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Durabilité des mortiers en terre stabilisée

Présentée par :

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الجمهورية الجزائرية الديمقراطية الشعبية

People's Democratic Republic of Algeria

وزارة التعليم العالي والبحث العلمي

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Durability of stabilized earth mortars

Presented by:

Kamal Saleh Mohsen AL-MEASAR

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بسم الله الرحمن الرحيم

DEDICATION

To the one whom Allah has graced with dignity and reverence, to the one who taught me the art of giving without expectation and whose name I bear with immense pride and honor I pray to Allah the Almighty to extend your life so you may witness the fruits of this labor after long anticipation, and your words will forever remain guiding stars for me today, tomorrow, and for eternity,

My dear Father.

To the angel of my life, to the embodiment of love, tenderness, and devotion, to the smile of existence and the secret of being, to the one whose prayers were the key to my success and whose affection healed my wounds, to my most cherished beloved,

My beloved Mother.

To my pillar, my strength, and my refuge after Allah, to those who prioritized my well-being over their own, to those who taught me the lessons of life, to those who revealed to me the beauty beyond life itself,

My brothers

To those whose love courses through my veins and whose memory resonates in my heart,

My sisters

To the one with whom I grew and on whom I rely, to the radiant light illuminating the darkness of my life, to the one whose presence grants me boundless strength and love, to the one with whom I discovered the true meaning of life,

My beloved Wife.

To those in whose eyes I see hope and in whose laughter I find joy, to their faces brimming with innocence,

My beloved sons: Malek and Abdullah.

To the souls whose departure has pained me, leaving an eternal memory in my heart,

My brother Abdulrahman and my son Saleh.

To all the AL-MEASAR family, all my dear relatives, and my friends, to those who brought joy to my studies and shared my burdens and joys.

To my wounded homeland, which, God willing, will return to happiness,

Yemen.

To my homeland, the land of martyrs,

Algeria.

This work is offered sincerely to Allah and then to you all, and I ask Allah to reward you abundantly and bless our collective efforts in service of knowledge and our nation.

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Kamal Saleh Mohsen Al-Measar

ABSTRACT

Throughout ancient times, earth mortar was a fundamental construction material across various civilizations due to its widespread availability, cost-effectiveness, and advantageous thermal comfort characteristics. Today, amidst increasing global concerns such as escalating energy demands and heightened carbon dioxide emissions, the study of earth-based construction methods is gaining renewed relevance. This study aims to optimize the formulation of earth mortar, a fundamental material in traditional earthen construction, through a systematic approach. Particular emphasis is placed on evaluating its mechanical, physical, and durability properties to enhance its performance in contemporary sustainable building practices. Central to this methodology is the exploration of date palm ash (DPA) as a potential additive to improve the performance of earth mortar. To scrutinize the effects of DPA on earth mortar, the study involved preparing samples incorporating lime stabilization alongside varying doses of DPA, ranging from 0% to 10% of the binder.

Experimental results demonstrate that incorporating DPA significantly enhances earth mortar properties. Optimal formulations containing 6% DPA exhibited a 40% reduction in linear shrinkage compared to the reference mixture and an 83.4% increase in elastic modulus. In chemical durability tests, this 6% DPA formulation notably improved resistance to aggressive agents, performing well against sodium sulfate and hydrochloric acid, though sulfuric acid presented the most significant challenge. Under accelerated carbonation, the 6% DPA formulation exhibited increased compressive strength and a stable mass gain. Moreover, DPA improved physical and durability properties. SEM and EDX analyses revealed that DPA reduces voids between earth mortar particles and increases C-S-H content, which supports the observed improvements in overall performance.

The enhanced properties of earth mortar with DPA supplementation offer notable advantages for sustainable construction. DPA mitigates shrinkage and boosts mechanical strength. Additionally, this approach supports environmental sustainability by effectively repurposing date palm cultivation byproducts, reducing waste, and promