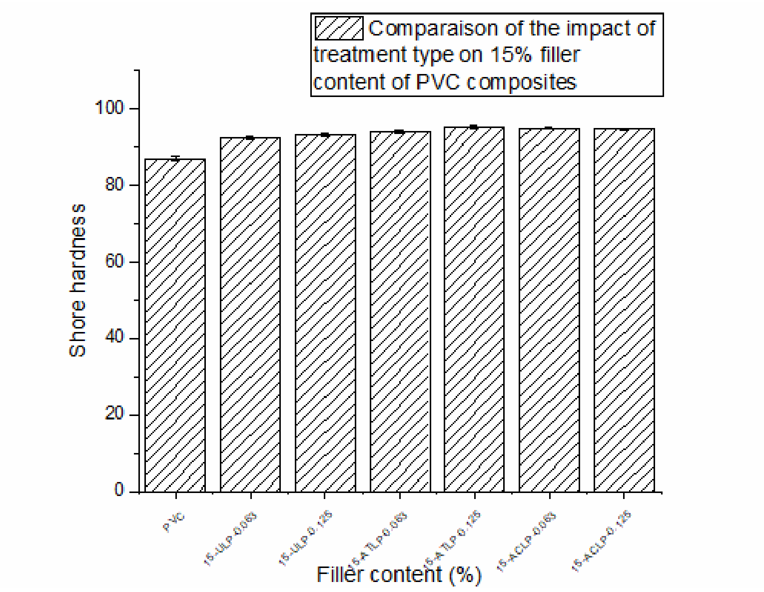

Figure 63 Comparison of the impact of treatment type on 15% filler content of PVC composites Shore hardness

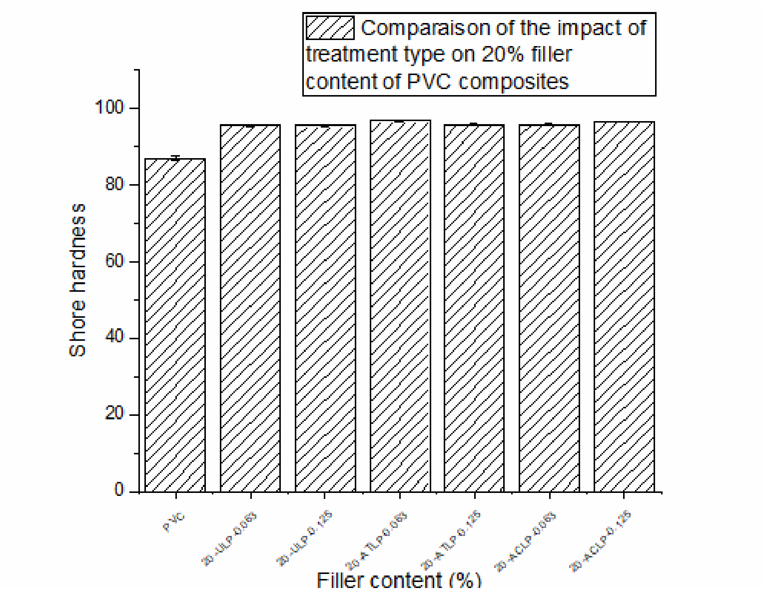

Figure 64 Comparison of the impact of treatment type on 20% filler content of PVC composites Shore hardness

To better understand the impact of the treatment type, we compare composites' shore hardness with the same filler content but with different treatments. For 5% filler content, 5-ULP-0.063 shows a shore hardness of 90.36 (↑ 3.8% from neat PVC), while 5-ATLP-0.063 shows 90.0 (↑ 2.4% from neat PVC), and 5-ACLP-0.063 shows 92.26 (↑ 6.0% from neat PVC). This comparison shows that the acetic acid treatment provides the highest shore hardness, followed by ULP, while the alkaline treatment slightly improves. For 10% filler content, 10-ULP-0.125 shows a shore hardness of 93.06 (↑ 6.9% from neat PVC), while 10-ATLP-0.125 shows 94.2 (↑ 8.2% from neat PVC), and 10-ACLP-0.125 shows 93.58 (↑ 7.5% from neat PVC). Here, alkaline treatment provides the highest shore hardness, followed by acetic acid treatment and ULP. For 20% filler content, 20-ULP-0.063 shows a Shore hardness of 95.62 (↑9.8% from neat PVC), while 20-ATLP-0.063 shows 96.74 (↑11.1% from neat PVC), and 20-ACLP-0.125 shows 96.54 (↑10.9% from neat PVC). At higher filler percentages, alkaline treatment provides the highest Shore hardness, while acetic acid treatment also significantly improves.

The shore hardness of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. ULP retain their natural impurities, which hinder adequate bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. However, the natural rigidity of the fibers can still enhance the hardness of the composites, particularly at higher filler content, where the increased filler content provides additional reinforcement to the PVC matrix. The slight improvement in Shore hardness at lower percentages can be attributed to the natural rigidity of the fibers, but the poor interfacial bonding limits this effect.

Alkaline treatment significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with PVC. The increase in shore hardness is particularly pronounced at higher filler percentages, as the ATLP integrates more effectively into the matrix, providing additional reinforcement and reducing weak interfacial bonding.

Acetic acid treatment also improves the compatibility between the fibers and the PVC matrix but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes alkali residuals, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in shore hardness.

III.2.6. Tensile test

Before start analysing the obtained results from the tensile test, the following figure shows the mechanical behaviour of the five specimen of neat PVC under tensile test.

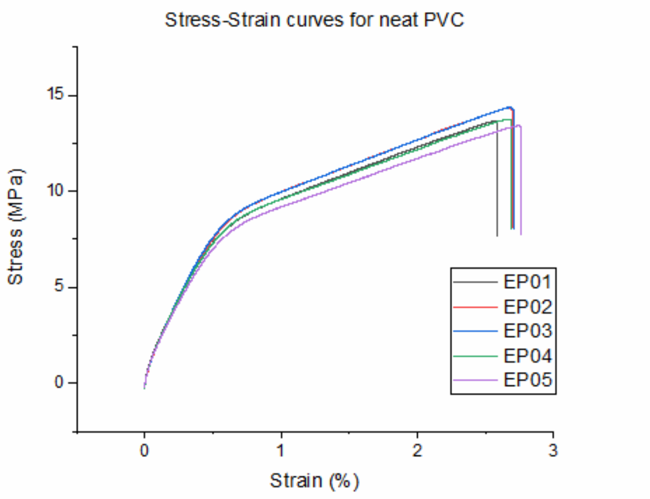

Figure 65 Stress-Strain curves of the five neat PVC specimens

a) ***Young's modulus***

Young's modulus values of neat PVC and their composites offer helpful analysis of how the stiffness of the PVC matrix is affected by the treatment (untreated, alkaline-treated, or acetic acid-treated) inclusion of date palm leaflet powder (DPLP). The Young's modulus of neat PVC is 39.718 MPa, serving as the baseline for comparison. When DPLP is added to the PVC matrix, Young's modulus changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction and the mechanical properties of the fibers. For ULP composites, Young's modulus ranges from 39.14 MPa to 174.47 MPa, showing an increase of up to 339.2% compared to neat PVC. At lower percentages (5% and 10%), the Young's modulus is slightly higher than neat PVC, with 5-ULP-0.125 showing a Young's modulus of 49.83 MPa (↑25.5% from neat PVC) and 10-ULP-0.125 showing 88.08 MPa (↑121.8% from neat PVC). This suggests that the addition of untreated fibers can enhance the stiffness of the composites due to their natural rigidity. As the percentage of ULP increases (from 15% to 20%), Young's modulus further increases, with 20-ULP-0.063 showing 174.47 MPa (↑339.2% from neat PVC) and 20-ULP-0.125 showing 134.92 MPa (↑239.7% from neat PVC). This increase is likely due to the higher filler content providing additional reinforcement to the PVC matrix. The particle size also plays a role, with 0.063 mm particles generally providing higher stiffness than 0.125 mm particles.

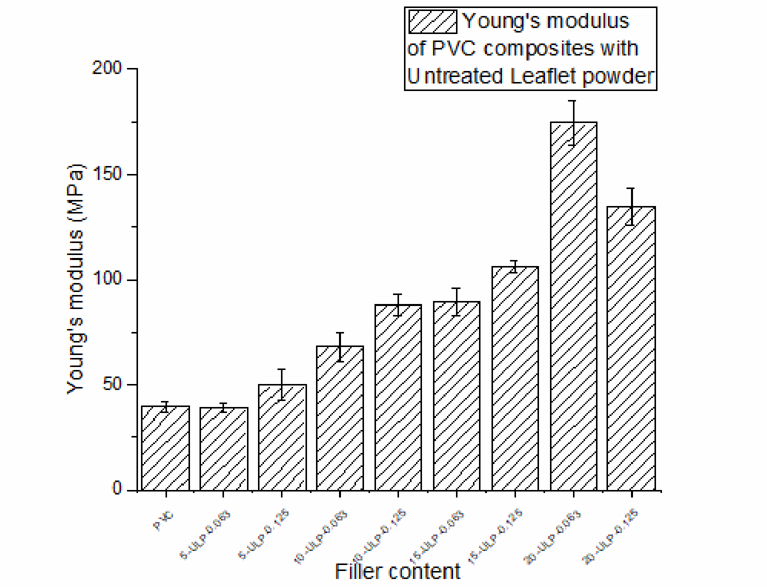

Figure 66 Young's Modulus results of PVC composites with ULP in function of filler content and filler particle size

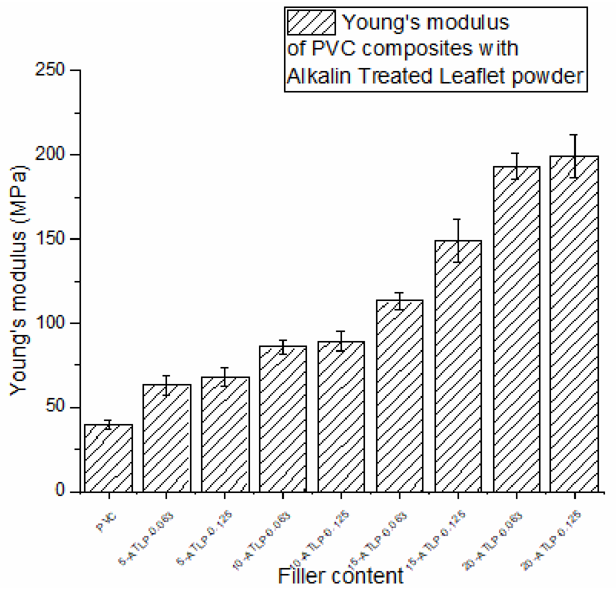

Figure 67 Young’s Modulus results of PVC composites with ATLP in function of filler content and filler particle size

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit significant improvements in Young's modulus, ranging from 63.28 MPa to 199.38 MPa, representing an increase of 59.3% to 402.0% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows Young's modulus of 63.28 MPa (↑59.3% from neat PVC), while 20-ATLP-0.125 shows 199.38 MPa (↑402.0% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), Young's modulus increases significantly, indicating that the alkaline treatment enhances the stiffness of the composites, particularly at higher filler percentages. The particle size also influences Young's modulus, with 0.125 mm particles generally providing slightly higher stiffness than 0.063 mm particles.

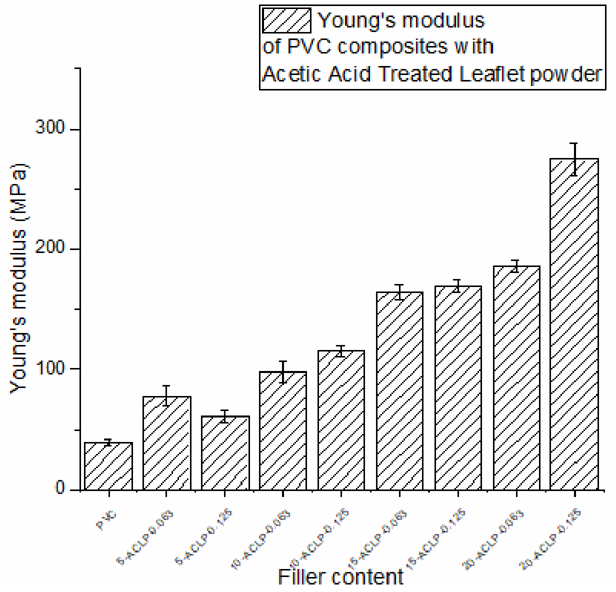

Figure 68 Young’s Modulus results of PVC composites with ACLP in function of filler content and filler particle size

The ACLP composites show the highest Young's modulus, ranging from 60.67 MPa to 274.74 MPa, representing an increase of 52.8% to 591.7% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.063 shows a Young's modulus of 78.14 MPa (↑96.8% from neat PVC), while 20-ACLP-0.125 shows 274.74 MPa (↑591.7% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), Young's modulus increases significantly, indicating that the acetic acid treatment enhances the stiffness of the composites, particularly at higher filler percentages. The particle size also plays a role, with 0.125 mm particles generally providing higher stiffness than 0.063 mm particles.

<u>Comparison of Treatment Effects at Fixed Filler Content</u>

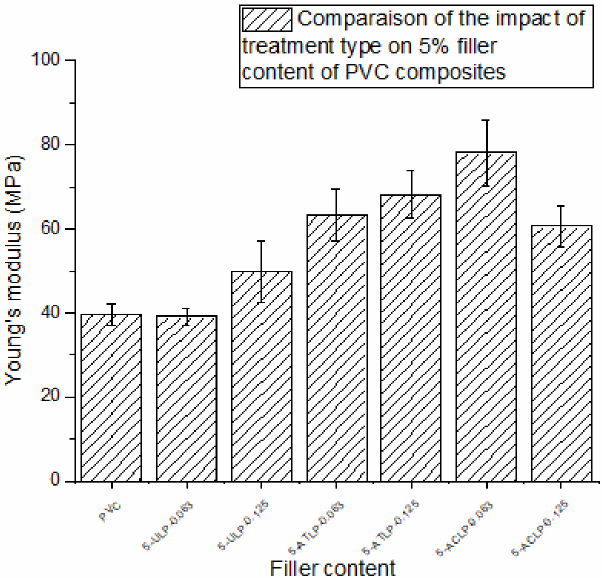

Figure 69 Comparison of the impact of treatment type on 5% filler content of PVC composites Young's Modulus

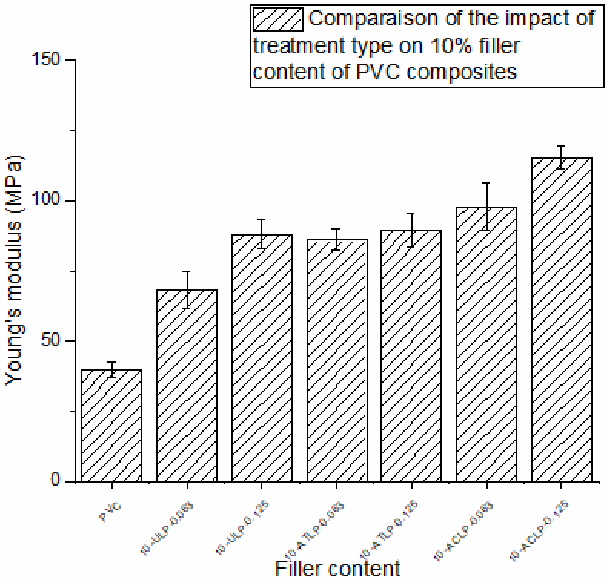

Figure 70 Comparison of the impact of treatment type on 10% filler content of PVC composites Young's Modulus

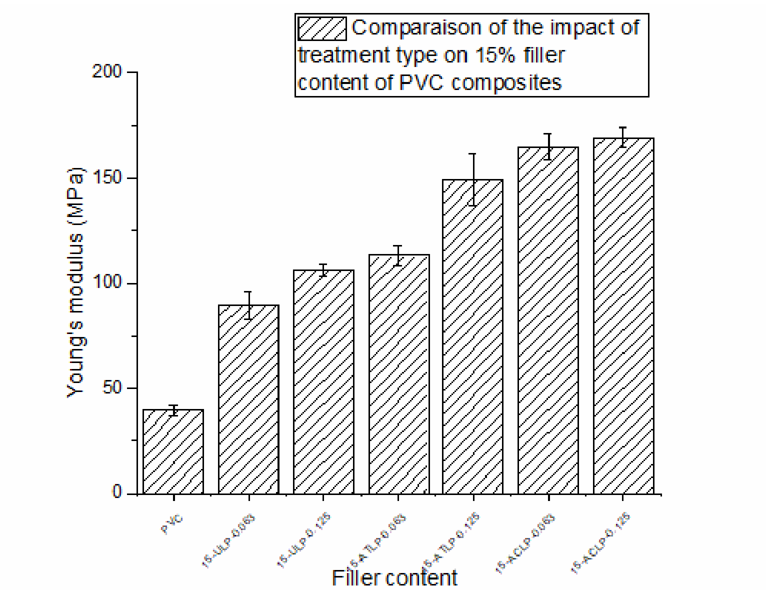

Figure 71 Comparison of the impact of treatment type on 15% filler content of PVC composites Young’s Modulus

To better understand the impact of treatment type, we compare Young's modulus of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.125 shows a Young's modulus of 49.83 MPa (↑25.5% from neat PVC), while 5-ATLP-0.063 shows 63.28 MPa (↑59.3% from neat PVC), and 5-ACLP-0.063 shows 78.14 MPa (↑96.8% from neat PVC). This comparison shows that acetic acid treatment provides the highest Young's modulus, followed by alkaline treatment, while untreated fibers show the most minor improvement. For 10% filler content, 10-ULP-0.125 shows a Young's modulus of 88.08 MPa (↑121.8% from neat PVC), while 10-ATLP-0.125 shows 89.43 MPa (↑125.2% from neat PVC), and 10-ACLP-0.125 shows 115.38 MPa (↑190.5% from neat PVC). Here, acetic acid treatment provides the highest Young's modulus, followed by alkaline treatment and untreated fibers. For 20% filler content, 20-ULP-0.063 shows a Young's modulus of 174.47 MPa (↑339.2% from neat PVC), while 20-ATLP-0.125 shows 199.38 MPa (↑402.0% from neat PVC), and 20-ACLP-0.125 shows 274.74 MPa (↑591.7% from neat PVC). At higher filler percentages, acetic acid treatment provides the highest Young's modulus, while alkaline treatment also significantly improves.

Young's modulus of composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment used. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicelluloses, which hinder adequate bonding with the PVC matrix. This leads to weak interfacial bonding and void formation due to poor filler-matrix interaction. Nonetheless, the inherent stiffness of the fibers can still improve the composites' stiffness, especially when the filler fraction is higher and the PVC matrix is further reinforced. The slight improvement in Young's modulus at lower percentages can be attributed to the natural rigidity of the fibers, but the poor interfacial bonding limits this effect.

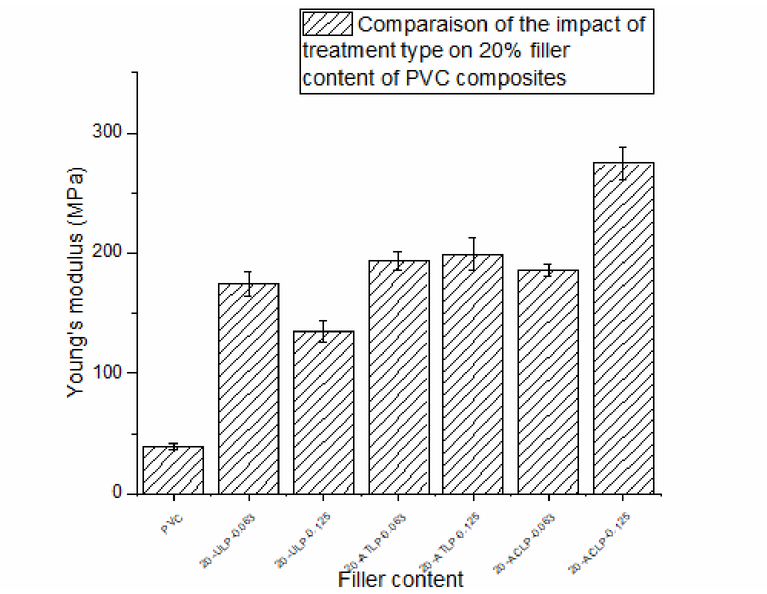

Figure 72 Comparison of the impact of treatment type on 20% filler content of PVC composites Young's Modulus

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicelluloses. More cellulose, which has superior mechanical qualities and creates stronger linkages with the PVC matrix, is exposed as a result. Eliminating impurities also improves the composite's overall packing and decreases void formation, which raises the composite's Young's modulus. The increase in Young's modulus is particularly pronounced at higher filler percentages, as the alkaline-treated fibers integrate more effectively into the matrix, providing additional reinforcement and reducing weak interfacial bonding.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix, but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than the alkaline treatment, leading to higher variability in Young's modulus. The acetic acid-treated fibers show the highest Young's modulus at higher filler percentages, as the partial removal of impurities and improved filler-matrix interaction enhance the reinforcement of the PVC matrix.

b) ***Ultimate stress***

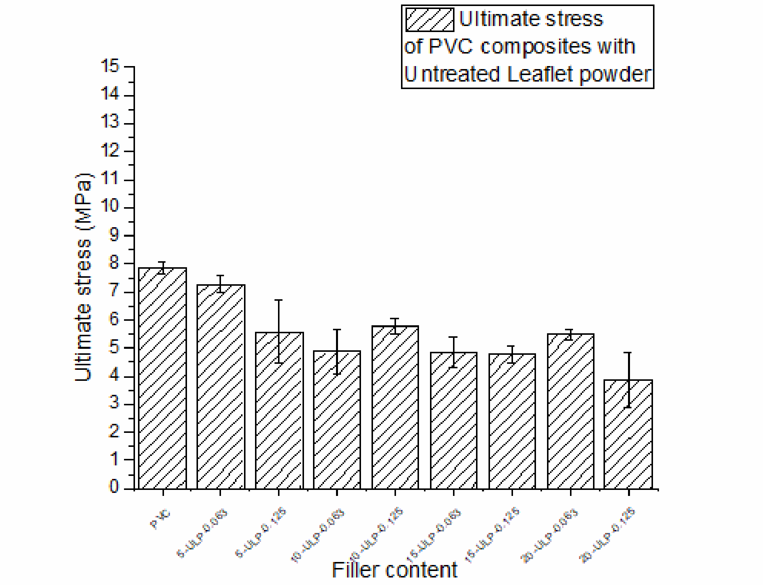

Figure 73 Ultimate stress results of PVC composites with ULP in function of filler content and filler particle size

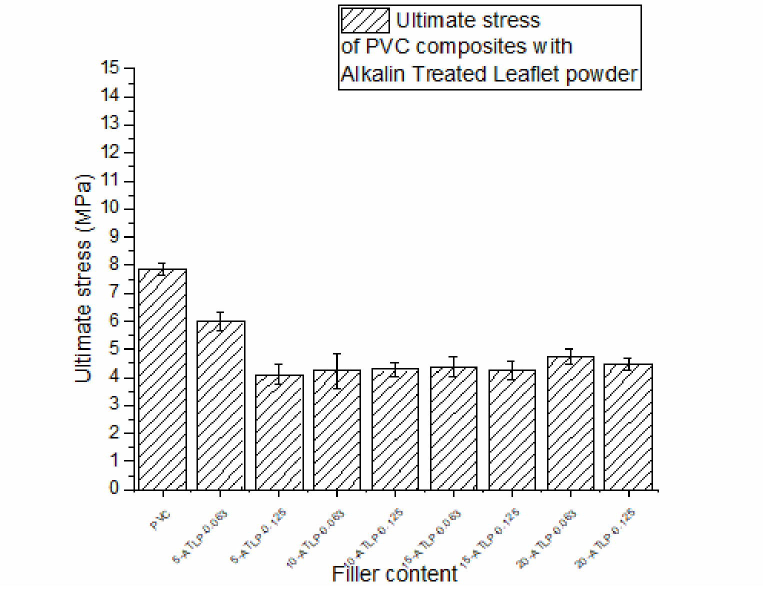

Figure 74 Ultimate stress results of PVC composites with ATLP in function of filler content and filler particle size

The ultimate strength analysis of neat PVC and its composites offers a detailed comprehension of how the types of date palm leaflet powder (DPLP) and its treatments contribute to the mechanical strength, chemical composition, and morphological properties of the PVC matrix, whether untreated, alkaline, or acetic acid-treated. The ultimate stress of neat PVC is 7.861 MPa, serving as the baseline for comparison. When DPLP is added to the PVC matrix, the ultimate stress changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology. For untreated leaflet powder (ULP) composites, the ultimate stress ranges from 3.865 MPa to 7.275 MPa, decreasing from 7.4% to 50.8% compared to neat PVC. At lower percentages (5% and 10%), the ultimate stress is slightly lower than neat PVC, with 5-ULP-0.063 showing an ultimate stress of 7.275 MPa (↓7.4% from neat PVC) and 10-ULP-0.125 showing 5.766 MPa (↓26.6% from neat PVC). This suggests that the addition of untreated fibers can reduce the ultimate stress of the composites due to poor filler-matrix interaction. As the percentage of ULP increases (from 15% to 20%), the ultimate stress further decreases, with 20-ULP-0.063 showing 5.504 MPa (↓30.0% from neat PVC) and 20-ULP-0.125 showing 3.865 MPa (↓50.8% from neat PVC). This decrease is likely due to the higher filler content introducing defects and weak interfacial bonding. The particle size also plays a role, with 0.063 mm particles generally providing slightly higher ultimate stress than 0.125 mm particles.

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit a decrease in ultimate stress, ranging from 4.113 MPa to 5.996 MPa, representing a decrease of 23.7% to 47.7% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows an ultimate stress of 5.996 MPa (↓23.7% from neat PVC), while 20-ATLP-0.125 shows 4.468 MPa (↓43.2% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the ultimate stress decreases, indicating that the alkaline treatment does not significantly enhance the ultimate stress of the composites. The particle size also influences the ultimate stress, with 0.063 mm particles generally providing slightly higher ultimate stress than 0.125 mm particles.

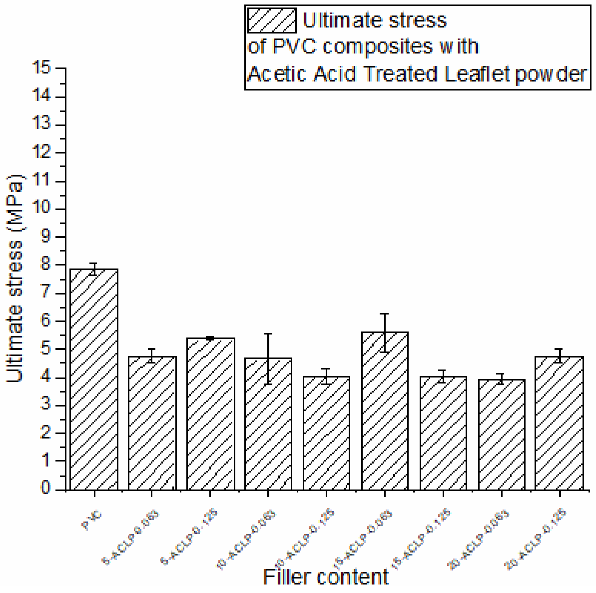

Figure 75 Ultimate stress results of PVC composites with ACLP in function of filler content and filler particle size

The acetic acid-treated leaflet powder (ACLP) composites show a decrease in ultimate stress, ranging from 3.946 MPa to 5.608 MPa, representing a decrease of 28.6% to 49.8% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.125 shows an ultimate stress of 5.380 MPa (↓31.6% from neat PVC), while 20-ACLP-0.063 shows 3.946 MPa (↓49.8% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the ultimate stress decreases, indicating that the acetic acid treatment does not significantly enhance the ultimate stress of the composites. The particle size also plays a role, with 0.125 mm particles generally providing slightly higher ultimate stress than 0.063 mm particles.

Comparison of Treatment Effects at Fixed Filler Content

To better understand the impact of treatment type, we compare the ultimate stress of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.063 shows an ultimate stress of 7.275 MPa (↓7.4% from neat PVC), while 5-ATLP-0.063 shows 5.996 MPa (↓23.7% from neat PVC), and 5-ACLP-0.063 shows 4.758 MPa (↓39.5% from neat PVC). This comparison shows that untreated fibers provide the highest ultimate stress, followed by alkaline treatment, while acetic acid treatment shows the lowest ultimate stress. For 10% filler content, 10-ULP-0.125 shows an ultimate stress of 5.766 MPa (↓26.6% from neat PVC), while 10-ATLP-0.125 shows 4.283 MPa (↓45.5% from neat PVC), and 10-ACLP-0.125 shows 4.030 MPa (↓48.7% from neat PVC). Here, untreated fibers provide the highest ultimate stress, followed by alkaline and acetic acid treatments. For 20% filler content, 20-ULP-0.063 shows an ultimate stress of 5.504 MPa (↓30.0% from neat PVC), while 20-ATLP-0.125 shows 4.468 MPa (↓43.2% from neat PVC), and 20-ACLP-0.125 shows 4.754 MPa (↓39.5% from neat PVC). At higher filler percentages, untreated fibers provide the highest ultimate stress, followed by acetic acid and alkaline treatments.

Comparison of Filler Content and Size Effects at Fixed Treatment Type

To better understand the impact of filler content and size, we compare the ultimate stress of composites with the same treatment but different filler content and size. For untreated fibers (ULP), the ultimate stress decreases with filler content, with 5-ULP-0.063 showing 7.275 MPa (↓7.4% from neat PVC) and 20-ULP-0.125 showing 3.865 MPa (↓50.8% from neat PVC). The particle size also plays a role, with 0.063 mm particles generally providing slightly higher ultimate stress than 0.125 mm particles. For alkaline-treated fibers (ATLP), the ultimate stress decreases with filler content, with 5-ATLP-0.063 showing 5.996 MPa (↓23.7% from neat PVC) and 20-ATLP-0.125 showing 4.468 MPa (↓43.2% from neat PVC). The particle size also influences the ultimate stress, with 0.063 mm particles generally providing slightly higher ultimate stress than 0.125 mm particles. For acetic acid-treated fibers (ACLP), the ultimate stress decreases with filler content, with 5-ACLP-0.125 showing 5.380 MPa (↓31.6% from neat PVC) and 20-ACLP-0.063 showing 3.946 MPa (↓49.8% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing slightly higher ultimate stress than 0.063 mm particles.

The ultimate stress of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicelluloses, which hinder adequate bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows firm peaks for hemicelluloses, lignin, and other non-cellulosic materials, contributing to poor compatibility with the PVC matrix. The SEM images display the rough, uneven surface and visible impurities, reinforcing the bad interaction between the filler and the matrix. Untreated fibers, however, afforded the greatest ultimate stress among the treated fibers; this can be attributed to their inherent stiffness and the lack of chemical degradation that could have weakened them.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicelluloses. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. Removing impurities also lessens the occurrence of voids, enhancing the general packing of the composite. However, the alkaline treatment does not considerably boost the ultimate stress of the composites since the chemical modification might weaken the fibers. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicelluloses and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix, but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in ultimate stress. The acetic acid-treated fibers show the lowest ultimate stress among the treated fibers, as the partial removal of impurities and improved filler-matrix interaction may not be sufficient to compensate for the weakening effect of the chemical modification. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicelluloses and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

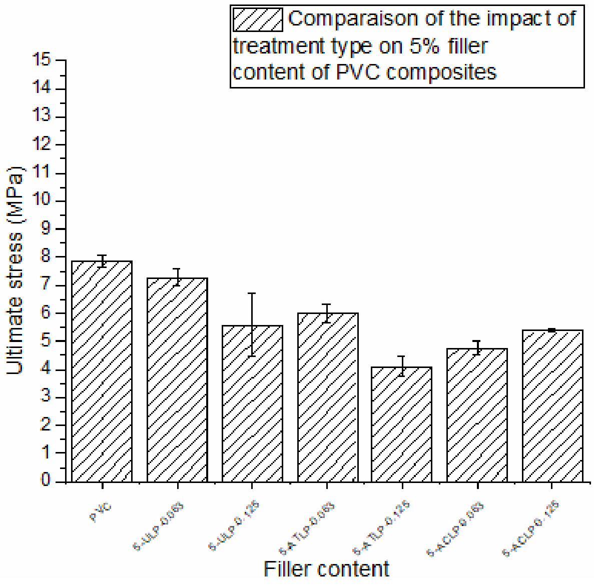

Figure 76 Comparison of the impact of treatment type on 5% filler content of PVC composites Ultimate stress

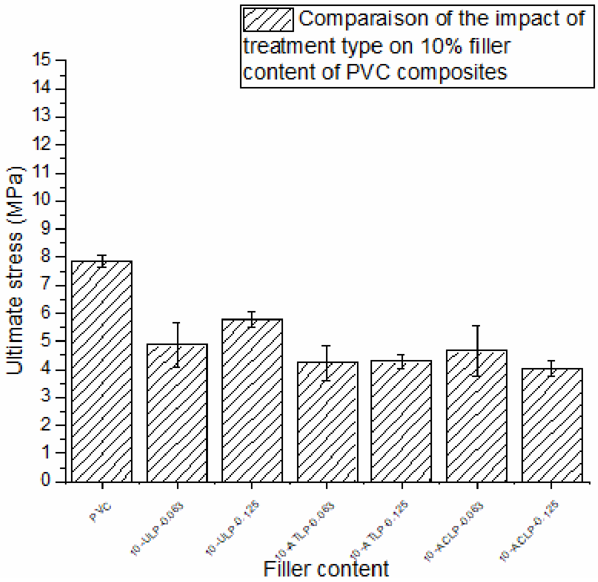

Figure 77 Comparison of the impact of treatment type on 10% filler content of PVC composites Ultimate stress

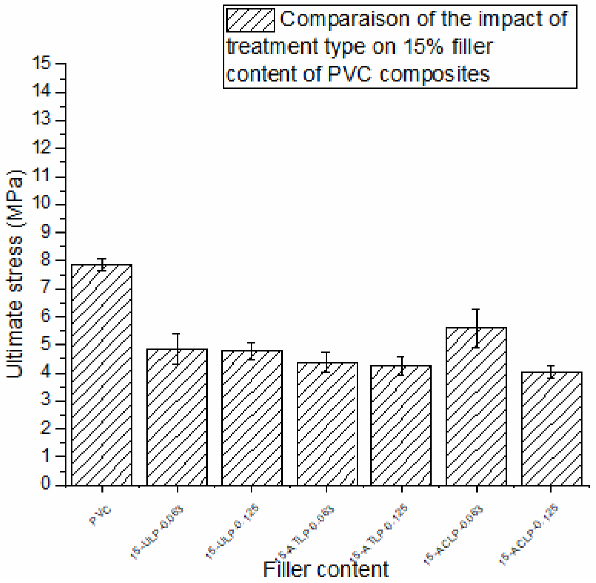

Figure 78 Comparison of the impact of treatment type on 15% filler content of PVC composites Ultimate stress

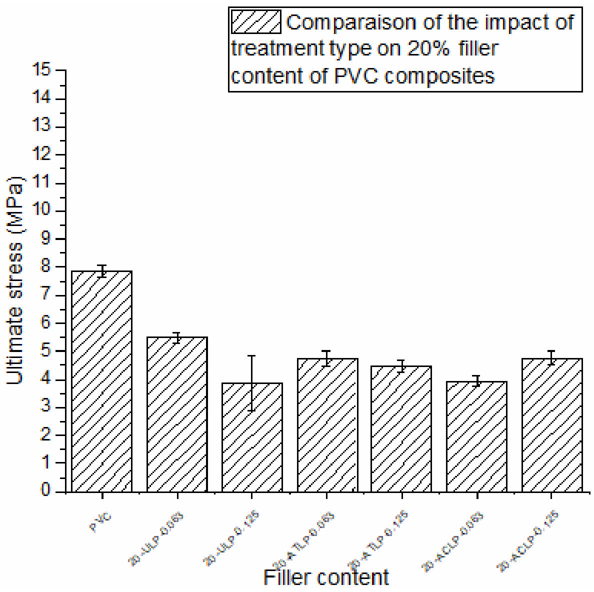

Figure 79 Comparison of the impact of treatment type on 20% filler content of PVC composites Ultimate stress

c) ***Maximum strength***

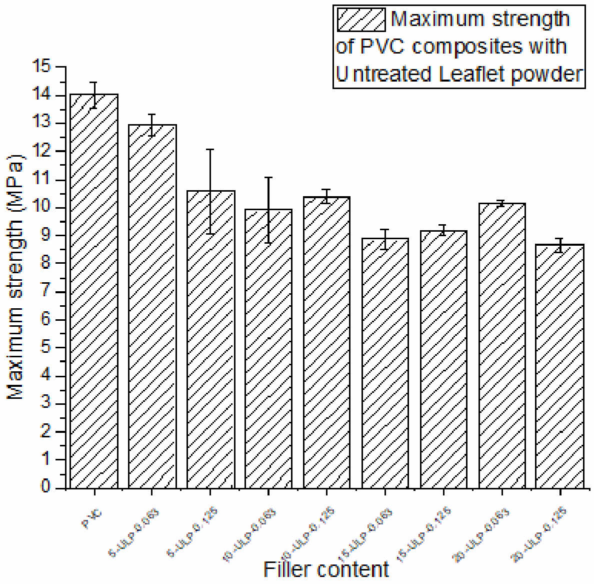

Figure 80 Maximum strength results of PVC composites with ULP in function of filler content and filler particle size

The Maximum strength results of neat PVC and its composites provide a comprehensive understanding of how the incorporation of date palm leaflet powder (DPLP) and its treatment (untreated, alkaline-treated, or acetic acid-treated) influence the mechanical strength properties of the PVC matrix. The Maximum strength of neat PVC is 14.026 MPa, serving as the baseline for comparison.When DPLP is added to the PVC matrix, the Maximum strength changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology.For untreated leaflet powder (ULP) composites, the Maximum strength ranges from 8.656 MPa to 12.940 MPa, decreasing from 7.7% to 38.3% compared to neat PVC.

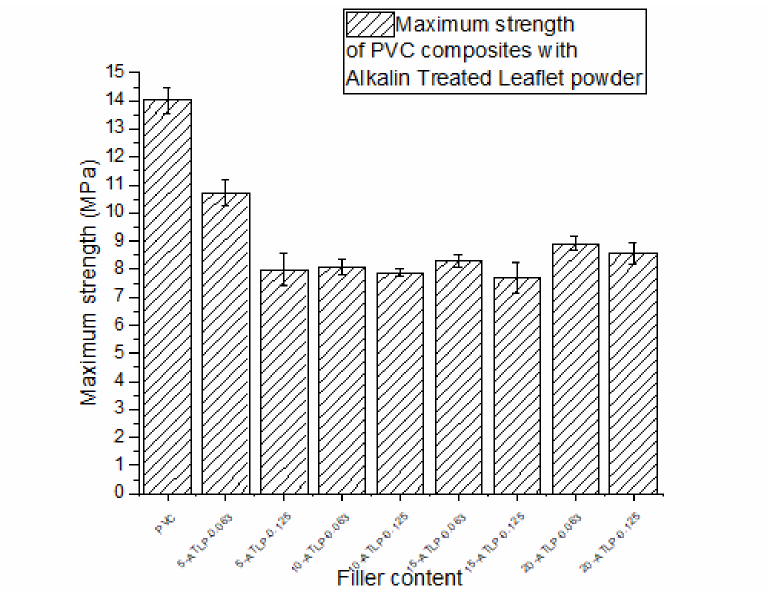

Figure 81 Maximum strength results of PVC composites with ATLP in function of filler content and filler particle size

At lower percentages (5% and 10%), the Maximum strength is slightly lower than neat PVC, with 5-ULP-0.063 showing a Maximum strength of 12.940 MPa (↓7.7% from neat PVC) and 10-ULP-0.125 showing 10.401 MPa (↓25.8% from neat PVC). This suggests that the addition of untreated fibers can reduce the Maximum strength of the composites. As the percentage of ULP increases (from 15% to 20%), the Maximum strength further decreases, with 20-ULP-0.063 showing 10.163 MPa (↓27.5% from neat PVC) and 20-ULP-0.125 showing 8.657 MPa (↓38.3% from neat PVC). This decrease is likely due to the higher filler content introducing defects and weak interfacial bonding. The particle size also plays a role, with 0.063 mm particles generally providing slightly higher Maximum strength than 0.125 mm particles.

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit a decrease in Maximum strength, ranging from 7.707 MPa to 10.719 MPa, representing a decrease of 23.6% to 45.0% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows a Maximum strength of 10.719 MPa (↓23.6% from neat PVC), while 20-ATLP-0.125 shows 8.575 MPa (↓38.9% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the Maximum strength decreases, indicating that the alkaline treatment does not significantly enhance the Maximum strength of the composites. The particle size also influences the Maximum strength, with 0.063 mm particles generally providing slightly higher Maximum strength than 0.125 mm particles.

The acetic acid-treated leaflet powder (ACLP) composites show a decrease in Maximum strength, ranging from 6.992 MPa to 9.665 MPa, representing a decrease of 31.1% to 50.2% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.125 shows a Maximum strength of 9.665 MPa (↓31.1% from neat PVC), while 20-ACLP-0.063 shows 8.090 MPa (↓42.3% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the Maximum strength decreases, indicating that the acetic acid treatment does not significantly enhance the Maximum strength of the composites. The particle size also plays a role, with 0.125 mm particles generally providing slightly higher Maximum strength than 0.063 mm particles.

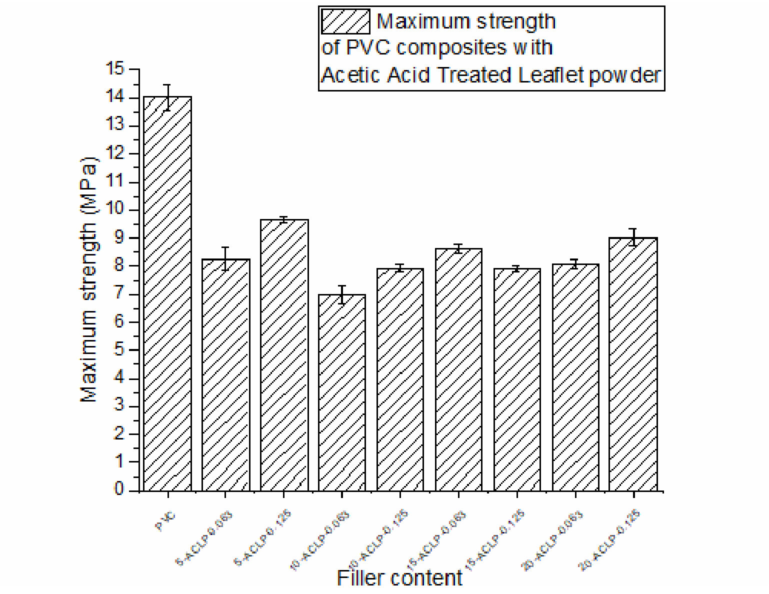

Figure 82 Maximum strength results of PVC composites with ACLP in function of filler content and filler particle size

<u>Comparison of Treatment Effects at Fixed Filler Content</u>

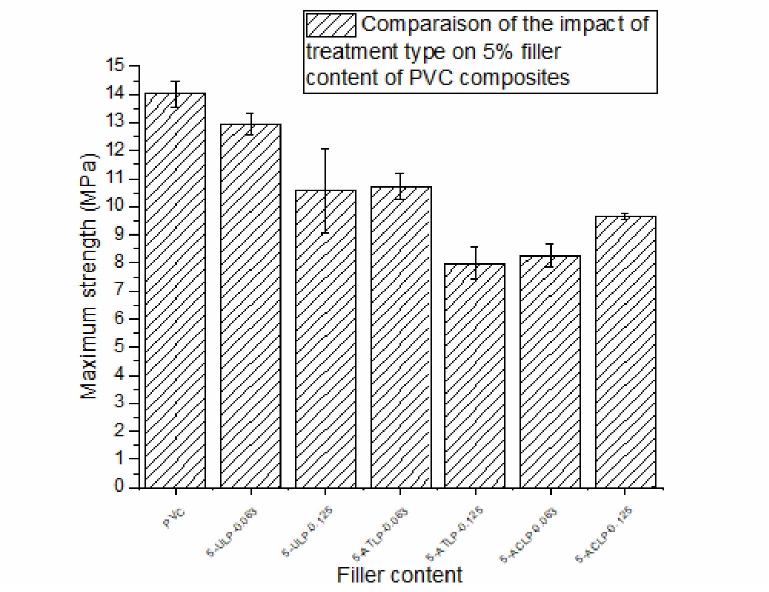

Figure 83 Comparison of the impact of treatment type on 5% filler content of PVC composites Maximum strength

To better understand the impact of treatment type, we compare the maximum strength of composites with the same filler content but with different treatments. For 5% filler content, 5-ULP-0.063 shows a Maximum strength of 12.940 MPa (↓7.7% from neat PVC), while 5-ATLP-0.063 shows 10.719 MPa (↓23.6% from neat PVC), and 5-ACLP-0.063 shows 8.263 MPa (↓41.1% from neat PVC). This comparison shows that untreated fibers provide the highest Maximum strength, followed by alkaline treatment, while acetic acid treatment shows the lowest Maximum strength. For 10% filler content, 10-ULP-0.125 shows a Maximum strength of 10.401 MPa (↓25.8% from neat PVC), while 10-ATLP-0.125 shows 7.886 MPa (↓43.8% from neat PVC), and 10-ACLP-0.125 shows 7.935 MPa (↓43.4% from neat PVC). Here, untreated fibers provide the highest Maximum strength, followed by acetic acid and alkaline treatments. For 20% filler content, 20-ULP-0.063 shows a Maximum strength of 10.163 MPa (↓27.5% from neat PVC), while 20-ATLP-0.125 shows 8.575 MPa (↓38.9% from neat PVC), and 20-ACLP-0.125 shows 9.037 MPa (↓35.6% from neat PVC). At higher filler percentages, untreated fibers provide the highest Maximum strength, followed by acetic acid and alkaline treatments.

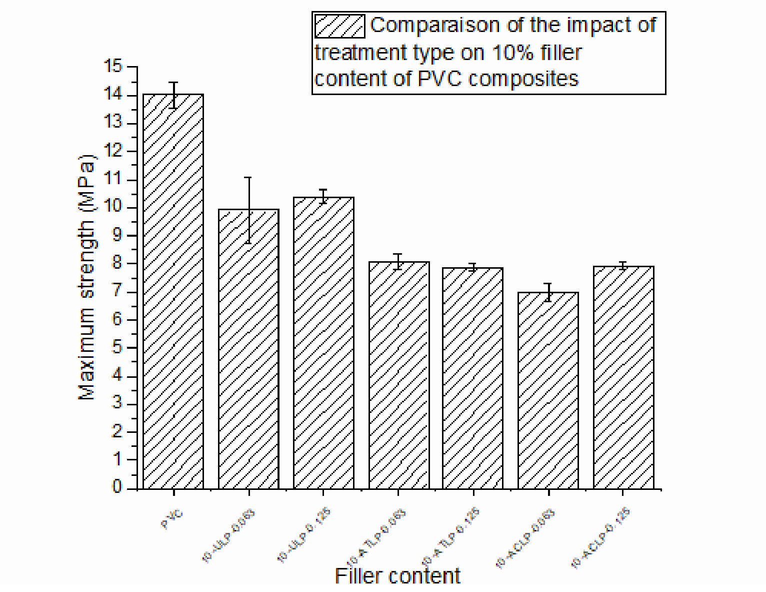

Figure 84 Comparison of the impact of treatment type on 10% filler content of PVC composites Maximum strength

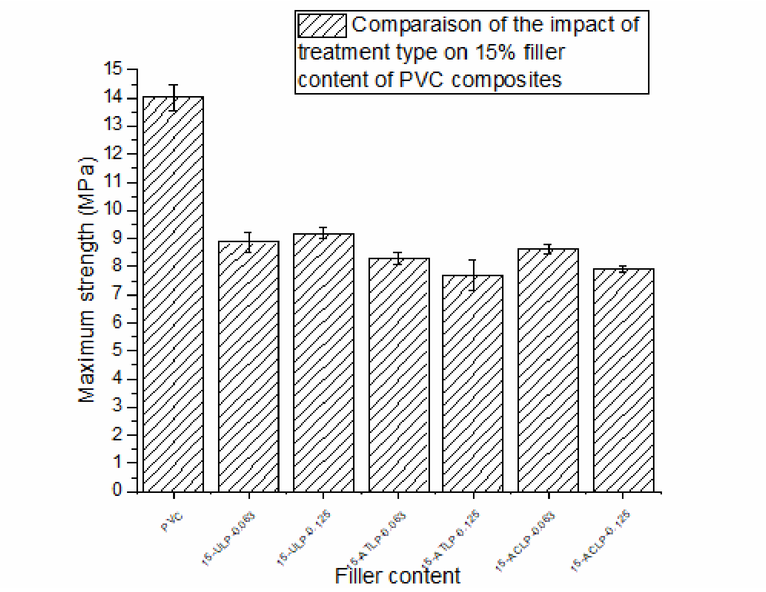

Figure 85 Comparison of the impact of treatment type on 15% filler content of PVC composites Maximum strength

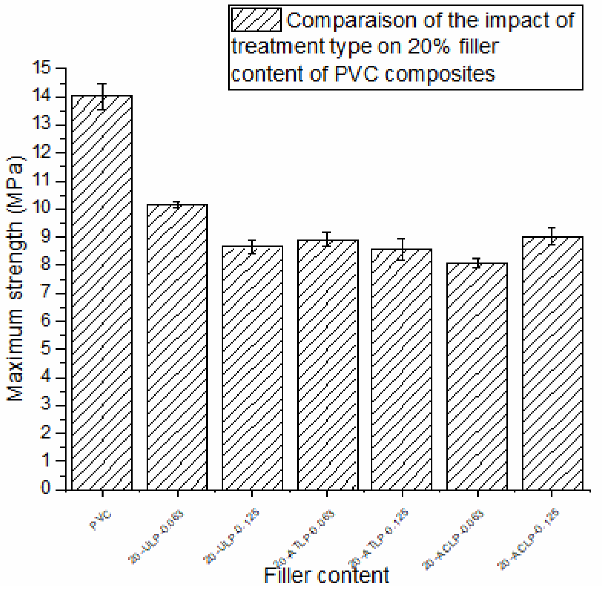

Figure 86 Comparison of the impact of treatment type on 20% filler content of PVC composites Maximum strength

Comparison of Filler Content and Size Effects at Fixed Treatment Type

To better understand the impact of filler content and size, we compare the maximum strength of composites with the same treatment but with different filler content and size. For untreated fibers (ULP), the Maximum strength decreases with filler content, with 5-ULP-0.063 showing 12.940 MPa (↓7.7% from neat PVC) and 20-ULP-0.125 showing 8.657 MPa (↓38.3% from neat PVC). The particle size also plays a role, with 0.063 mm particles generally providing slightly higher Maximum strength than 0.125 mm particles. For alkaline-treated fibers (ATLP), the Maximum strength decreases with filler content, with 5-ATLP-0.063 showing 10.719 MPa (↓23.6% from neat PVC) and 20-ATLP-0.125 showing 8.575 MPa (↓38.9% from neat PVC). The particle size also influences the Maximum strength, with 0.063 mm particles generally providing slightly higher Maximum strength than 0.125 mm particles. For acetic acid-treated fibers (ACLP), the Maximum strength decreases with filler content, with 5-ACLP-0.125 showing 9.665 MPa (↓31.1% from neat PVC) and 20-ACLP-0.063 showing 8.090 MPa (↓42.3% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing slightly higher Maximum strength than 0.063 mm particles.

The Maximum strength of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicelluloses, which hinder adequate bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows firm peaks for hemicelluloses, lignin, and other non-cellulosic materials, contributing to poor compatibility with the PVC matrix. SEM images show that the surface is rough and uneven with appreciable amount of impurities, providing an additional evidence for the poor fibermatrix interaction. Untreated fibers, however, possess the Maximum strength of any of the treated ones, understandably because of their natural stiffness and absence of chemical modification that could make them weaker.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicelluloses. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. Removal of impurities also reduces the formation of voids and packs specific combined materials more closely. However, the alkaline treatment has no significant effect on the maximum strength of the composite for the reason that chemical modification may weaken the fibers. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicelluloses and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes any residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in Maximum strength. The acetic acid-treated fibres show the lowest Maximum strength among the treated fibres, as the partial removal of impurities and improved filler-matrix interaction may not be sufficient to compensate for the weakening effect of the chemical modification. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicelluloses and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

d) <u>Ultimate strain</u>

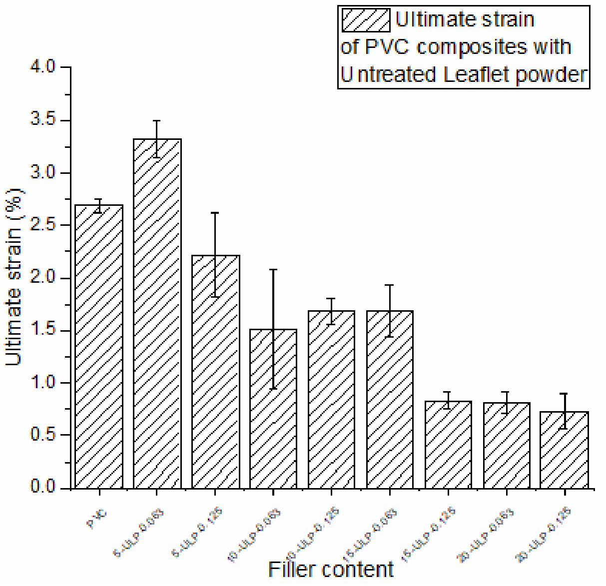

Figure 87Ultimate strain results of PVC composites with ULP in function of filler content and filler particle size

The ultimate strain results of neat PVC and its composites, combined with insights from FTIR and SEM analyses, provide a comprehensive understanding of how the incorporation of date palm leaflet powder (DPLP) and its treatment (untreated, alkaline-treated, or acetic acid-treated) influence the ductility, chemical composition, and morphological properties of the PVC matrix. The ultimate strain of neat PVC is 2.689, serving as the baseline for comparison. When DPLP is added to the PVC matrix, the ultimate strain changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology. For untreated leaflet powder (ULP) composites, the ultimate strain ranges from 0.728 to 3.323, showing both increases and decreases compared to neat PVC. At lower percentages (5% and 10%), the ultimate strain is slightly higher than neat PVC, with 5-ULP-0.063 showing an ultimate strain of 3.323 (↑23.6% from neat PVC) and 10-ULP-0.125 showing 1.685 (↓37.3% from neat PVC). This suggests that the addition of untreated fibers can enhance the ductility of the composites at lower percentages due to their natural flexibility. As the percentage of ULP increases (from 15% to 20%), the ultimate strain further decreases, with 20-ULP-0.063 showing 0.816 (↓69.6% from neat PVC) and 20-ULP-0.125 showing 0.728 (↓72.9% from neat PVC). This decrease is likely due to the higher filler content introducing defects and weak interfacial bonding. The particle size also plays a role, with 0.063 mm particles generally providing higher ultimate strain than 0.125 mm particles.

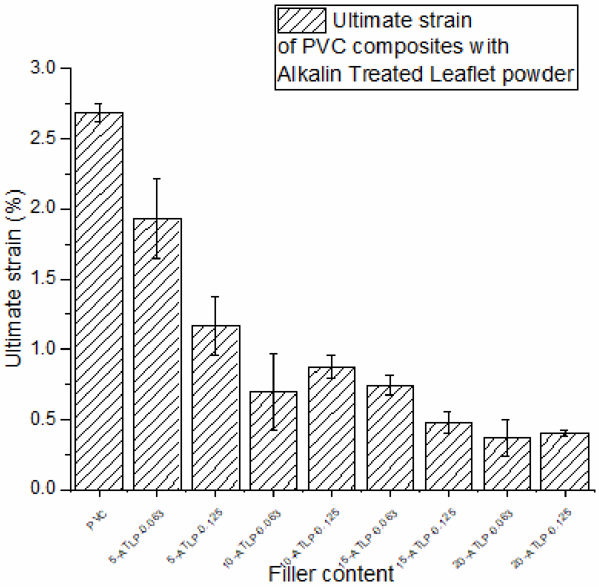

Figure 88 Ultimate strain results of PVC composites with ATLP in function of filler content and filler particle size

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit a decrease in ultimate strain, ranging from 0.367 to 1.933, representing a decrease of 28.1% to 86.3% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows an ultimate strain of 1.933 (↓28.1% from neat PVC), while 20-ATLP-0.125 shows 0.400 (↓85.1% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the ultimate strain decreases, indicating that the alkaline treatment reduces the ductility of the composites. The particle size also influences the ultimate strain, with 0.063 mm particles generally providing a higher ultimate strain than 0.125 mm particles.

The acetic acid-treated leaflet powder (ACLP) composites show a decrease in ultimate strain, ranging from 0.252 to 1.813, representing a decrease of 32.6% to 90.6% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.063 shows an ultimate strain of 1.813 (↓32.6% from neat PVC), while 20-ACLP-0.125 shows 0.252 (↓90.6% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the ultimate strain decreases, indicating that the acetic acid treatment reduces the ductility of the composites. The particle size also plays a role, with 0.063 mm particles generally providing higher ultimate strain than 0.125 mm particles.

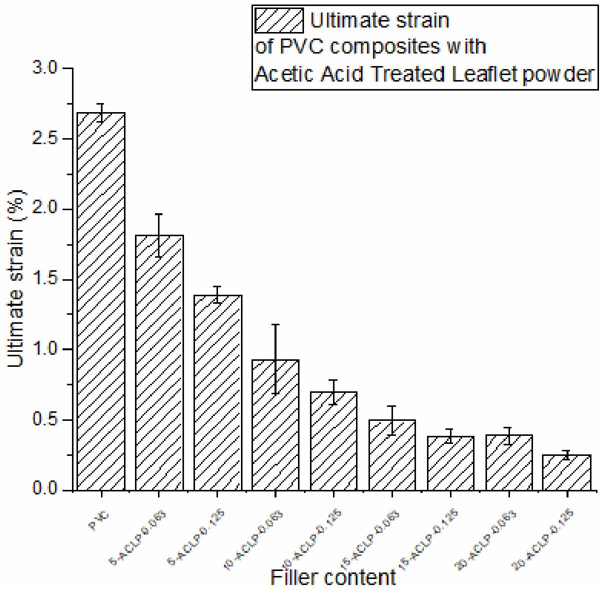

Figure 89 Ultimate strain results of PVC composites with ACLP in function of filler content and filler particle size

Comparison of Treatment Effects at Fixed Filler Content

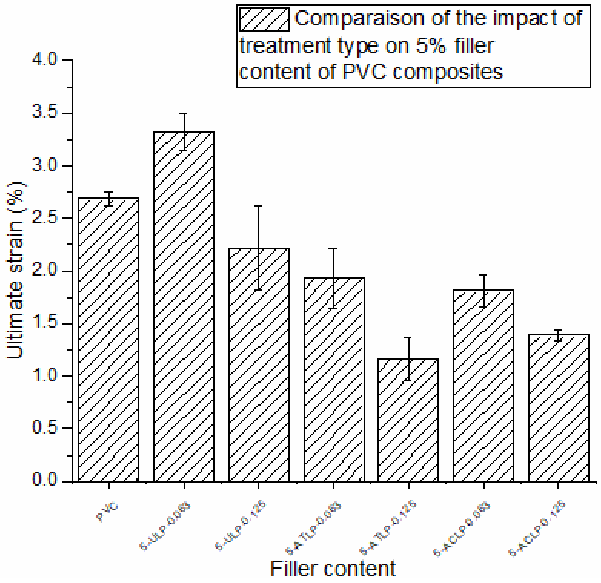

Figure 90 Comparison of the impact of treatment type on 5% filler content of PVC composites Ultimate strain

To better understand the impact of treatment type, we compare the ultimate strain of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.063 shows an ultimate strain of 3.323 (↑23.6% from neat PVC), while 5-ATLP-0.063 shows 1.933 (↓28.1% from neat PVC), and 5-ACLP-0.063 shows 1.813 (↓32.6% from neat PVC). This comparison shows that untreated fibers provide the highest ultimate strain, followed by alkaline treatment, while acetic acid treatment shows the lowest ultimate strain. For 10% filler content, 10-ULP-0.125 shows an ultimate strain of 1.685 (↓37.3% from neat PVC), while 10-ATLP-0.125 shows 0.876 (↓67.4% from neat PVC), and 10-ACLP-0.125 shows 0.695 (↓74.1% from neat PVC). Here, untreated fibers provide the highest ultimate strain, followed by alkaline and acetic acid treatments. For 20% filler content, 20-ULP-0.063 shows an ultimate strain of 0.816 (↓69.6% from neat PVC), while 20-ATLP-0.125 shows 0.400 (↓85.1% from neat PVC), and 20-ACLP-0.125 shows 0.252 (↓90.6% from neat PVC). At higher filler percentages, untreated fibers provide the highest ultimate strain, followed by alkaline and acetic acid treatments.

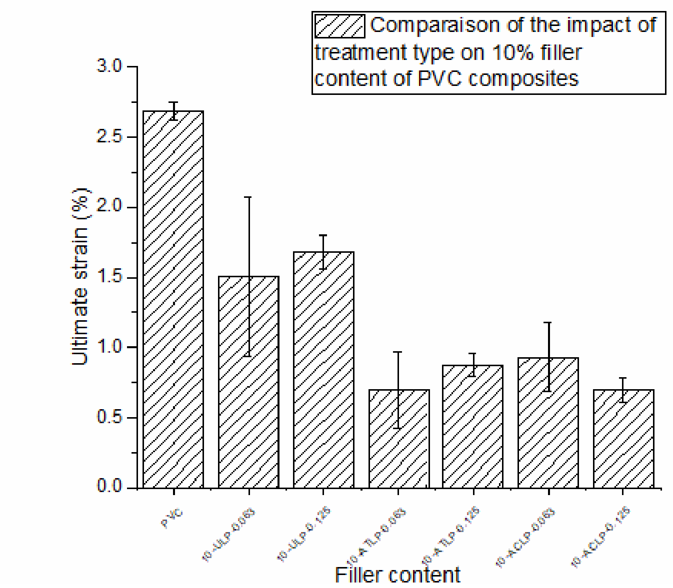

Figure 91 Comparison of the impact of treatment type on 10% filler content of PVC composites Ultimate strain

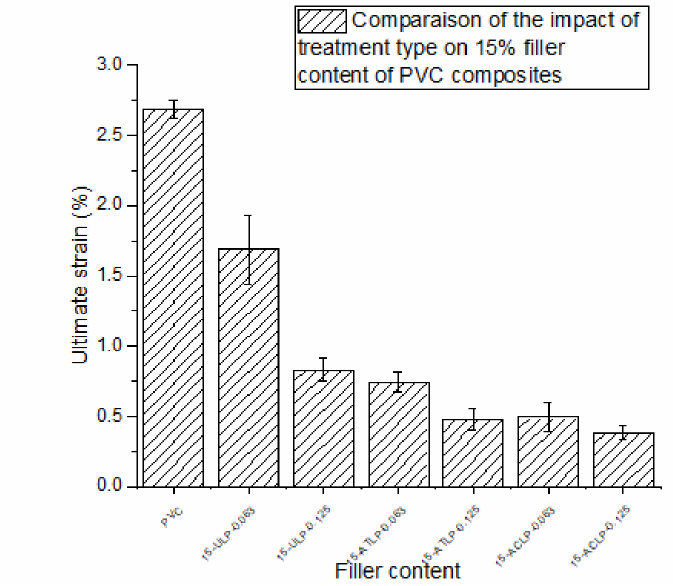

Figure 92 Comparison of the impact of treatment type on 15% filler content of PVC composites Ultimate strain

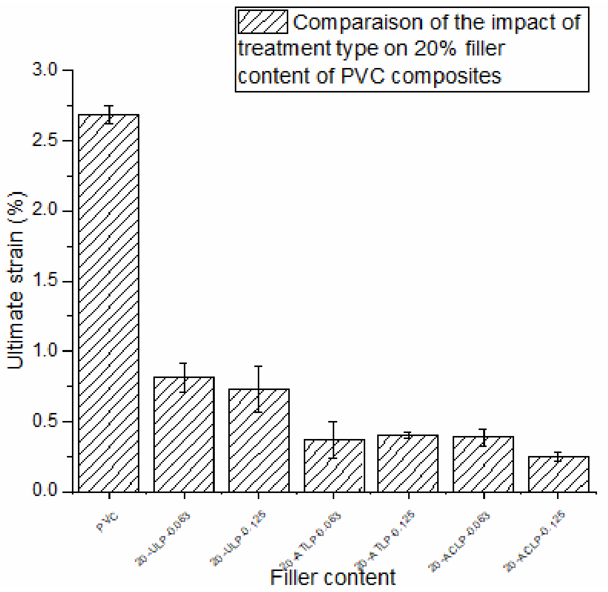

Figure 93 Comparison of the impact of treatment type on 20% filler content of PVC composites Ultimate strain

<u>Comparison of Filler Content and Size Effects at Fixed Treatment Type</u>

To better understand the impact of filler content and size, we compare the ultimate strain of composites with the same treatment but different filler content and size. For untreated fibers (ULP), the ultimate strain decreases with filler content, with 5-ULP-0.063 showing 3.323 (↑23.6% from neat PVC) and 20-ULP-0.125 showing 0.728 (↓72.9% from neat PVC). The particle size also plays a role, with 0.063 mm particles generally providing higher ultimate strain than 0.125 mm particles. For alkaline-treated fibers (ATLP), the ultimate strain decreases with filler content, with 5-ATLP-0.063 showing 1.933 (↓28.1% from neat PVC) and 20-ATLP-0.125 showing 0.400 (↓85.1% from neat PVC). The particle size also influences the ultimate strain, with 0.063 mm particles generally providing a higher ultimate strain than 0.125 mm particles. For acetic acid-treated fibers (ACLP), the ultimate strain decreases with filler content, with 5-ACLP-0.063 showing 1.813 (↓32.6% from neat PVC) and 20-ACLP-0.125 showing 0.252 (↓90.6% from neat PVC). The particle size also plays a role, with 0.063 mm particles generally providing higher ultimate strain than 0.125 mm particles.

The ultimate strain of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicelluloses, which hinder adequate bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows firm peaks for hemicelluloses, lignin, and other non-cellulosic materials, contributing to poor compatibility with the PVC matrix. The SEM images show a rough surface with visible impurities, further proving the poor filler-matrix interaction. Notwithstanding these drawbacks, untreated fibers, due to their inherent flexibility and lack of any stiffness from chemical modification, provided the most arduous ultimate strain among the three kinds of treated fibers.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicelluloses. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. While removing impurities will help reduce the formation of voids and improve the overall packing of the composite, the alkaline treatment will reduce the ultimate strain of the composites, since chemical modification may stiffen the fibers. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicelluloses and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix, but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in ultimate strain. The acetic acid-treated fibers show the lowest ultimate strain among the treated fibers, as the partial removal of impurities and improved filler-matrix interaction may not be sufficient to compensate for the stiffening effect of the chemical modification. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicelluloses and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

e) <u>Maximum strain</u>

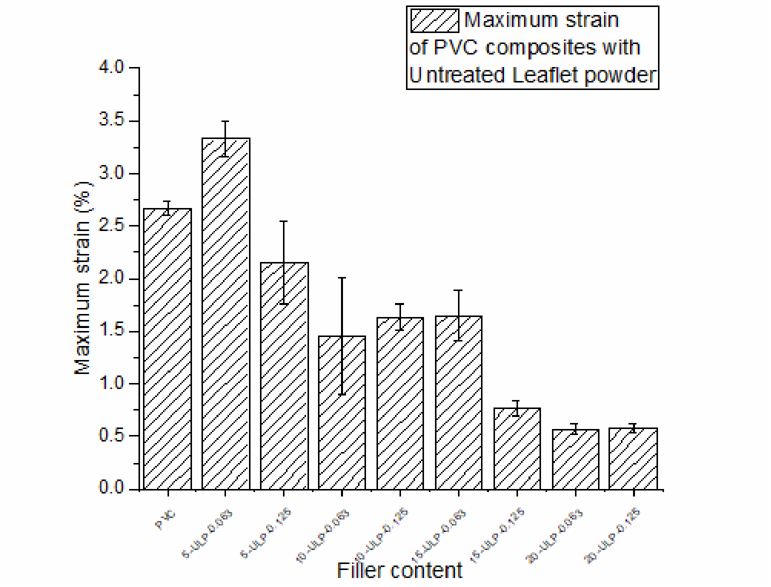

Figure 94 Maximum strain results of PVC composites with ULP in function of filler content and filler particle size

The maximum strain results of neat PVC and its composites, combined with insights from FTIR and SEM analyses, provide a comprehensive understanding of how the incorporation of date palm leaflet powder (DPLP) and its treatment (untreated, alkaline-treated, or acetic acid-treated) influence the ductility, chemical composition, and morphological properties of the PVC matrix. The maximum strain of neat PVC is 2.670, serving as the baseline for comparison. When DPLP is added to the PVC matrix, the maximum strain changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology. For untreated leaflet powder (ULP) composites, the maximum strain ranges from 0.572 to 3.329, showing both increases and decreases compared to neat PVC. At lower percentages (5% and 10%), the maximum strain is slightly higher than neat PVC, with 5-ULP-0.063 showing a maximum strain of 3.329 (↑24.7% from neat PVC) and 10-ULP-0.125 showing 1.636 (↓38.7% from neat PVC). This suggests that the addition of untreated fibers can enhance the ductility of the composites at lower percentages due to their natural flexibility. As the percentage of ULP increases (from 15% to 20%), the maximum strain further decreases, with 20-ULP-0.063 showing 0.572 (↓78.6% from neat PVC) and 20-ULP-0.125 showing 0.576 (↓78.4% from neat PVC). This decrease is likely due to the higher filler content introducing defects and weak interfacial bonding. The particle size also plays a role, with 0.063 mm particles generally providing a higher maximum strain than 0.125 mm particles.

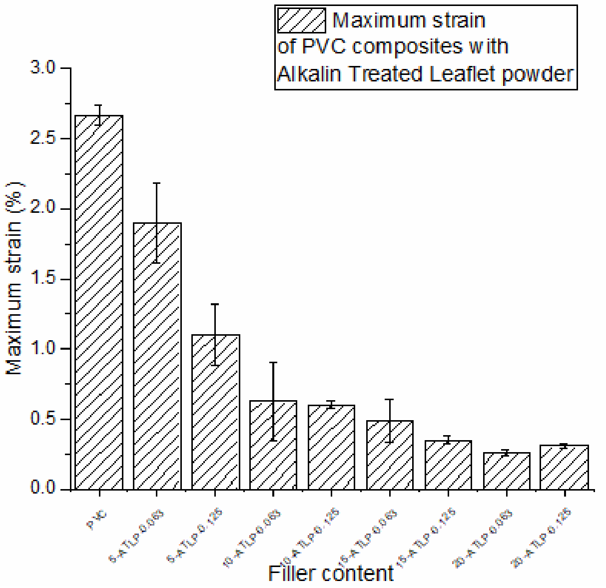

Figure 95 Maximum strain results of PVC composites with ATLP in function of filler content and filler particle size

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit a decrease in maximum strain, ranging from 0.260 to 1.898, representing a decrease of 28.9% to 90.3% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows a maximum strain of 1.898 (↓28.9% from neat PVC), while 20-ATLP-0.125 shows 0.310 (↓88.4% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the maximum strain decreases, indicating that the alkaline treatment reduces the ductility of the composites. The particle size also influences the maximum strain, with 0.063 mm particles generally providing a higher maximum strain than 0.125 mm particles.

The acetic acid-treated leaflet powder (ACLP) composites show a decrease in maximum strain, ranging from 0.202 to 1.782, representing a decrease of 33.2% to 92.4% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.063 shows a maximum strain of

1.782 (↓33.2% from neat PVC), while 20-ACLP-0.125 shows 0.202 (↓92.4% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the maximum strain decreases, indicating that the acetic acid treatment reduces the ductility of the composites. The particle size also plays a role, with 0.063 mm particles generally providing a higher maximum strain than 0.125 mm particles.

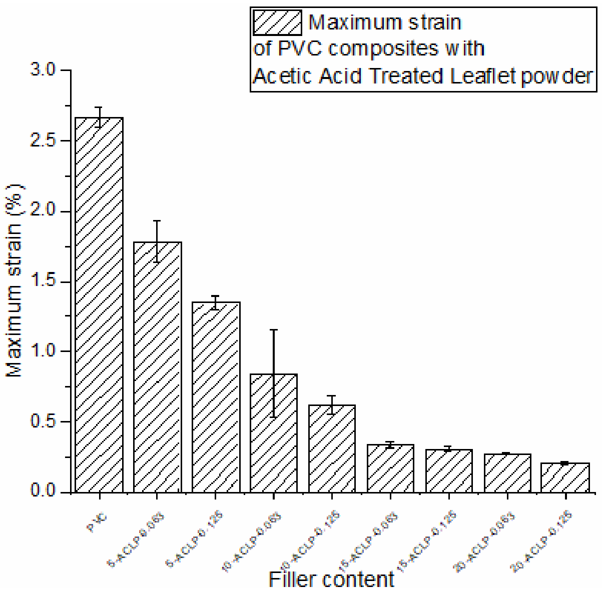

Figure 96 Maximum strain results of PVC composites with ACLP in function of filler content and filler particle size

Comparison of Treatment Effects at Fixed Filler Content

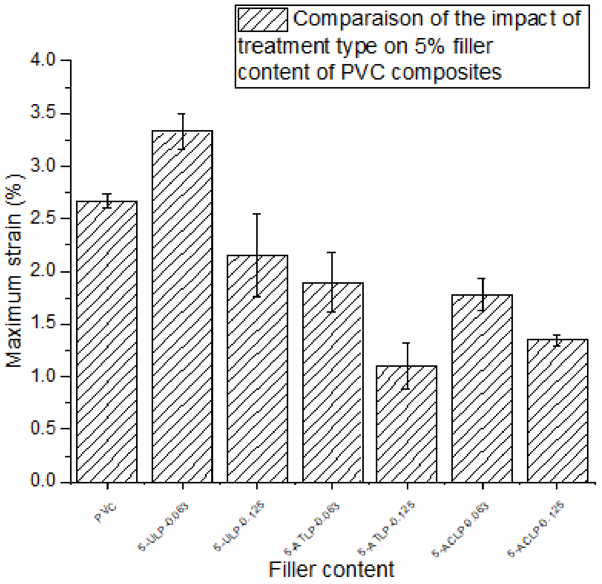

Figure 97 Comparison of the impact of treatment type on 5% filler content of PVC composites Maximum strain

To better understand the impact of treatment type, we compare the maximum strain of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.063 shows a maximum strain of 3.329 (↑24.7% from neat PVC), while 5-ATLP-0.063 shows 1.898 (↓28.9% from neat PVC), and 5-ACLP-0.063 shows 1.782 (↓33.2% from neat PVC). This comparison shows that untreated fibers provide the highest maximum strain, followed by alkaline treatment, while acetic acid treatment shows the lowest maximum strain. For 10% filler content, 10-ULP-0.125 shows a maximum strain of 1.636 (↓38.7% from neat PVC), while 10-ATLP-0.125 shows 0.600 (↓77.5% from neat PVC), and 10-ACLP-0.125 shows 0.620 (↓76.8% from neat PVC). Untreated fibers provide the highest maximum strain, followed by acetic acid and alkaline treatments. For 20% filler content, 20-ULP-0.063 shows a maximum strain of 0.572 (↓78.6% from neat PVC), while 20-ATLP-0.125 shows 0.310 (↓88.4% from neat PVC), and 20-ACLP-0.125 shows 0.202 (↓92.4% from neat PVC). At higher filler percentages, untreated fibers provide the highest maximum strain, followed by alkaline and acetic acid treatments.

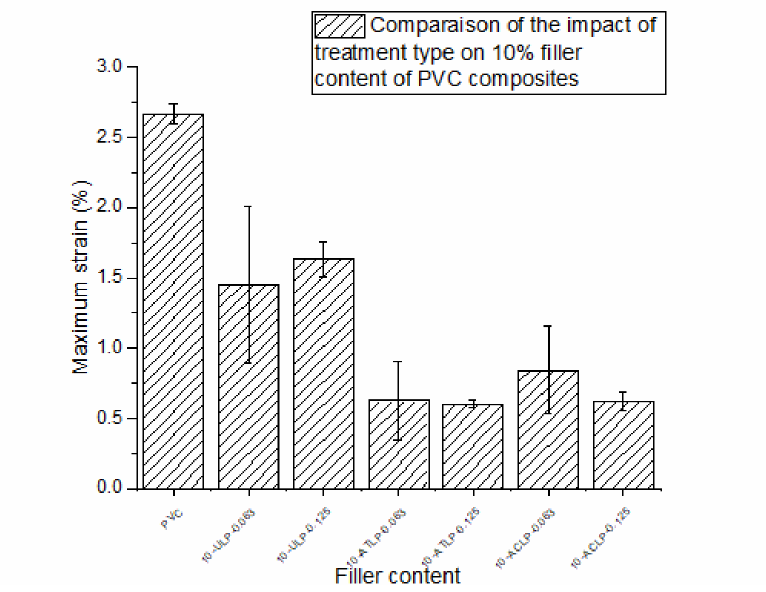

Figure 98 Comparison of the impact of treatment type on 10% filler content of PVC composites Maximum strain

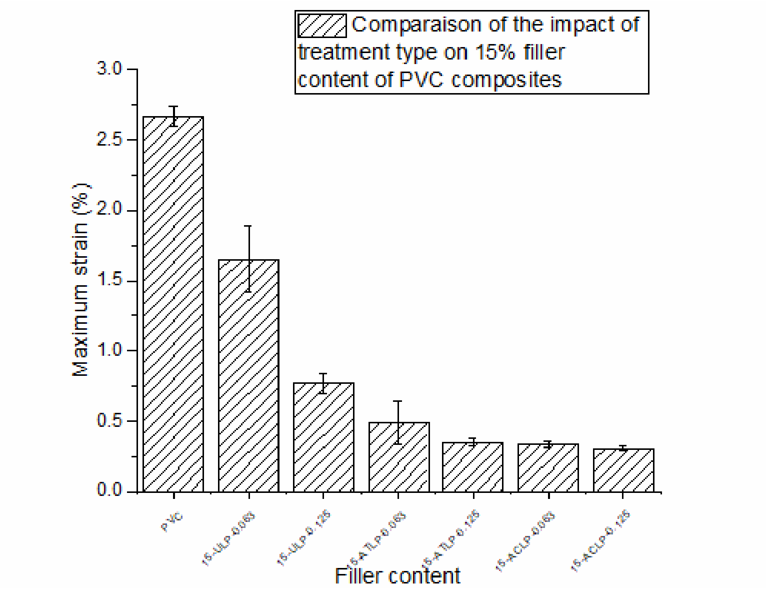

Figure 99 Comparison of the impact of treatment type on 15% filler content of PVC composites Maximum strain

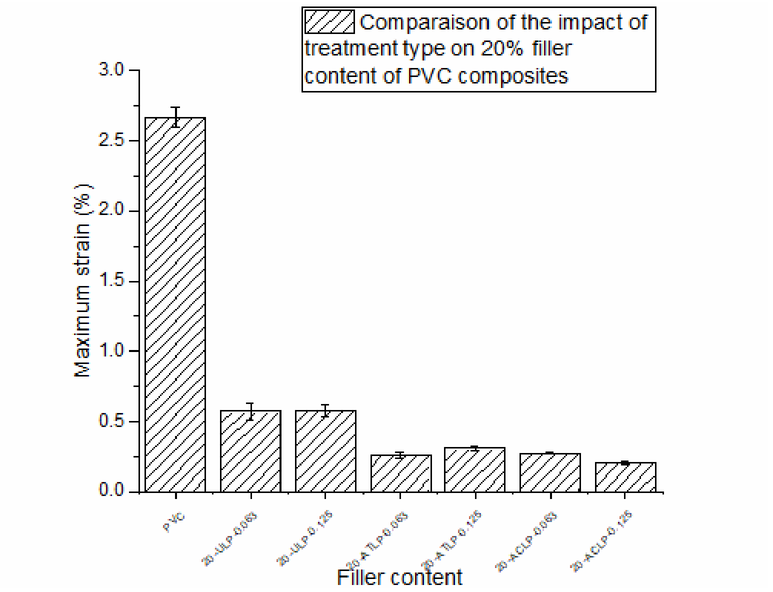

Figure 100 Comparison of the impact of treatment type on 20% filler content of PVC composites Maximum strain

Comparison of Filler Content and Size Effects at Fixed Treatment Type

To better understand the impact of filler content and size, we compare the maximum strain of composites with the same treatment but different filler content and size. For untreated fibers (ULP), the maximum strain decreases with filler content, with 5-ULP-0.063 showing 3.329 (↑24.7% from neat PVC) and 20-ULP-0.125 showing 0.576 (↓78.4% from neat PVC). The particle size also plays a role, with 0.063 mm particles generally providing a higher maximum strain than 0.125 mm particles. For alkaline-treated fibers (ATLP), the maximum strain decreases with filler content, with 5-ATLP-0.063 showing 1.898 (↓28.9% from neat PVC) and 20-ATLP-0.125 showing 0.310 (↓88.4% from neat PVC). The particle size also influences the maximum strain, with 0.063 mm particles generally providing a higher maximum strain than 0.125 mm particles. For acetic acid-treated fibers (ACLP), the maximum strain decreases with filler content, with 5-ACLP-0.063 showing 1.782 (↓33.2% from neat PVC) and 20-ACLP-0.125 showing 0.202 (↓92.4% from neat PVC). The particle size also plays a role, with 0.063 mm particles generally providing a higher maximum strain than 0.125 mm particles.

The maximum strain of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicelluloses, which hinder adequate bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows firm peaks for hemicelluloses, lignin, and other non-cellulosic materials, contributing to poor compatibility with the PVC matrix. The SEM images illustrate that the fiber has a rough, uneven surface with visible impurities acting against the filler-matrix interaction. Despite these limitations, untreated fibers have the highest maximum strain of all the treated fibers, probably because of their natural flexibility and the fact that no chemical modifications were performed that could stiffen the fibers.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicelluloses. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. The elimination of impurities minimizes the development of voids and drives a better packing capacity of the composite. However, the alkaline treatment reduces the maximum strain of the composites since the chemical modification may stiffen the fibers. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicelluloses and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes any residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in maximum strain. The acetic acid-treated fibers show the lowest maximum strain among the treated fibers, as the partial removal of impurities and improved filler-matrix interaction may not be sufficient to compensate for the stiffening effect of the chemical modification. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicelluloses and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

III.2.7. Bending test

a) Bending modulus

The bending modulus results of neat PVC and its composites, combined with insights from FTIR and SEM analyses, provide a comprehensive understanding of how the incorporation of date palm leaflet powder (DPLP) and its treatment (untreated, alkaline-treated, or acetic acid-treated) influence the stiffness, chemical composition, and morphological properties of the PVC matrix. The bending modulus of neat PVC is 118.30 MPa, serving as the baseline for comparison. When DPLP is added to the PVC matrix, the bending modulus changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology. For untreated leaflet powder (ULP) composites, the bending modulus ranges from 62.38 MPa to 218.22 MPa, showing both decreases and increases compared to neat PVC. At lower percentages (5% and 10%), the bending modulus is slightly lower than neat PVC, with 5-ULP-0.063 showing a bending modulus of 73.89 MPa (↓37.5% from neat PVC) and 10-ULP-0.125 showing 129.53 MPa (↑9.5% from neat PVC). This suggests that the addition of untreated fibers can reduce the stiffness of the composites at lower percentages due to poor filler-matrix interaction. As the percentage of ULP increases (from 15% to 20%), the bending modulus further increases, with 20-ULP-0.063 showing 164.93 MPa (↑39.4% from neat PVC) and 20-ULP-0.125 showing 218.22 MPa (↑84.5% from neat PVC). This increase is likely due to the higher filler content providing additional reinforcement to the PVC matrix. The particle size also plays a role, with 0.125 mm particles generally providing higher bending modulus than 0.063 mm particles.

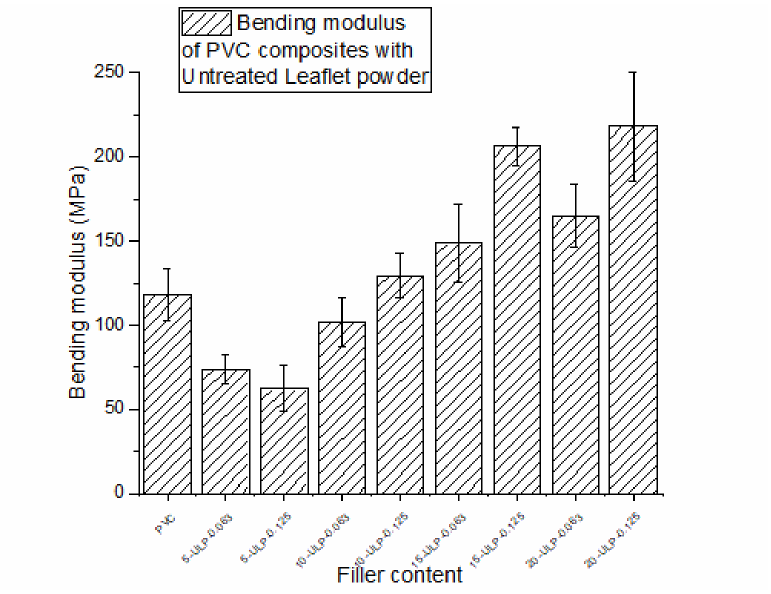

Figure 101 Bending modulus results of PVC composites with ULP in function of filler content and filler particle size

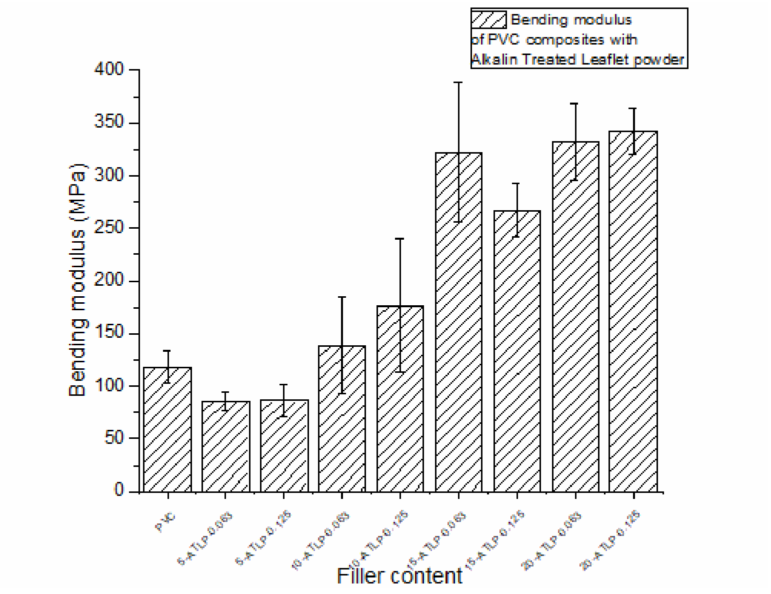

Figure 102 Bending modulus results of PVC composites with ATLP in function of filler content and filler particle size

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit significant improvements in bending modulus, ranging from 86.21 MPa to 342.49 MPa, representing an increase of up to 189.5% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows a bending modulus of 86.21 MPa (↓27.1% from neat PVC), while 20-ATLP-0.125 shows 342.49 MPa (↑189.5% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the bending modulus increases significantly, indicating that the alkaline treatment enhances the stiffness of the composites, particularly at higher filler percentages. The particle size also influences the bending modulus, with 0.125 mm particles generally providing higher bending modulus than 0.063 mm particles.

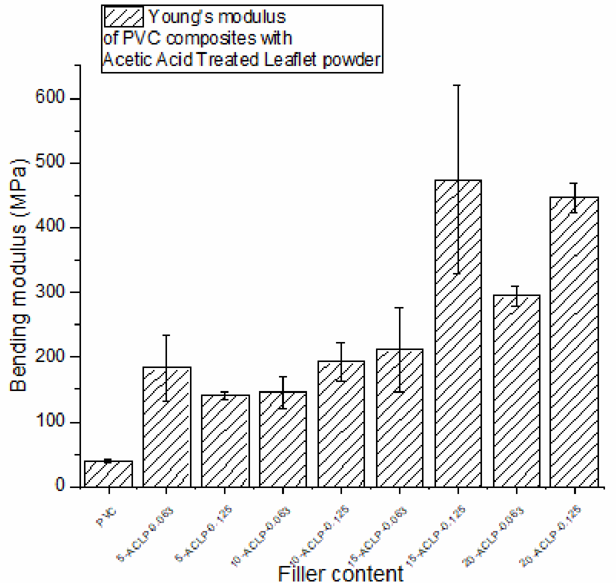

Figure 103 Bending modulus results of PVC composites with ACLP in function of filler content and filler particle size

The acetic acid-treated leaflet powder (ACLP) composites show the highest bending modulus, ranging from 141.01 MPa to 474.46 MPa, representing an increase of 19.2% to 301.0% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.063 shows a bending modulus of 183.30 MPa (↑54.9% from neat PVC), while 15-ACLP-0.125 shows 474.46 MPa (↑301.0% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the bending modulus increases significantly, indicating that the acetic acid treatment enhances the stiffness of the composites, particularly at higher filler percentages. The particle size also plays a role, with 0.125 mm particles generally providing higher bending modulus than 0.063 mm particles.

Comparison of Treatment Effects at Fixed Filler Content

To better understand the impact of treatment type, we compare the bending modulus of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.063 shows a bending modulus of 73.89 MPa (↓37.5% from neat PVC), while 5-ATLP-0.063 shows 86.21 MPa (↓27.1% from neat PVC), and 5-ACLP-0.063 shows 183.30 MPa (↑54.9% from neat PVC). This comparison shows that acetic acid treatment provides the highest bending modulus, followed by alkaline treatment, while untreated fibers show the lowest bending modulus. For 10% filler content, 10-ULP-0.125 shows a bending modulus of 129.53 MPa (↑9.5% from neat PVC), while 10-ATLP-0.125 shows 176.86 MPa (↑49.5% from neat PVC), and 10-ACLP-0.125 shows 192.67 MPa (↑62.9% from neat PVC). Here, acetic acid treatment provides the highest bending modulus, followed by alkaline treatment and untreated fibers. For 20% filler content, 20-ULP-0.125 shows a bending modulus of 218.22 MPa (↑84.5% from neat PVC), while 20-ATLP-0.125 shows 342.49 MPa (↑189.5% from neat PVC), and 20-ACLP-0.125 shows 447.00 MPa (↑277.9% from neat PVC). At higher filler percentages, acetic acid treatment provides the highest bending modulus, while alkaline treatment also shows significant improvement.

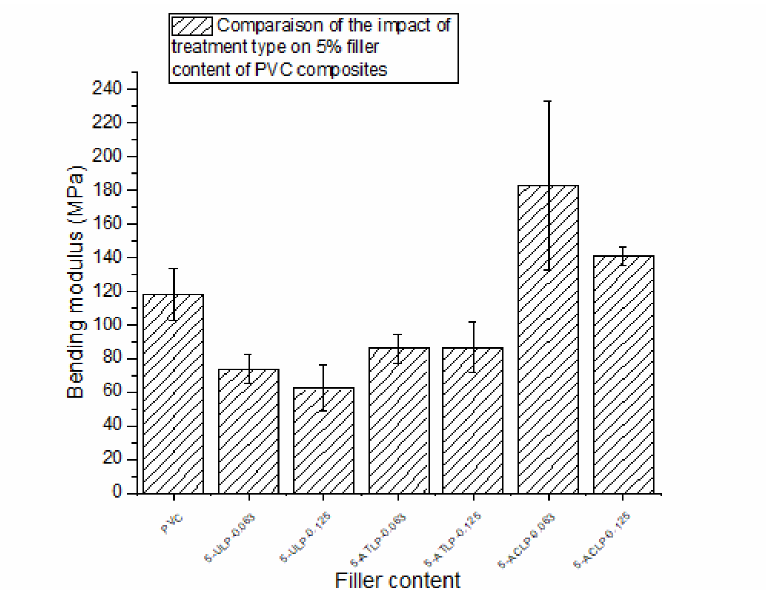

Figure 104 Comparison of the impact of treatment type on 5% filler content of PVC composites bending modulus

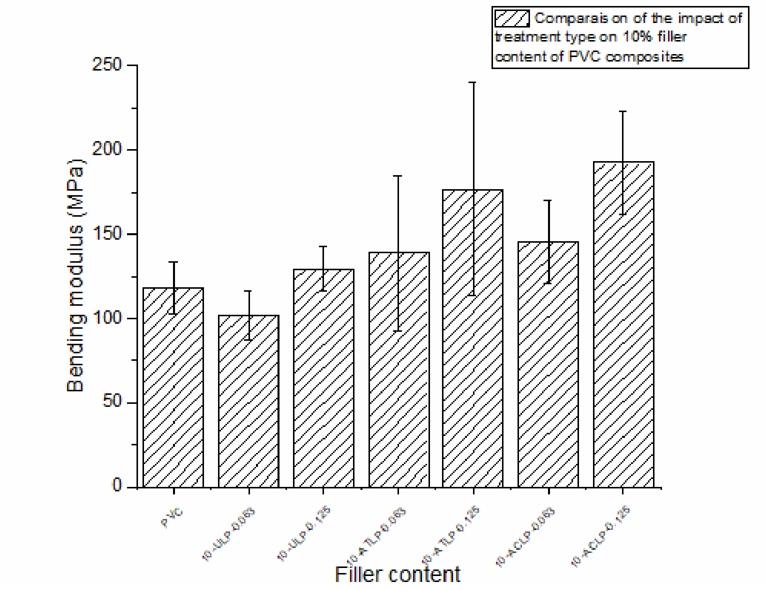

Figure 105 Comparison of the impact of treatment type on 10% filler content of PVC composites bending modulus

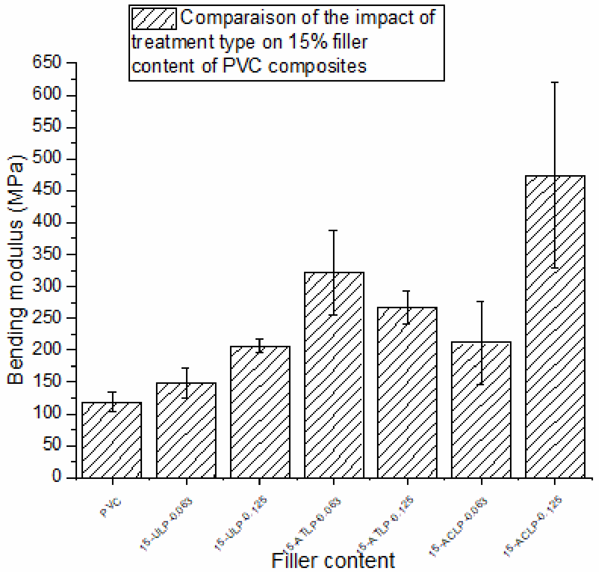

Figure 106 Comparison of the impact of treatment type on 15% filler content of PVC composites bending modulus

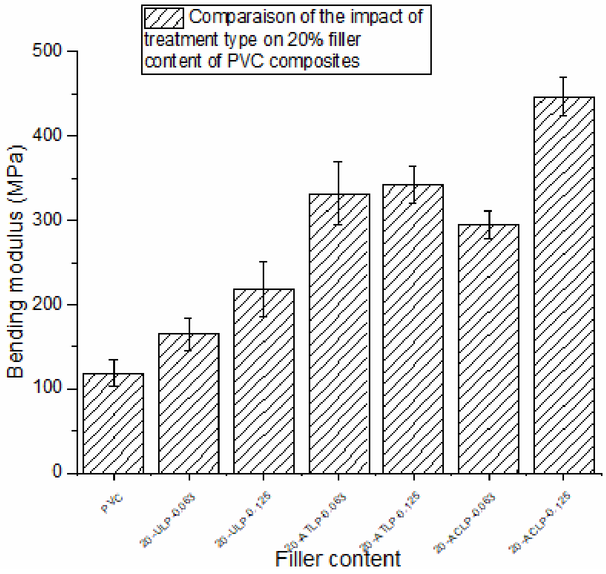

Figure 107 Comparison of the impact of treatment type on 20% filler content of PVC composites bending modulus

Comparison of Filler Content and Size Effects at Fixed Treatment Type

To better understand the impact of filler content and size, we compare the bending modulus of composites with the same treatment but different filler content and size. For untreated fibers (ULP), the bending modulus increases with filler content, with 5-ULP-0.063 showing 73.89 MPa (↓37.5% from neat PVC) and 20-ULP-0.125 showing 218.22 MPa (↑84.5% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing higher bending modulus than 0.063 mm particles. For alkaline-treated fibers (ATLP), the bending modulus increases with filler content, with 5-ATLP-0.063 showing 86.21 MPa (↓27.1% from neat PVC) and 20-ATLP-0.125 showing 342.49 MPa (↑189.5% from neat PVC). The particle size also influences the bending modulus, with

0.125 mm particles generally providing higher bending modulus than 0.063 mm particles. For acetic acid-treated fibers (ACLP), the bending modulus increases with filler content, with 5-ACLP-0.063 showing 183.30 MPa (↑54.9% from neat PVC) and 15-ACLP-0.125 showing 474.46 MPa (↑301.0% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing higher bending modulus than 0.063 mm particles.

The bending modulus of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicellulose, which hinder effective bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows strong peaks for hemicellulose, lignin, and other non-cellulosic materials, which contribute to the poor compatibility with the PVC matrix. The SEM images reveal a rough and uneven surface with visible impurities, further supporting the poor filler-matrix interaction. Despite these limitations, untreated fibers provide some improvement in bending modulus at higher filler percentages, likely due to their natural rigidity and the increased filler content providing additional reinforcement.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicellulose. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. The removal of impurities also reduces the formation of voids and enhances the overall packing of the composite, leading to improved bending modulus. The increase in bending modulus is particularly pronounced at higher filler percentages, as the alkaline-treated fibers integrate more effectively into the matrix, providing additional reinforcement and reducing weak interfacial bonding. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicellulose and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix, but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes any residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in bending modulus. The acetic acid-treated fibers show the highest bending modulus at higher filler percentages, as the partial removal of impurities and improved filler-matrix interaction enhance the reinforcement of the PVC matrix. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicellulose and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

b) Bending strength

The bending strength results of neat PVC and its composites, combined with insights from FTIR and SEM analyses, provide a comprehensive understanding of how the incorporation of date palm leaflet powder (DPLP) and its treatment (untreated, alkaline-treated, or acetic acid-treated) influence the mechanical strength, chemical composition, and morphological properties of the PVC matrix. The bending strength of neat PVC is 4.199 MPa, serving as the baseline for comparison. When DPLP is added to the PVC matrix, the bending strength changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology. For untreated leaflet powder (ULP) composites, the bending strength ranges from 3.099 MPa to 7.275 MPa, showing both decreases and increases compared to neat PVC. At lower percentages (5% and 10%), the bending strength is slightly lower than neat PVC, with 5-ULP-0.063 showing a bending strength of 3.099 MPa (↓26.2% from neat PVC) and 10-ULP-0.125 showing 5.061 MPa (↑20.5% from neat PVC). This suggests that the addition of untreated fibers can reduce the bending strength of the composites at lower percentages due to poor filler-matrix interaction. As the percentage of ULP increases (from 15% to 20%), the bending strength further increases, with 20-ULP-0.063 showing 7.275 MPa (↑73.3% from neat PVC) and 20-ULP-0.125 showing 6.809 MPa (↑62.2% from neat PVC). This increase is likely due to the higher filler content providing additional reinforcement to the PVC matrix. The particle size also plays a role, with 0.125 mm particles generally providing higher bending strength than 0.063 mm particles.

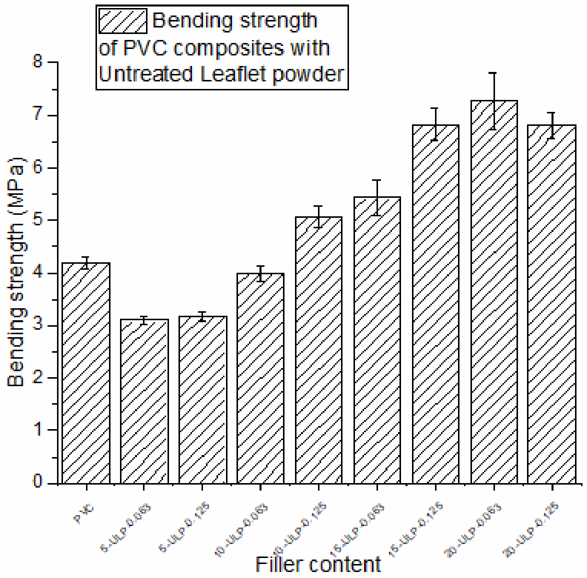

Figure 108 Bending strength results of PVC composites with ULP in function of filler content and filler particle size

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit significant improvements in bending strength, ranging from 3.920 MPa to 10.030 MPa, representing an increase of up to 139.0% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows a bending strength of 3.920 MPa (↓6.6% from neat PVC), while 20-ATLP-0.125 shows 10.030 MPa (↑139.0% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the bending strength increases significantly, indicating that the alkaline treatment enhances the strength of the composites, particularly at higher filler percentages. The particle size also influences the bending strength, with 0.125 mm particles generally providing higher bending strength than 0.063 mm particles.

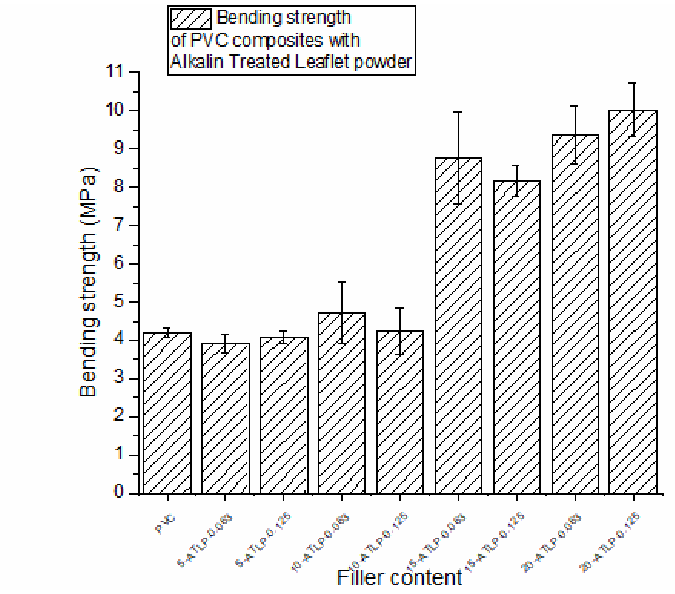

Figure 109 Bending strength results of PVC composites with ATLP in function of filler content and filler particle size

The acetic acid-treated leaflet powder (ACLP) composites show the highest bending strength, ranging from 5.249 MPa to 12.699 MPa, representing an increase of 25.0% to 202.5% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.063 shows a bending strength of 5.302 MPa (↑26.3% from neat PVC), while 15-ACLP-0.125 shows 12.699 MPa (↑202.5% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the bending strength increases significantly, indicating that the acetic acid treatment enhances the strength of the composites, particularly at higher filler percentages. The particle size also plays a role, with 0.125 mm particles generally providing higher bending strength than 0.063 mm particles.

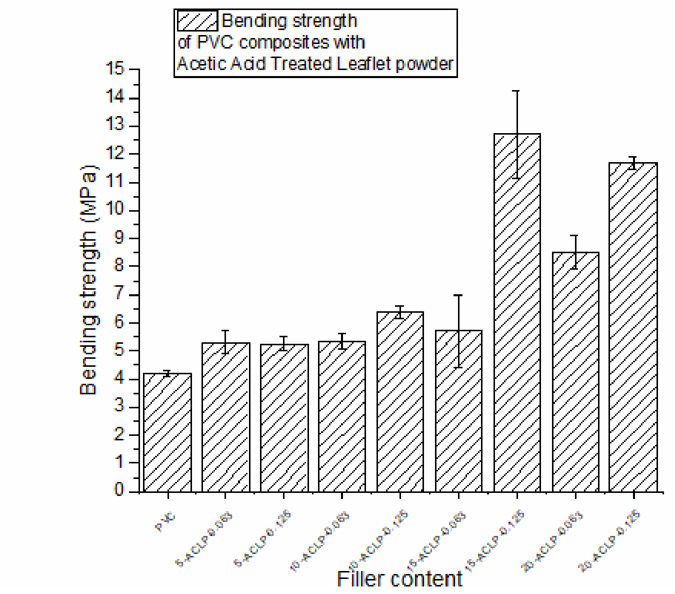

Figure 110 Bending strength results of PVC composites with ACLP in function of filler content and filler particle size

Comparison of Treatment Effects at Fixed Filler Content

To better understand the impact of treatment type, we compare the bending strength of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.063 shows a bending strength of 3.099 MPa (↓26.2% from neat PVC), while 5-ATLP-0.063 shows 3.920 MPa (↓6.6% from neat PVC), and 5-ACLP-0.063 shows 5.302 MPa (↑26.3% from neat PVC). This comparison shows that acetic acid treatment provides the highest bending strength, followed by alkaline treatment, while untreated fibers show the lowest bending strength. For 10% filler content, 10-ULP-0.125 shows a bending strength of 5.061 MPa (↑20.5% from neat PVC), while 10-ATLP-0.125 shows 4.237 MPa (↑0.9% from neat PVC), and 10-ACLP-0.125 shows 6.390 MPa (↑52.2% from neat PVC). Here, acetic acid treatment provides the highest bending strength, followed by untreated fibers and alkaline treatment. For 20% filler content, 20-ULP-0.125 shows a bending strength of 6.809 MPa (↑62.2% from neat PVC), while 20-ATLP-0.125 shows 10.030 MPa (↑139.0% from neat PVC), and 20-ACLP-0.125 shows 11.701 MPa (↑178.8% from neat PVC). At higher filler percentages, acetic acid treatment provides the highest bending strength, while alkaline treatment also shows significant improvement.

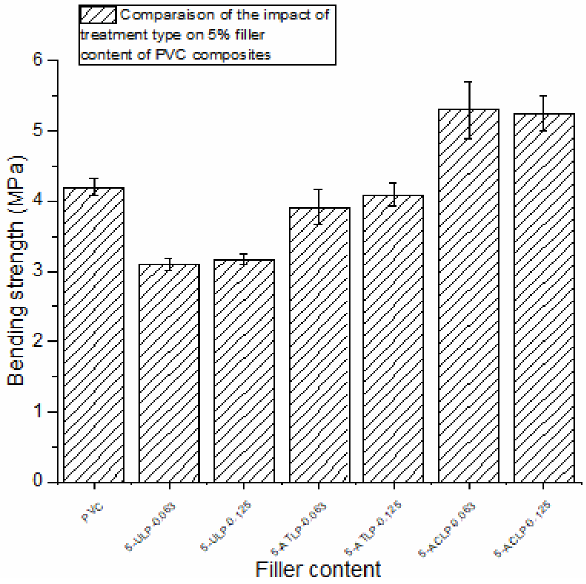

Figure 111 Comparison of the impact of treatment type on 5% filler content of PVC composites bending strength

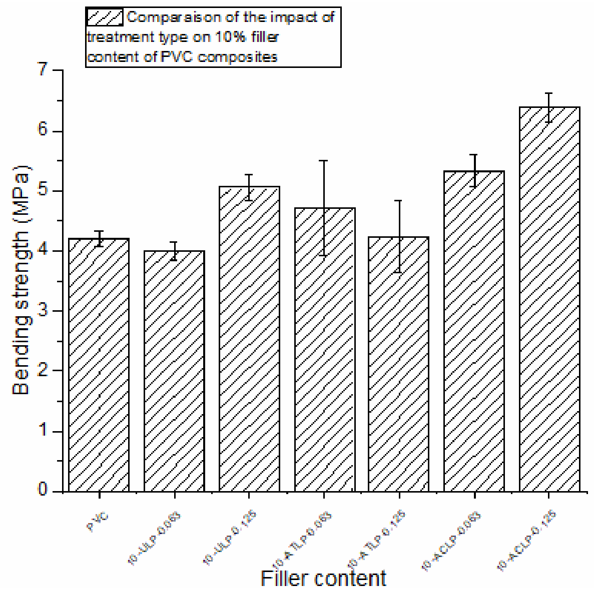

Figure 112 Comparison of the impact of treatment type on 10% filler content of PVC composites bending strength

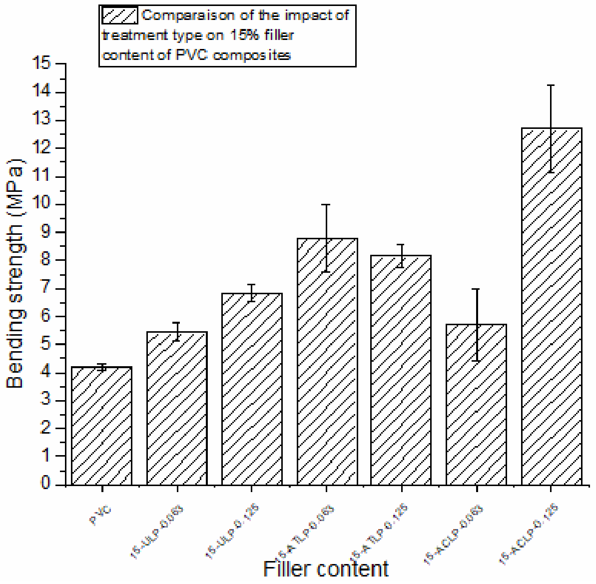

Figure 113 Comparison of the impact of treatment type on 15% filler content of PVC composites bending strength

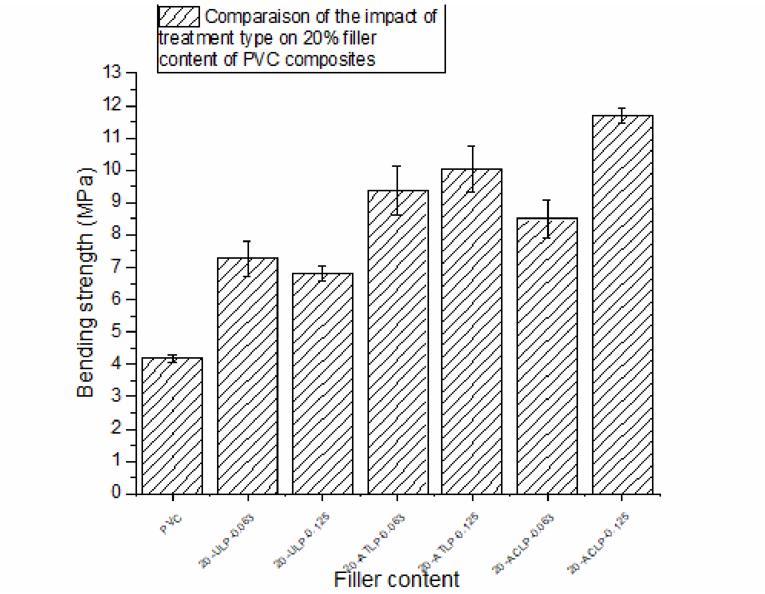

Figure 114 Comparison of the impact of treatment type on 20% filler content of PVC composites bending strength

Comparison of Filler Content and Size Effects at Fixed Treatment Type

To better understand the impact of filler content and size, we compare the bending strength of composites with the same treatment but different filler content and size. For untreated fibers (ULP), the bending strength increases with filler content, with 5-ULP-0.063 showing 3.099 MPa (↓26.2% from neat PVC) and 20-ULP-0.125 showing 6.809 MPa (↑62.2% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing higher bending strength than 0.063 mm particles. For alkaline-treated fibers (ATLP), the bending strength increases with filler content, with 5-ATLP-0.063 showing 3.920 MPa (↓6.6% from neat PVC) and 20-ATLP-0.125 showing 10.030 MPa (↑139.0% from neat PVC). The particle size also influences the bending strength, with 0.125 mm particles generally providing higher bending strength than 0.063 mm particles. For acetic acid-treated fibers (ACLP), the bending strength increases with filler content, with 5-ACLP-0.063 showing 5.302 MPa (↑26.3% from neat PVC) and 15-ACLP-0.125 showing 12.699 MPa (↑202.5% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing higher bending strength than 0.063 mm particles.

The bending strength of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicellulose, which hinder effective bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows strong peaks for hemicellulose, lignin, and other non-cellulosic materials, which contribute to the poor compatibility with the PVC matrix. The SEM images reveal a rough and uneven surface with visible impurities, further supporting the poor filler-matrix interaction. Despite these limitations, untreated fibers provide some improvement in bending strength at higher filler percentages, likely due to their natural rigidity and the increased filler content providing additional reinforcement.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicellulose. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. The removal of impurities also reduces the formation of voids and enhances the overall packing of the composite, leading to improved bending strength. The increase in bending strength is particularly pronounced at higher filler percentages, as the alkaline-treated fibers integrate more effectively into the matrix, providing additional reinforcement and reducing weak interfacial bonding. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicellulose and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix, but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes any residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in bending strength. The acetic acid-treated fibers show the highest bending strength at higher filler percentages, as the partial removal of impurities and improved filler-matrix interaction enhance the reinforcement of the PVC matrix. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicellulose and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

c) Bending strain

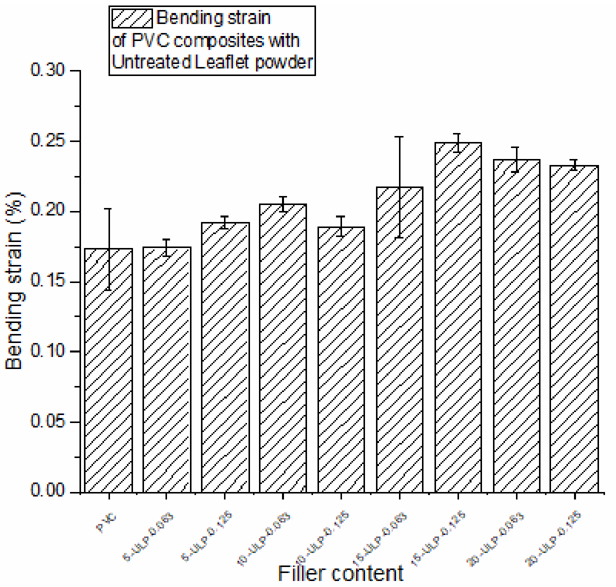

Figure 115 Bending strain results of PVC composites with ULP in function of filler content and filler particle size

The bending strain results of neat PVC and its composites, combined with insights from FTIR and SEM analyses, provide a comprehensive understanding of how the incorporation of date palm leaflet powder (DPLP) and its treatment (untreated, alkaline-treated, or acetic acid-treated) influence the ductility, chemical composition, and morphological properties of the PVC matrix. The bending strain of neat PVC is 0.173, serving as the baseline for comparison. When DPLP is added to the PVC matrix, the bending strain changes depending on the type of treatment, particle size, and filler percentage, reflecting variations in filler-matrix interaction, chemical bonding, and fiber morphology. For untreated leaflet powder (ULP) composites, the bending strain ranges from 0.174 to 0.249, showing both increases and decreases compared to neat PVC. At lower percentages (5% and 10%), the bending strain is slightly higher than neat PVC, with 5-ULP-0.063 showing a bending strain of 0.174 (↑0.5% from neat PVC) and 10-ULP-0.125 showing 0.189 (↑9.1% from neat PVC). This suggests that the addition of untreated fibers can enhance the ductility of the composites at lower percentages due to their natural flexibility. As the percentage of ULP increases (from 15% to 20%), the bending strain further increases, with 20-ULP-0.063 showing 0.237 (↑36.7% from neat PVC) and 20-ULP-0.125 showing 0.233 (↑34.5% from neat PVC). This increase is likely due to the higher filler content providing additional reinforcement to the PVC matrix. The particle size also plays a role, with 0.125 mm particles generally providing higher bending strain than 0.063 mm particles.

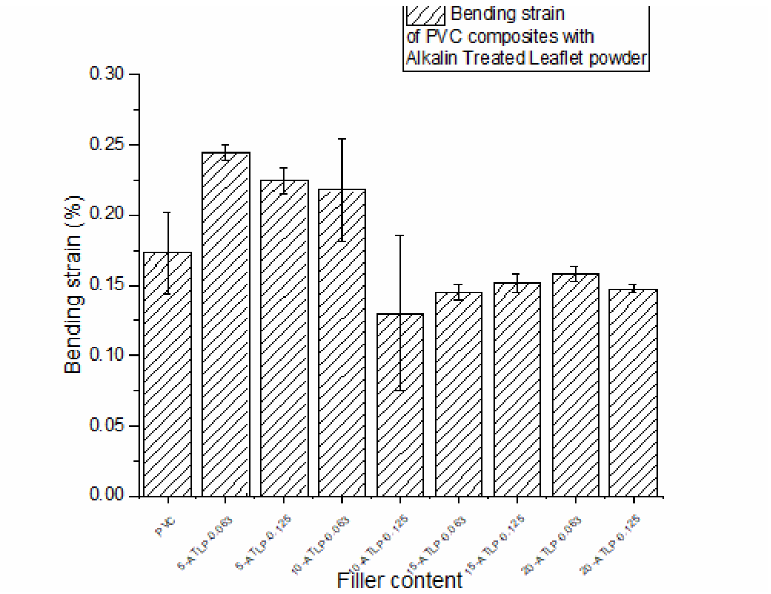

Figure 116 Bending strain results of PVC composites with ATLP in function of filler content and filler particle size

In contrast, alkaline-treated leaflet powder (ATLP) composites exhibit a decrease in bending strain, ranging from 0.130 to 0.244, representing a decrease of 24.9% to 41.0% compared to neat PVC. The alkaline treatment removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ATLP-0.063 shows a bending strain of 0.244 (↑40.9% from neat PVC), while 20-ATLP-0.125 shows 0.148 (↓14.6% from neat PVC). As the percentage of ATLP increases (from 5% to 20%), the bending strain decreases, indicating that the alkaline treatment reduces the ductility of the composites. The particle size also influences the bending strain, with 0.063 mm particles generally providing higher bending strain than 0.125 mm particles.

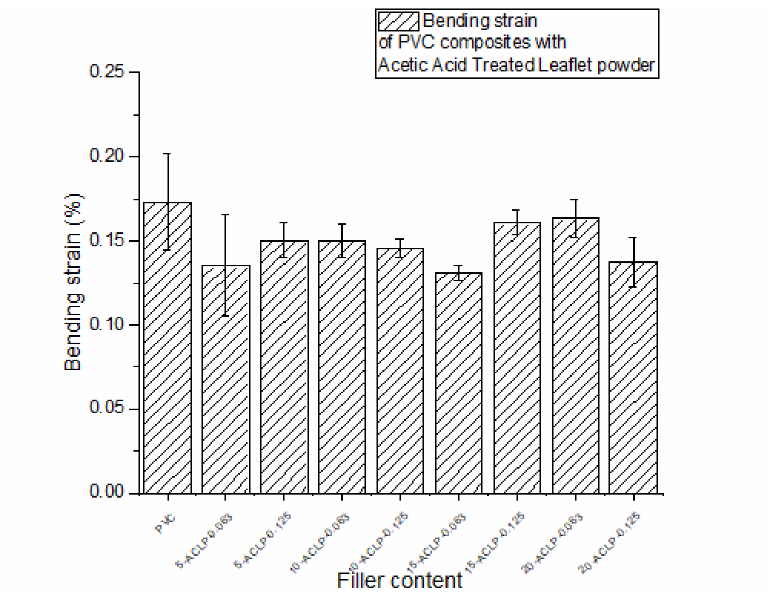

Figure 117 Bending strain results of PVC composites with ACLP in function of filler content and filler particle size

The acetic acid-treated leaflet powder (ACLP) composites show a decrease in bending strain, ranging from 0.131 to 0.164, representing a decrease of 24.3% to 20.6% compared to neat PVC. The acetic acid treatment partially removes non-cellulosic materials, improving the compatibility between the fibers and the PVC matrix. For example, 5-ACLP-0.063 shows a bending strain of 0.135 (↓22.1% from neat PVC), while 20-ACLP-0.125 shows 0.137 (↓20.9% from neat PVC). As the percentage of ACLP increases (from 5% to 20%), the bending strain decreases, indicating that the acetic acid treatment reduces the ductility of the composites. The particle size also plays a role, with 0.125 mm particles generally providing higher bending strain than 0.063 mm particles.

Comparison of Treatment Effects at Fixed Filler Content

To better understand the impact of treatment type, we compare the bending strain of composites with the same filler content but different treatments. For 5% filler content, 5-ULP-0.063 shows a bending strain of 0.174 (↑0.5% from neat PVC), while 5-ATLP-0.063 shows 0.244 (↑40.9% from neat PVC), and 5-ACLP-0.063 shows 0.135 (↓22.1% from neat PVC). This comparison shows that alkaline treatment provides the highest bending strain, followed by untreated fibers, while acetic acid treatment shows the lowest bending strain. For 10% filler content, 10-ULP-0.125 shows a bending strain of 0.189 (↑9.1% from neat PVC), while 10-ATLP-0.125 shows 0.130 (↓24.9% from neat PVC), and 10-ACLP-0.125 shows 0.146 (↓15.7% from neat PVC). Here, untreated fibers provide the highest bending strain, followed by acetic acid treatment and alkaline treatment. For 20% filler content, 20-ULP-0.125 shows a bending strain of 0.233 (↑34.5% from neat PVC), while 20-ATLP-0.125 shows 0.148 (↓14.6% from neat PVC), and 20-ACLP-0.125 shows 0.137 (↓20.9% from neat PVC). At higher filler percentages, untreated fibers provide the highest bending strain, followed by alkaline treatment and acetic acid treatment.

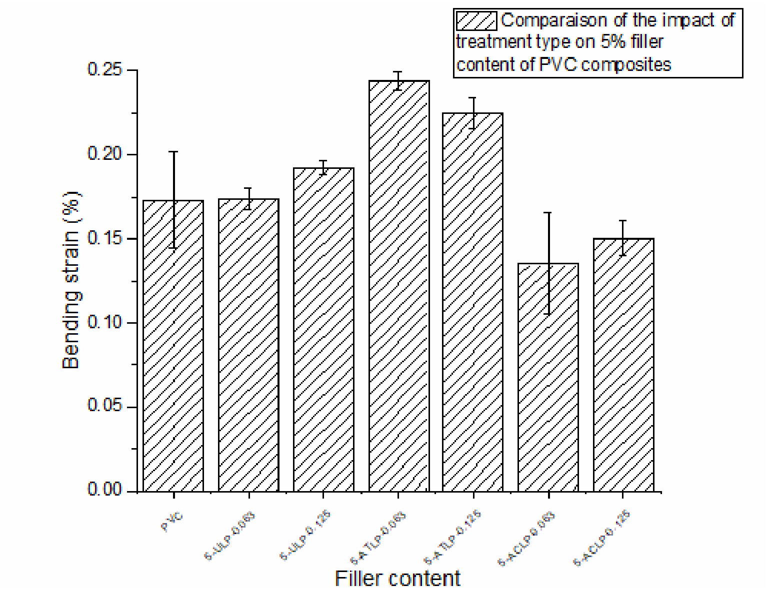

Figure 118 Comparison of the impact of treatment type on 5% filler content of PVC composites bending strain

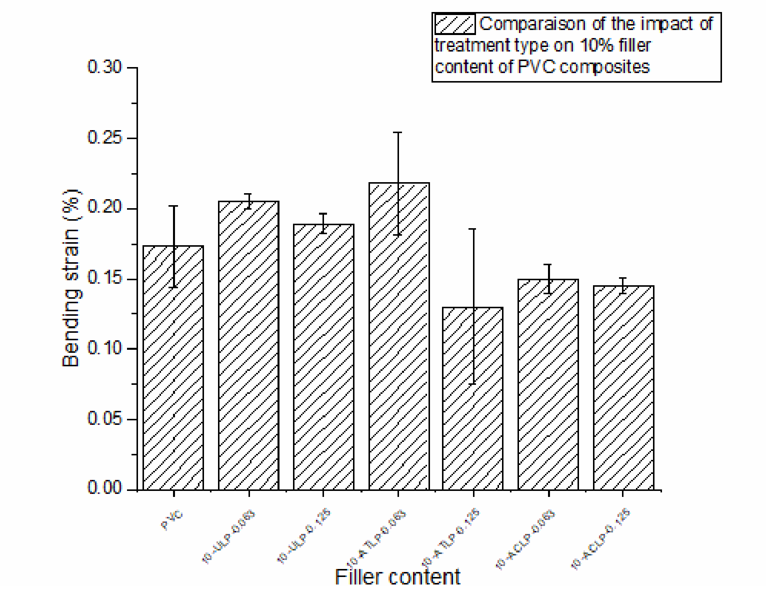

Figure 119 Comparison of the impact of treatment type on 10% filler content of PVC composites bending strain

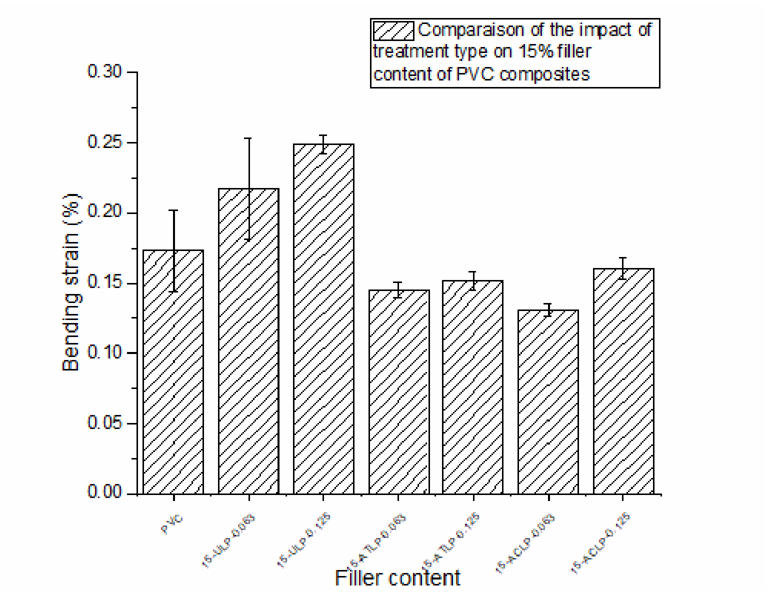

Figure 120 Comparison of the impact of treatment type on 15% filler content of PVC composites bending strain

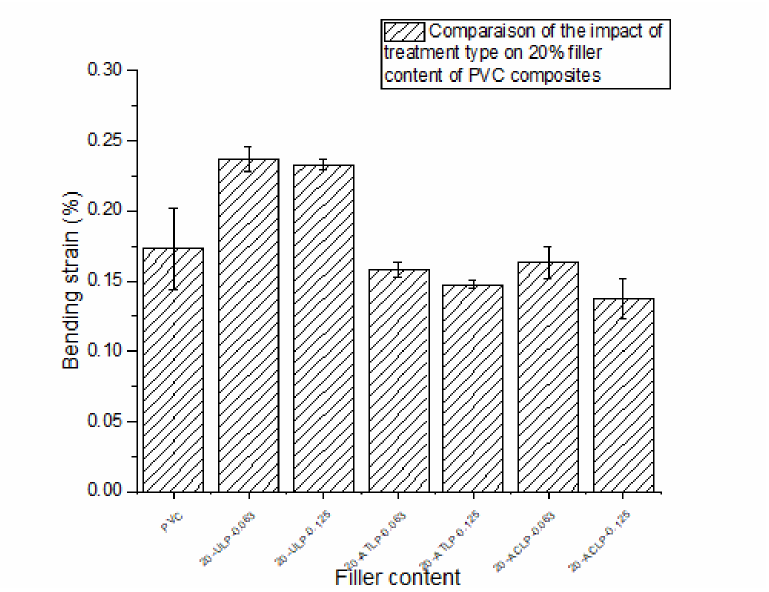

Figure 121 Comparison of the impact of treatment type on 20% filler content of PVC composites bending strain

Comparison of Filler Content and Size Effects at Fixed Treatment Type

To better understand the impact of filler content and size, we compare the bending strain of composites with the same treatment but different filler content and size. For untreated fibers (ULP), the bending strain increases with filler content, with 5-ULP-0.063 showing 0.174 (↑0.5% from neat PVC) and 20-ULP-0.125 showing 0.233 (↑34.5% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing higher bending strain than 0.063 mm particles. For alkaline-treated fibers (ATLP), the bending strain decreases with filler content, with 5-ATLP-0.063 showing 0.244 (↑40.9% from neat PVC) and 20-ATLP-0.125 showing 0.148 (↓14.6% from neat PVC). The particle size also influences the bending strain, with 0.063 mm particles generally providing higher bending strain than 0.125 mm particles. For acetic acid-treated fibers (ACLP), the bending strain decreases with filler content, with 5-ACLP-0.063 showing 0.135 (↓22.1% from neat PVC) and 20-ACLP-0.125 showing 0.137 (↓20.9% from neat PVC). The particle size also plays a role, with 0.125 mm particles generally providing higher bending strain than 0.063 mm particles.

The bending strain of the composites is closely related to the compatibility between the DPLP and the PVC matrix, which is influenced by the type of treatment. Untreated fibers (ULP) retain their natural impurities, such as waxes, lignin, and hemicellulose, which hinder effective bonding with the PVC matrix. This results in poor filler-matrix interaction, leading to the formation of voids and weak interfacial bonding. The FTIR analysis of untreated fibers shows strong peaks for hemicellulose, lignin, and other non-cellulosic materials, which contribute to the poor compatibility with the PVC matrix. The SEM images reveal a rough and uneven surface with visible impurities, further supporting the poor filler-matrix interaction. Despite these limitations, untreated fibers provide some improvement in bending strain at higher filler percentages, likely due to their natural flexibility and the increased filler content providing additional reinforcement.

Alkaline treatment (ATLP) significantly improves the compatibility between the fibers and the PVC matrix by removing non-cellulosic materials such as lignin and hemicellulose. This exposes more cellulose, which has better mechanical properties and forms stronger bonds with the PVC matrix. The removal of impurities also reduces the formation of voids and enhances the overall packing of the composite, leading to improved bending strain. The increase in bending strain is particularly pronounced at higher filler percentages, as the alkaline-treated fibers integrate more effectively into the matrix, providing additional reinforcement and reducing weak interfacial bonding. The FTIR analysis of alkaline-treated fibers shows a reduction in peaks corresponding to hemicellulose and lignin, indicating the removal of these non-cellulosic materials. The SEM images reveal a cleaner and smoother surface with increased visibility of microfibrils, further supporting the improved filler-matrix interaction.

Acetic acid treatment (ACLP) also improves the compatibility between the fibers and the PVC matrix, but to a lesser extent than alkaline treatment. The acetic acid treatment partially removes non-cellulosic materials and neutralizes any residual alkali, resulting in a cleaner fiber surface with improved mechanical properties. However, the effect is less aggressive than alkaline treatment, leading to higher variability in bending strain. The acetic acid-treated fibers show the lowest bending strain among the treated fibers, as the partial removal of impurities and improved filler-matrix interaction may not be sufficient to compensate for the stiffening effect of the chemical modification. The FTIR analysis of acetic acid-treated fibers shows a partial reduction in peaks corresponding to hemicellulose and lignin, indicating the partial removal of these non-cellulosic materials. The SEM images reveal a moderately clean surface with some residual impurities, further supporting the partial improvement in filler-matrix interaction.

III.3. SCA treated Results:

III.3.1. FTIR results

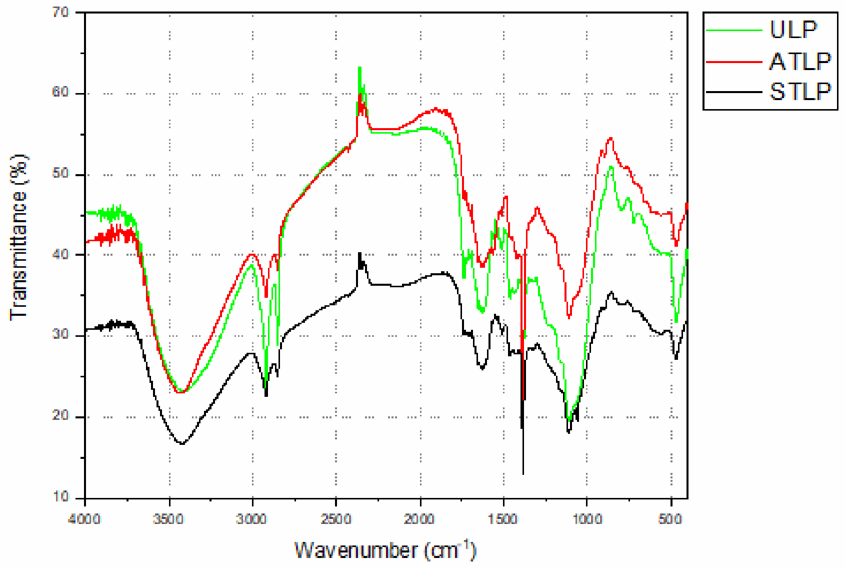

Figure 122 FTIR spectra of ULP, ATLP and STLP

The infrared spectra of untreated leaflets powder (ULP), soda (ATLP), and silane coupling agent (STLP) treated are shown in Figure 119. The peak around 3406 cm^{-1} corresponds to all the hydroxyls (primary and secondary), which have a slight decrease in intensity after modification. This is explained by converting some primary alcohols into a carboxylic acid. The peak at 709 cm^{-1}, which reflects the group's deformation (-OH), also vanishes. The bands centred around 2920 and 2854 cm^{-1}, which reflect the symmetrical and asymmetrical elongation vibrations of the C-H bonds of the -CH2 group of the cellulose and lignin segments, are also decreasing.The peaks corresponding to the C=O carbonyl groups of lignin (1735, 1238 cm^{-1}) were no longer observed after alkaline treatment (ATLP). This is explained by the partial hydrolysis of hemicelluloses in an alkaline medium, characterized by the rupture of C-O-C bonds between two monomers. Peak intensity at 1509 cm^{-1} indicates the decrease of C=C groups. The presence of lignin peaks in the treated fibre spectrum shows that the fibres still contain some lignin. Mercerization removes the waxy epidermal tissue, sticky pectin, and hemicelluloses that bind fibre bundles together, as well as pectin and hemicelluloses-rich sheaths in the nucleus. Equation 2 describes the reaction between cellulose and soda:

Cellulose-OH + NaOH →Cellulose-O-Na+ + H2O + impurities (2)

For the spectrum of leaflet powder treated with the silane coupling agent (STLP), the same peaks as recorded for ATLP can be seen. But new groups assigned to the Si-groups O-cellulose or Si-O-Siin the region between 1050 and 1100 cm^{-1}appear. This confirms the substitution of the OH groups of hemicelluloses by silanol groups (obtained during the hydrolysis of silanes) by a condensation reaction. The signature of the silane grafting reaction on the powder indicates that intermolecular condensation occurred between adjacent silanol groups. The band at 807 cm^{-1} represents the Si-O bond from the reaction between the silane and the powder. The silanization reaction of date palm fibres with silane as a modifying agent was confirmed by the FTIR spectroscopic analysis and the reaction mechanismsare presented in figure 120:

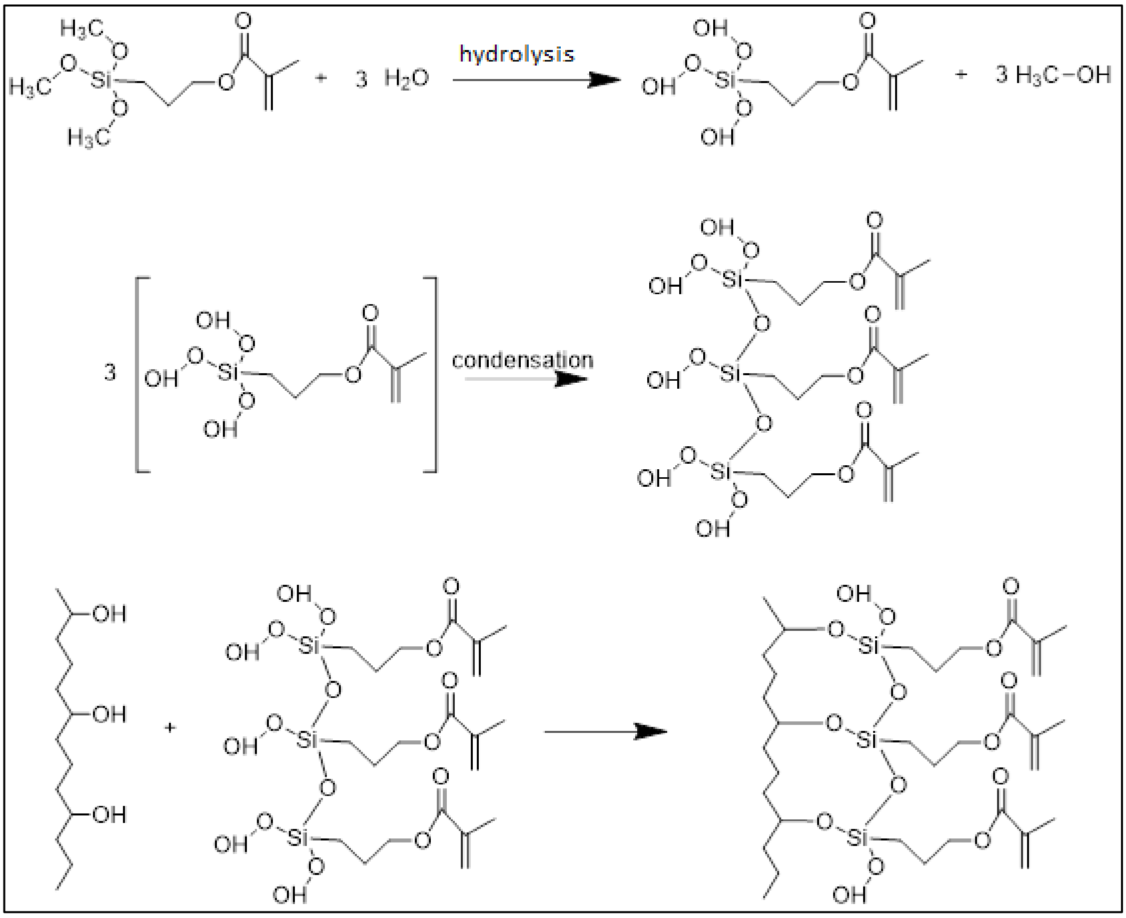

Figure 123Reactionnal mechanism between date palm leaflet powder and silane (3-triméthoxysilyl) propyl méthacrylate.

III.3.2. Tensile test

a) Young's modulus

Figures 121, 122 and 123 illustrate the evolution of the Young's modulus of the PVC composites with different filler treatment. In general, the Young’s modulus increases with filler content; i.e. adding the fillers into the PVC matrix increases the rigidity of the composites. This behavior is attributed to the rigidity of the dispersed filler, which is transfered to the final composites. The results also show that the surface treatments perform better than the untreated particles. Specifically, PVC with 5% STLP exhibits the highet Young’s modulus with 151 MPa, followed by PVC with 5% ATLP with 118 MPa, and PVC with 5% ULP 92.3 MPa. Similary, at a higher filler content of 25%, PVC with STLP outperforms PVC with ATLP and PVC with ULP by 221.08 MPa, 208.13 MPa and 169.33 MPa, respectively. In all cases, mercerization provides better results, but the silane treatment gives the best results. This increase is mainly due to a better fibre/matrix interfacial adhesion due to the excellent dispersion of the treated fibre in the PVC matrix. The treatment resulted in the formation of chemical bonds between the hydrophobic part of the silane and the matrix surface and the hydrophilic part with the fibre surface.

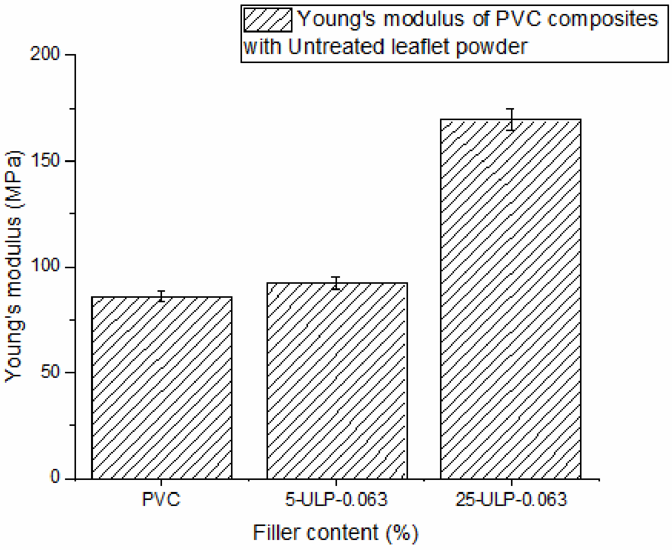

Figure 124 Young's modulus results of PVC composites with ULP in function of filler content and filler particle size

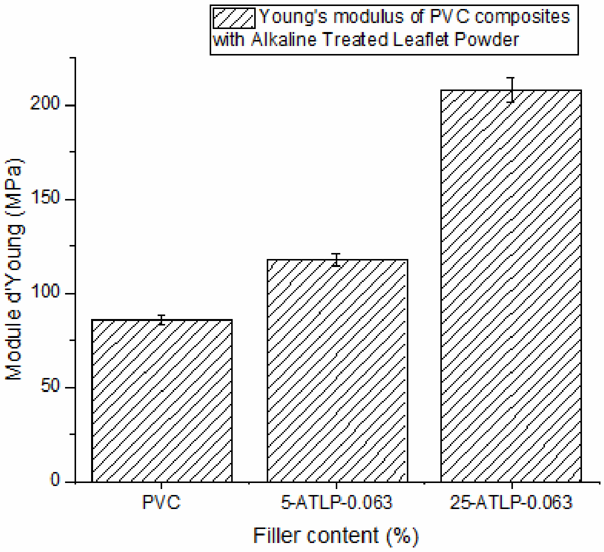

Figure 125 Young's modulus results of PVC composites with ATLP in function of filler content and filler particle size

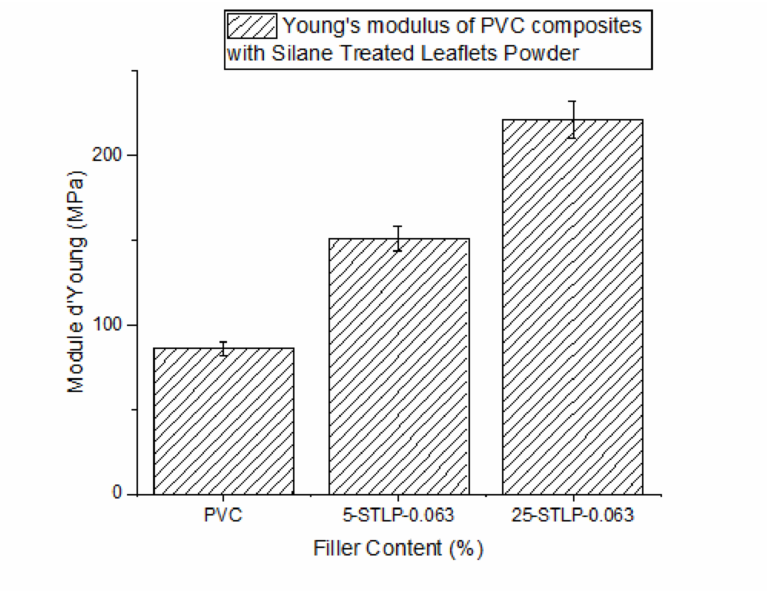

Figure 126 Young's modulus results of PVC composites with STLP in function of filler content and filler particle size

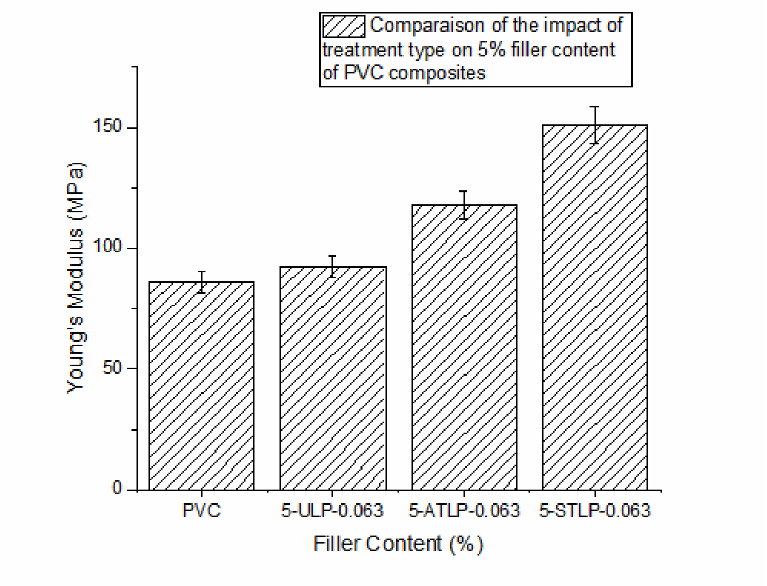

Figure 127 Comparison of the impact of treatment type on 5% filler content of PVC composites Young's modulus

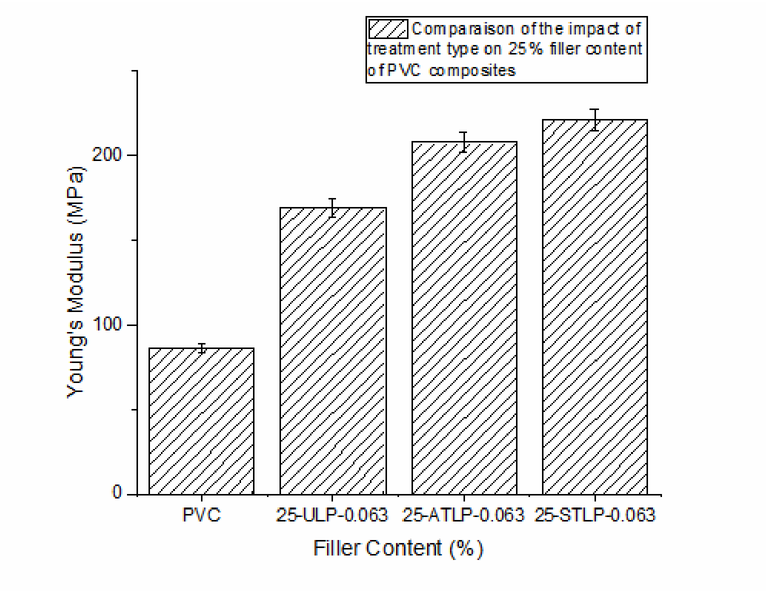

Figure 128 Comparison of the impact of treatment type on 25% filler content of PVC composites Young's modulus

b) ***Tensile strength***

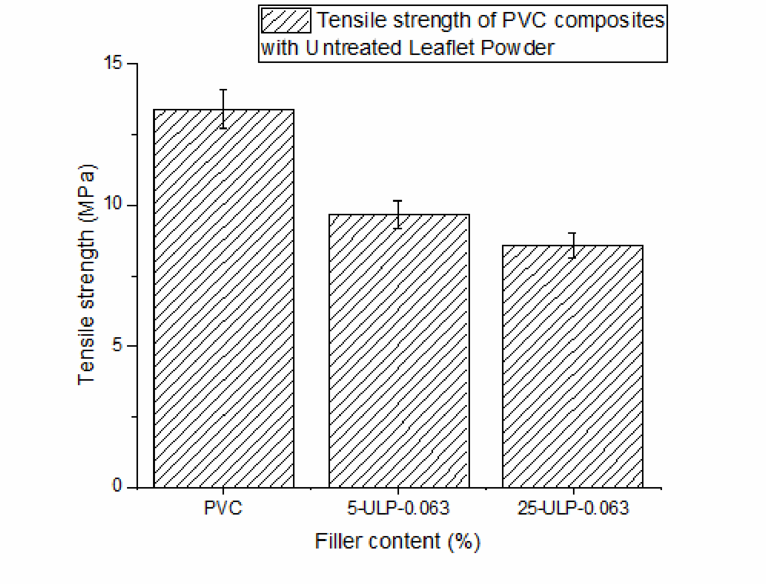

Figure 129 Tensile strength results of PVC composites with ULP in function of filler content and filler particle size

Figures 126, 127 and 128shows the evolution of the tensile strength of the composites produced as a function of filler content. A noticeable decrease in the fracture stress for the composites is observed compared to the neat matrix (PVC) which have 13.4 MPa. However, better results are observed for ATLP composites compared to PVC/FNT, at 5% filler content, ATLP composites reached 10.1 MPa and PVC/FNT reached 9.66 MPa that means: 24.62% and 27.91% decrease compared to neat matrix. Similary, at 25% filler content, ATLP outperforms ULP composites with 9.14 MPa against 8.58 MPa. Because of the hydrophobic nature of the PVC matrix and the hydrophilic nature of the filler (ULP), the binding force between the fibres and the matrix is originally weak and the fibres tend to agglomerates inducing heterogeneities and non-uniform stress transfer within the matrix, leading to embrittlement and a reduction in the composite's strength.

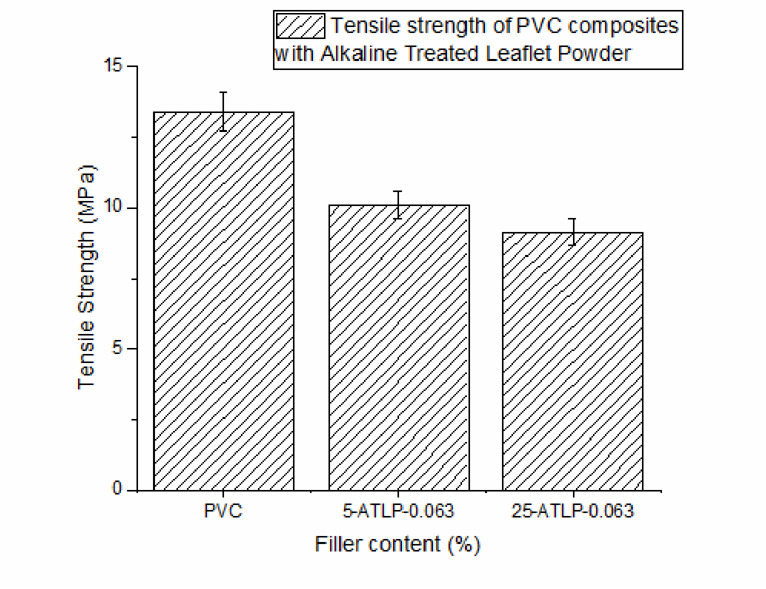

Figure 130 Tensile strength results of PVC composites with ATLP in function of filler content and filler particle size

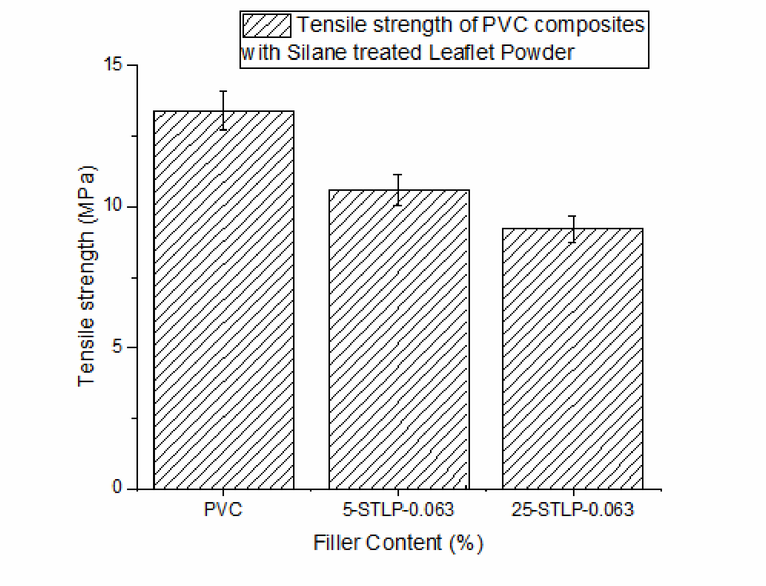

Figure 131 Tensile strength results of PVC composites with STLP in function of filler content and filler particle size

STLP composites had the best results in 5% and 25% filler content, the results revealed 10.6 MPa and 9.21 MPa, respectively. This is why the treatments improve the stress at the fracture of composites (ATLP and STLP) compared to untreated fibres (ULP). Better adhesion between the treated fibres and the polymeric matrix is achieved.

It is important to note that the alkaline treatment removes impurities and parietal constituents such as lignins, pectins and waxy substances covering the outer surface of the fibre cell walls. This promotes the formation of fibrils and gives a rough surface to the fibre to facilitate physico-chemical interactions at the filler-polymer interface. Figure 2 also shows that the composites treated with the silane coupling agent (STLP) have better tensile strength than mercerized ones (ATLP). This can be explained by the ability of silane to form strong interfacial bonds between the PVC and fibres. Therefore, a better stress transfer between both phases is achieved compared to alkaline treatment.

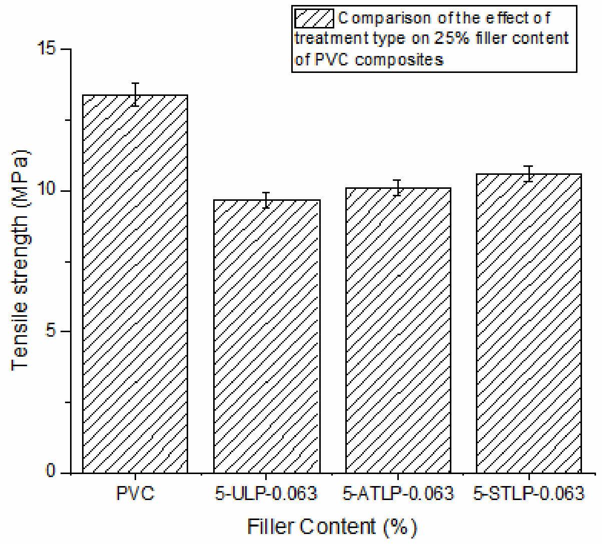

Figure 132 Comparison of the impact of treatment type on 5% filler content of PVC composites Tensile strength

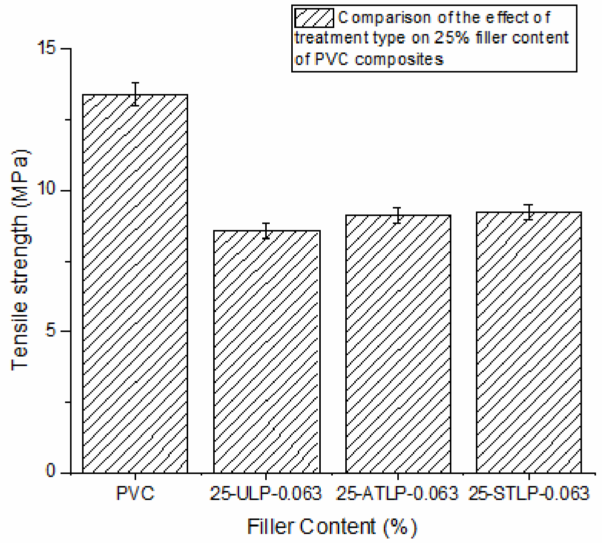

Figure 133 Comparison of the impact of treatment type on 25% filler content of PVC composites Tensile strength

c) ***Elongation at break***

The evolution of the elongation at fracture of the treated and untreated composites is represented in Figures 131; 132 and 133. There is a significant decrease in the values for the composites made with untreated fibres to fall from 179.6% of the neat matrix to 100.3% and 31.2% for 5 and 25% filler content respectively. This decrease is explained by: i) the hydrophilic nature of untreated fibres that absorb more moisture and cause swelling in the PVC matrix, which causes the embrittlement of the material. ii) increasing volume of fibre creating defects in the system and reducing inter-chain interactions associated with fragile ductile variation in material behaviour.

Adding NaOH-treated fibres to significantly reduced the elongation at break as a function of the filler content. At 5% ATLP in PVC, the result was 73.5%, and at 25% ATLP in PVC the result was 34%. This is related to the presence of lignin in the fibres, generating cracks and probable composite failure due to the time and concentration of soda used in the fibre treatment not having a major effect on lignin elimination. In addition, fibre agglomeration may have contributed to creating stress concentration zones requiring less energy for crack initiation and propagation.

Comparing the elongation at fracture of the different composites, it is clear that the silane coupling agent treatment produced a slight improvement with 105.5% and 70.46% for 5 and 25% filler content respectively. This increase is mainly attributed to a better dispersion of the treated fibre, giving some flexibility to the material.

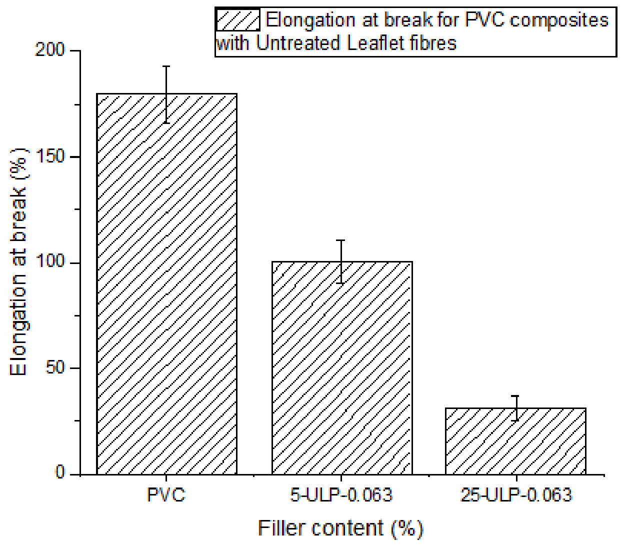

Figure 134 Elongation at break results of PVC composites with ULP in function of filler content and filler particle size

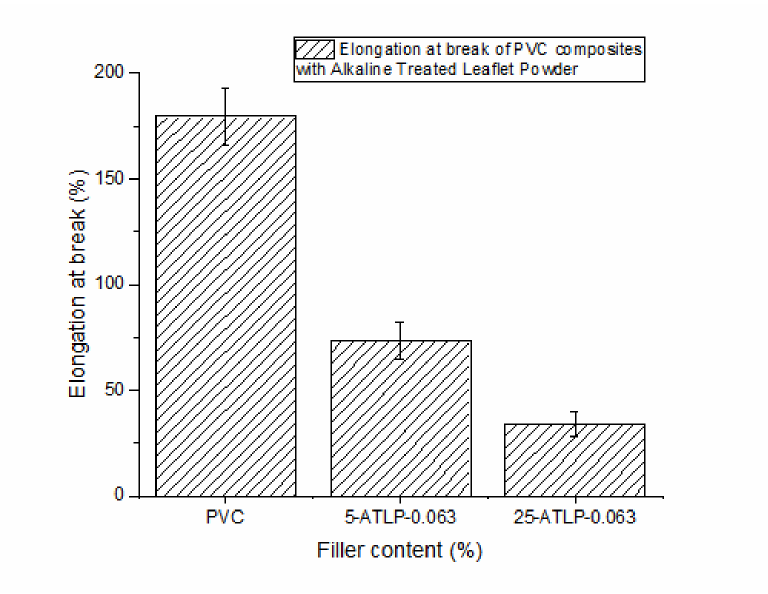

Figure 135 Elongation at break results of PVC composites with ATLP in function of filler content and filler particle size

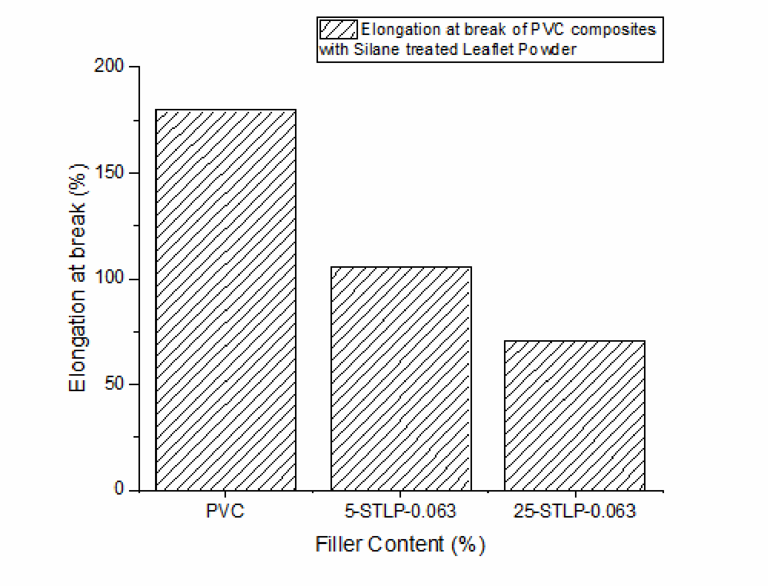

Figure 136 Elongation at break results of PVC composites with STLP in function of filler content and filler particle size

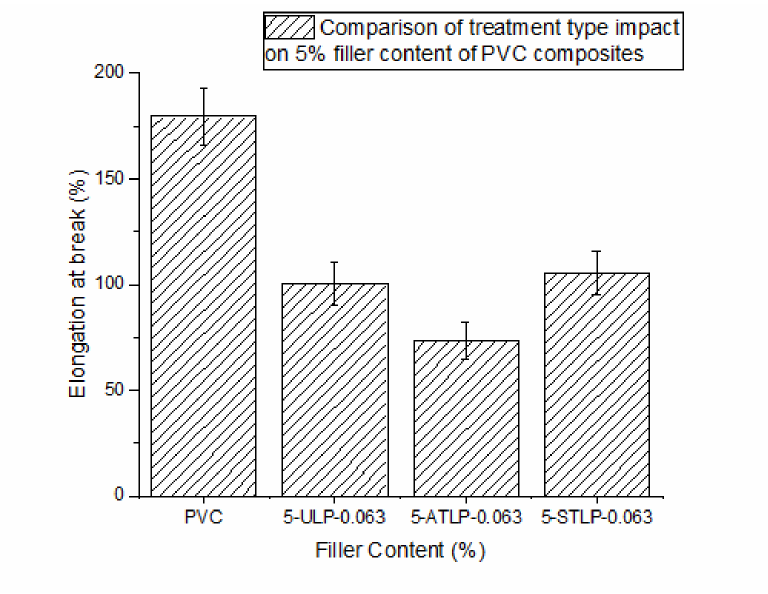

Figure 137 Comparison of the impact of treatment type on 5% filler content of PVC composites Elongation at break

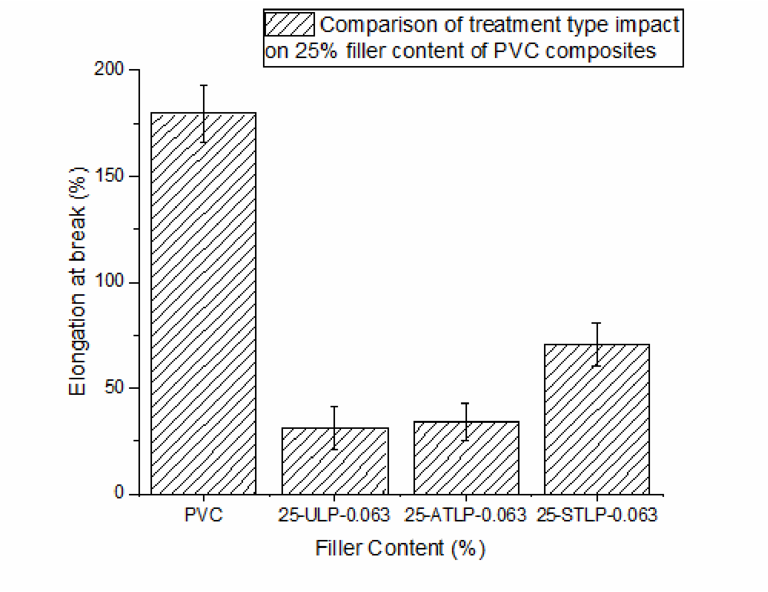

Figure 138 Comparison of the impact of treatment type on 25% filler content of PVC composites Elongation at break

III.3.3. Bending Test

a) Bending modulus

The bending modulus of the PVC composites reinforced by treated and untreated fibres is shown in Figure 136, 137 and 138. From the figures, it is evident that the incorporation of rigid fibers into the PVC matrix increased the bending modulus of the composites with respect to the virgin PVC. Comparing the values shows that the composites treated with the silane coupling agent have again the best properties. This is due improved interfacial adhesion compared to the other samples (ULP and ATLP).

The variation of these properties can be explained based on changes in chemical interaction at the filler-matrix interface on the different treatments, as described in the section above.

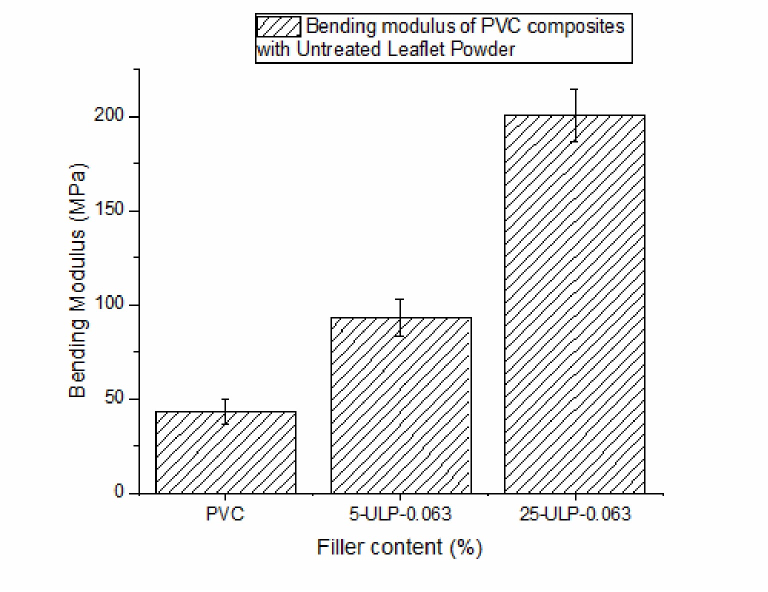

Figure 139 Bending modulus results of PVC composites with ULP in function of filler content and filler particle size

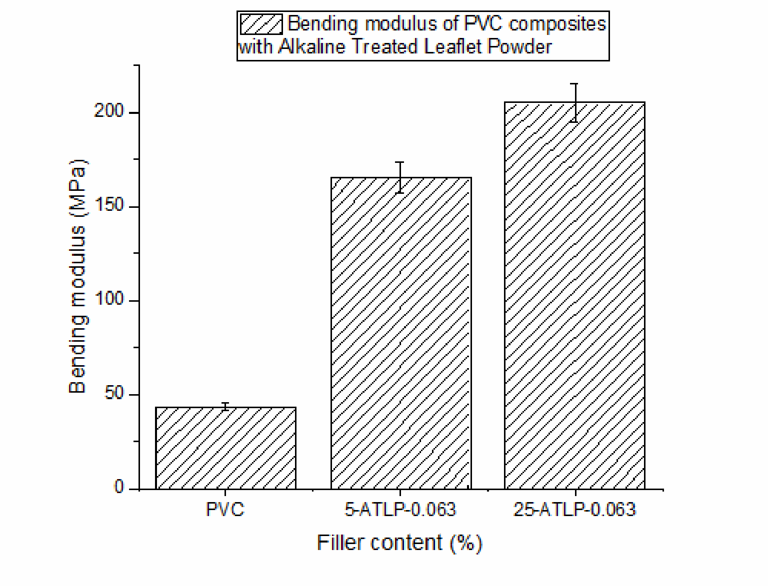

Figure 140 Bending modulus results of PVC composites with ATLP in function of filler content and filler particle size

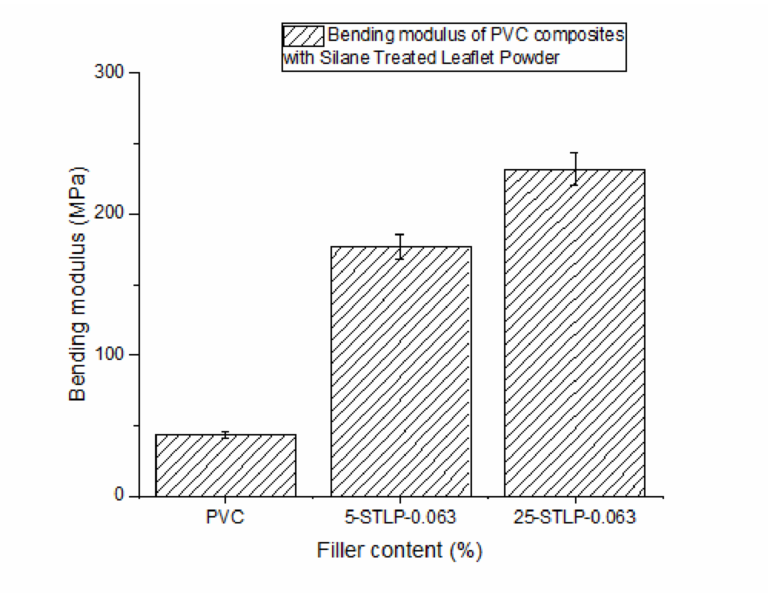

Figure 141 Bending modulus results of PVC composites with STLP in function of filler content and filler particle size

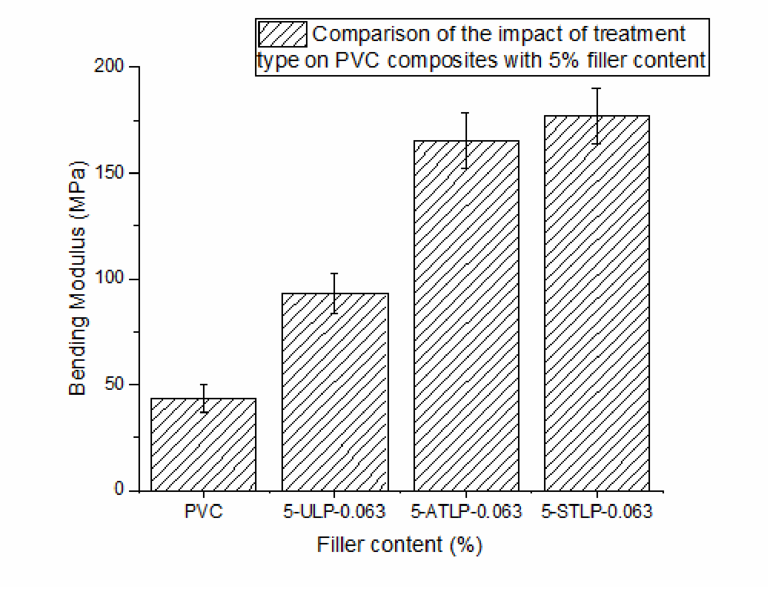

Figure 142 Comparison of the impact of treatment type on 5% filler content of PVC composites Bending modulus

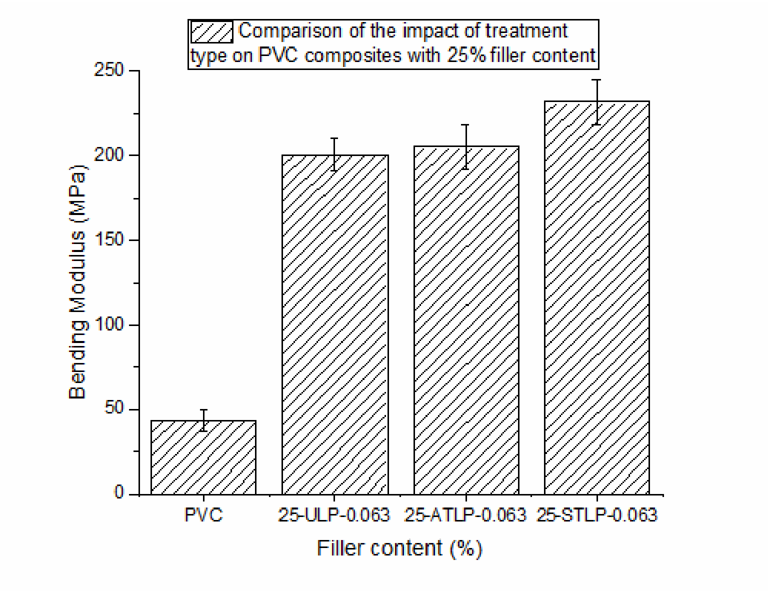

Figure 143 Comparison of the impact of treatment type on 25% filler content of PVC composites Bending modulus

b) Bending stress

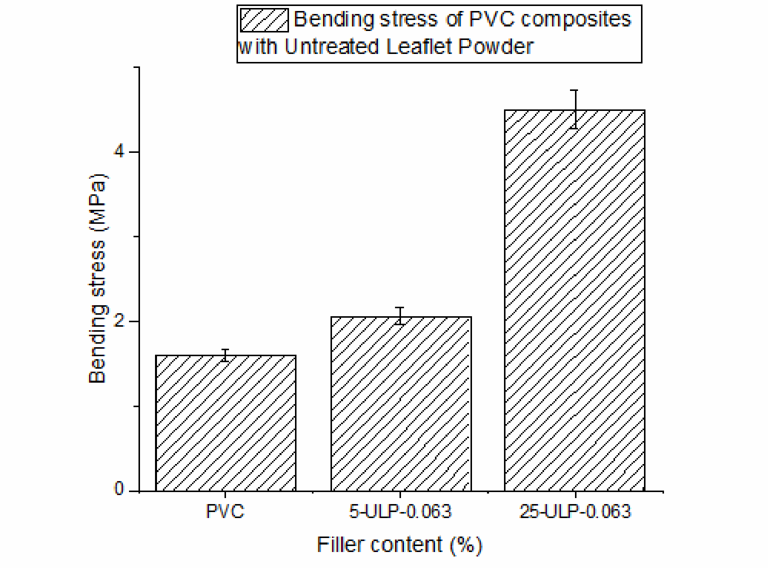

Figure 144 Bending stress results of PVC composites with ULP in function of filler content and filler particle size

The evolution of the bending stress of composites as a function of treated and untreated date palm leaflet powder is illustrated in Figures 141, 142 and 143. PVC composites reinforced with treated fibres have higher bending strength compared to PVC composites reinforced with untreated fibres. Palm fibre treatments improve the filler/matrix interface, resulting in higher bending properties.

The lower bending resistance of untreated composites is due to hydroxyl and other polar groups' presence on the fibre surface, resulting in a lower fibre/matrix interface quality. It should be noted that the combination of alkaline and silane treatments gives the best performance compared to individual treatments. This can be explained by the fact that after alkaline treatment, the network of hydrogen bonds within the cellulose structure can be broken, and the cellulose hydroxyl groups can become more active and thus improve the hydrophilic character of the fibres and their compatibility with silane agents.

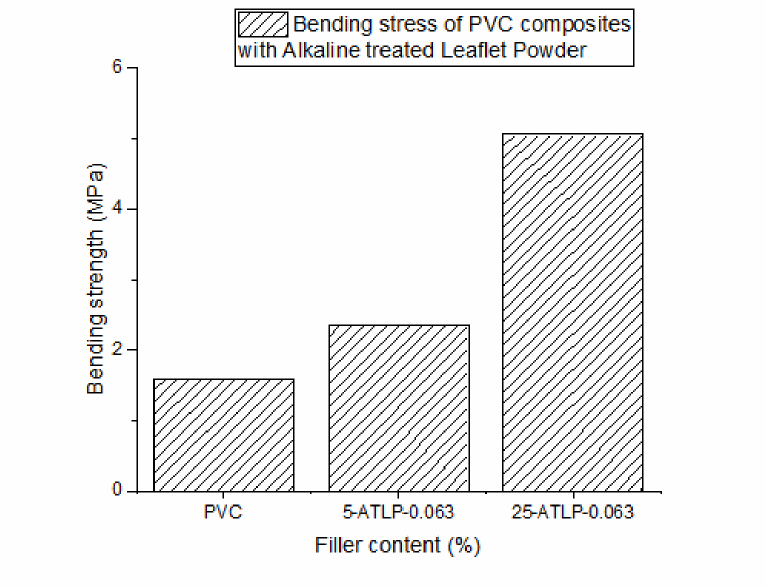

Figure 145 Bending stress results of PVC composites with ATLP in function of filler content and filler particle size

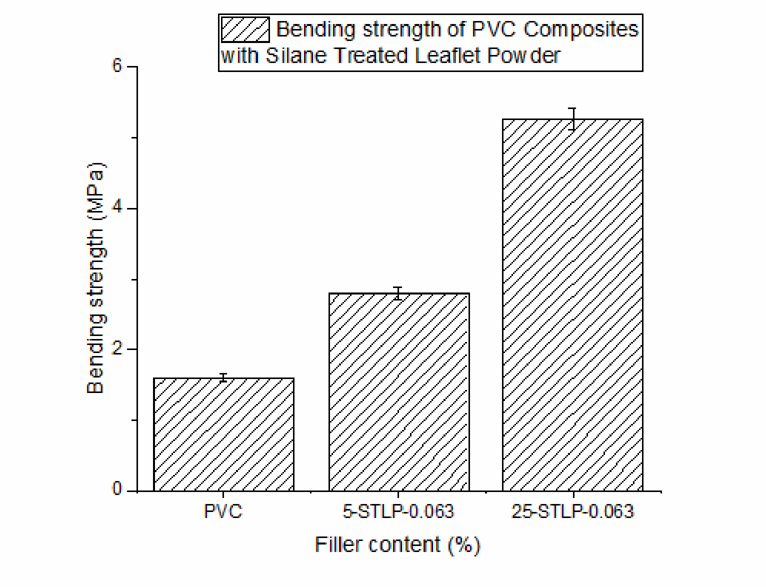

Figure 146 Bending stress results of PVC composites with STLP in function of filler content and filler particle size

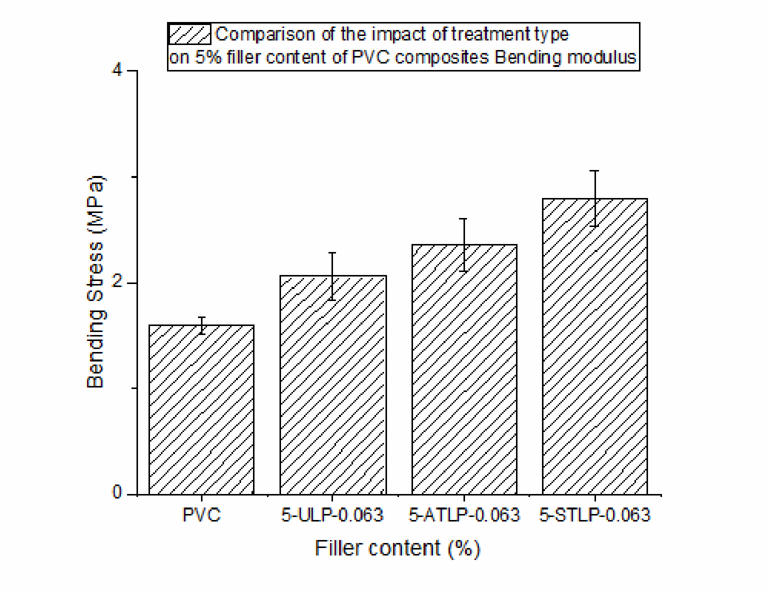

Figure 147 Comparison of the impact of treatment type on 5% filler content of PVC composites Bending stress

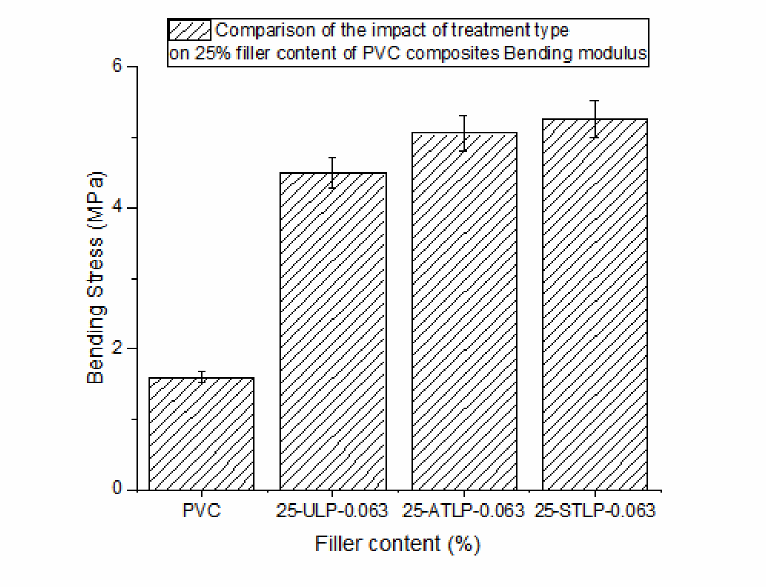

Figure 148 Comparison of the impact of treatment type on 25% filler content of PVC composites Bending stress

III.4. Conclusion:

The FTIR results highlight the effectiveness of both treatments in modifying the chemical composition of DPLP. The NaOH treatment provides a more aggressive approach, resulting in cellulose-rich fibre with enhanced surface properties, while the acetic acid treatment offers a gentler alternative, preserving more of the fibres natural structure while still improving its suitability for composite applications. These findings underscore the importance of selecting the appropriate treatment based on the desired balance between refinement and preservation of natural fibre properties.

The bulk density of the composites is influenced by the type of treatment, particle size, and filler content. ULP composites exhibit lower bulk density due to poor filler-matrix interaction and increased porosity. Alkaline-treated composites show improved bulk density at lower filler percentages, reflecting better filler-matrix interaction. ACLP composites exhibit moderate improvements, with higher variability in bulk density and better results at higher filler percentages. The particle size also plays a significant role, with finer particles often leading to lower density in ULP composites but better integration in treated composites. These findings highlight the importance of selecting the appropriate treatment and particle size to achieve the desired bulk density and mechanical properties in PVC-based composites.

The shore hardness of the composites is influenced by the type of treatment, particle size, and filler percentage. ULP composites show moderate improvements in shore hardness, with significant increases at higher filler percentages due to the natural rigidity of the fibres. ATLP composites exhibit significant improvements in shore hardness, particularly at higher filler percentages, due to the removal of non-cellulosic materials and improved compatibility with the PVC matrix. ACLP composites.

The Young's modulus of the composites is influenced by the type of treatment, particle size, and filler percentage. Untreated composites show moderate improvements in Young's modulus, with significant increases at higher filler percentages due to the natural rigidity of the fibres. Alkaline-treated composites exhibit significant improvements in Young's modulus, particularly at higher filler percentages, due to the removal of non-cellulosic materials and improved compatibility with the PVC matrix. Acetic acid-treated composites show the highest Young's modulus, particularly at higher filler percentages, due to the partial removal of non-cellulosic materials and improved filler-matrix interaction. These findings highlight the importance of selecting the appropriate treatment and particle size to achieve the desired Young's modulus in PVC-based composites. The compatibility between the DPLP and the PVC matrix plays a crucial role in determining the Young's modulus, with alkaline and acetic acid treatments providing significant improvements by enhancing filler-matrix interaction and reducing weak interfacial bonding.

The Maximum strength of the composites is influenced by the type of treatment, particle size, and filler percentage. Untreated composites show the highest Maximum strength, with significant decreases at higher filler percentages due to poor filler-matrix interaction. Alkaline-treated composites exhibit a decrease in Maximum strength, particularly at higher filler percentages, due to the removal of non-cellulosic materials and improved compatibility with the PVC matrix. Acetic acid-treated composites show the lowest Maximum strength, particularly at higher filler percentages, due to the partial removal of non-cellulosic materials and improved filler-matrix interaction. These findings highlight the importance of selecting the appropriate treatment and particle size to achieve the desired Maximum strength in PVC-based composites. The compatibility between the DPLP and the PVC matrix plays a crucial role in determining the Maximum strength, with untreated fibres providing the highest Maximum strength due to their natural rigidity, while treated fibres show reduced Maximum strength due to chemical modifications. The FTIR and SEM analyses further support these findings, showing the removal of non-cellulosic materials and improved surface morphology in treated fibres, which contribute to the reduced mechanical properties of the composites.

The ultimate stress of the composites is influenced by the type of treatment, particle size, and filler percentage. Untreated composites show the highest ultimate stress, with significant decreases at higher filler percentages due to poor filler-matrix interaction. Alkaline-treated composites exhibit a decrease in ultimate stress, particularly at higher filler percentages, due to the removal of non-cellulosic materials and improved compatibility with the PVC matrix. Acetic acid-treated composites show the lowest ultimate stress, particularly at higher filler percentages, due to the partial removal of non-cellulosic materials and improved filler-matrix interaction. These findings highlight the importance of selecting the appropriate treatment and particle size to achieve the desired ultimate stress in PVC-based composites. The compatibility between the DPLP and the PVC matrix plays a crucial role in determining the ultimate stress, with untreated fibres providing the highest ultimate stress due to their natural rigidity, while treated fibres show reduced ultimate stress due to chemical modifications. The FTIR and SEM analyses further support these findings, showing the removal of non-cellulosic materials and improved surface morphology in treated fibres, which contribute to the reduced mechanical properties of the composites.

The maximum strain of the composites is influenced by the type of treatment, particle size, and filler percentage. Untreated composites show the highest maximum strain, with significant decreases at higher filler percentages due to poor filler-matrix interaction. Alkaline-treated composites exhibit a decrease in maximum strain, particularly at higher filler percentages, due to the removal of non-cellulosic materials and improved compatibility with the PVC matrix. Acetic acid-treated composites show the lowest maximum strain, particularly at higher filler percentages, due to the partial removal of non-cellulosic materials and improved filler-matrix interaction. These findings highlight the importance of selecting the appropriate treatment and particle size to achieve the desired maximum strain in PVC-based composites. The compatibility between the DPLP and the PVC matrix plays a crucial role in determining the maximum strain, with untreated fibres providing the highest maximum strain due to their natural flexibility, while treated fibres show reduced maximum strain due to chemical modifications. The FTIR and SEM analyses further support these findings, showing the removal of non-cellulosic materials and improved surface morphology in treated fibres, which contribute to the reduced ductility of the composites.

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The bending modulus of the composites is influenced by the type of treatment, particle size, and filler percentage. Untreated composites show moderate improvements in bending modulus, with significant increases at higher filler percentages due to the natural rigidity of the fibers. Alkaline-treated composites exhibit significant improvements in bending modulus, particularly at higher filler percentages, due to the removal of non-cellulosic materials and improved compatibility with the PVC matrix. Acetic acid-treated composites show the highest bending modulus, particularly at higher filler percentages, due to the partial removal of non-cellulosic materials and improved filler-matrix interaction. These findings highlight the importance of selecting the appropriate treatment and particle size to achieve the desired bending modulus in PVC-based composites. The compatibility between the DPLP and the PVC matrix plays a crucial role in determining the bending modulus, with alkaline and acetic acid treatments providing significant improvements by enhancing filler-matrix interaction and reducing weak interfacial bonding. The FTIR and SEM analyses further support these findings, showing the removal of non-cellulosic materials and improved surface morphology in treated fibers, which contribute to the enhanced mechanical properties of the composites.

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The application of a silane coupling agent (SCA) as a surface treatment for date palm leaflet powder significantly enhanced the overall performance of PVC-based composites. FTIR analysis confirmed the successful grafting of silane groups onto the fibre surface, as evidenced by the appearance of Si–O–Si and Si–O–cellulose peaks, indicating covalent bonding and improved chemical interaction between the filler and the matrix.

Mechanical tests revealed a consistent trend across all parameters (Young’s modulus, tensile strength, elongation at break, bending modulus, and bending strength), where composites reinforced with silane-treated fibres (STLP) outperformed those made with alkaline-treated (ATLP) and untreated (ULP) fibres. Notably:

- Young’s modulus increased significantly, reaching 221.08 MPa at 25% STLP—demonstrating improved rigidity due to strong interfacial bonding.
- Tensile strength showed a notable recovery compared to untreated fibres, with STLP composites reaching 10.6 MPa at 5% filler content, reflecting better stress transfer across the interface.
- Elongation at break was preserved better in STLP composites, with 105.5% at 5% filler content, suggesting retained ductility thanks to better dispersion and reduced fibre agglomeration.
- Bending properties (modulus and strength) were also significantly enhanced, confirming that silane treatment effectively promotes mechanical reinforcement through improved adhesion.

The combination of chemical surface activation and tailored interfacial chemistry made possible by silane treatment proves highly beneficial in developing reinforced PVC composites with superior mechanical behaviour.

General Conclusion

This work examined the applicability of date palm leaf powder (DPLP) as a bio filler to enhance polyvinyl chloride (PVC) composites, aiming at the development of new eco-friendly materials. This research analyzed the effect of different chemical surface treatments on the composite's properties, including (untreated) ULP, alkaline (ATLP), acetic acid (ACLP), and silane coupling agent (STLP).

FTIR analysis confirmed the effectiveness of each treatment. Alkaline treatment resulted in the partial delignification and removal of hemicelluloses as shown by the peak C=O and C-O-C disappearance. This resulted in increased fibre roughness and more exposed cellulose which can enhance the fibre's tensile strength. Acetic acid treatment effectively cleaned the surface of the fibre by removing sodium hydroxide. The silane treatment was the most chemically effective because it added Si–O–Si bonds and confirmed covalent bonds claim with Si–O-cellulose bonds which strengthened the interface with the silane and improved compatibility with the PVC matrix.

The bulk density results demonstrated that the chemical treatments improved the bulk density of DPLP, thus enhancing the performance of the filler in the composite. ACLP showed the highest bulk density followed by STLP and ATLP, while ULP had the lowest.

Mechanical performance varied significantly with filler type, size, and treatment:Young's modulus increased with filler content for all composites. The highest values were obtained for STLP, reaching up to 221.08 MPa at 25% filler content. This reflects the superior stiffness provided by the treated fibres, particularly due to enhanced filler-matrix bonding.

Tensile strength decreased slightly compared to neat PVC (13.4 MPa) but was better preserved in treated composites. STLP composites reached 10.6 MPa at 5% filler content, outperforming ATLP (10.1 MPa), ACLP (9.84 MPa), and ULP (9.66 MPa).

Elongation at break significantly dropped in untreated composites, indicating brittleness due to poor interfacial adhesion. The silane-treated composites showed the best compromise, maintaining elongation values of 105.5% at 5% filler content and 70.46% at 25%, thanks to better dispersion and interfacial bonding.

Bending properties followed similar trends. Both bending modulus and bending strength increased with filler content and were highest in STLP composites due to stronger chemical interactions and improved stress transfer.

Across all treatments, silane-modified fibres (STLP) consistently delivered the most favourable performance, followed by acetic acid-treated Leaflet Powder(ACLP) and alkaline-treated Leaflet Powder (ATLP). Untreated Leaflet Powder (ULP), while preserving ductility to some extent, showed poor compatibility with the hydrophobic PVC matrix and led to reduced mechanical strength.

Overall, the study demonstrates that:

- Surface treatment of date palm leaflets significantly enhances their performance as reinforcing fillers in thermoplastic matrices.

- Silane treatment is the most effective, enabling strong fibre/matrix interfacial bonding through chemical grafting, resulting in composites with superior rigidity, strength, and toughness.
- The combination of alkaline-acetic acid treatment also proved efficient in removing impurities and increasing fibre purity.
- The use of waste agricultural biomass, especially when chemically treated, offers a viable, eco-friendly alternative to conventional fillers, contributing to circular economy strategies and green material development.

Future Perspectives

Building upon the findings of this research, several promising avenues can be pursued to deepen and broaden the scope of sustainable bio-composite development using date palm leaflet powder (DPLP) as reinforcement in thermoplastic matrices:

Development of Hybrid Bio-fillers

Future studies could explore the combination of DPLP with other natural or mineral fillers (e.g., flax, hemp, kenaf, basalt, or clay nanoparticles) to develop hybrid composites with tailored mechanical and functional properties. Such synergy could offer enhanced stiffness, reduced water absorption, improved thermal stability, and lower production costs. Investigations should focus on the optimal blending ratios and processing parameters to achieve the best performance-to-weight and cost-efficiency ratio.

Advanced Surface Modification Techniques and Coupling Agents

While silane treatment showed excellent results, alternative or complementary coupling agents such as **maleic anhydride, titanates, or enzymatic surface modifiers** could be investigated to further improve fibre–matrix adhesion. Moreover, the development of bio-based or green coupling agents in line with environmental sustainability principles would be a key area of innovation.

Thermal and Environmental Durability

A crucial aspect for real-world application is the long-term performance of the composites under various environmental conditions. Future research should include:

- Thermal aging tests to evaluate heat resistance and thermal degradation.
- UV exposure and weathering simulations to assess durability in outdoor applications.
- Moisture uptake and biodegradation studies to understand the materials’ behavior in humid or wet environments. These studies will enable a comprehensive understanding of composite life span and service performance.

Processing Techniques and Optimization

Investigating the influence of different processing methods (e.g., extrusion, injection molding, compression molding) on the dispersion of treated fillers and the resulting composite properties could lead to process optimization and industrial scalability. Rheological behavior, melt flow properties, and fiber breakage during processing should also be assessed.

Scale-up and Industrial Integration

To move from laboratory to market, efforts must focus on scaling up production while maintaining reproducibility and quality. This includes:

- Economic feasibility studies.
- Life cycle assessments (LCA) and environmental impact analysis.
- Integration into existing production lines in industries such as construction (panels, partitions), packaging (biodegradable trays, containers), and automotive (interior parts, trims).

Functionally Graded and Smart Composites

A frontier research direction would be the development of functionally graded bio-composites, where the material properties vary across the volume to meet specific functional demands (e.g., rigid core with flexible outer layers). Additionally, incorporating smart functionalities such as self-healing, sensing, or flame retardancy through additive strategies could open new application domains.

Standardization and Certification

For successful commercialization, it is vital to establish technical standards and certification protocols specific to natural fibre composites. Future work could collaborate with industrial partners and standardization bodies to ensure regulatory compliance and market acceptance.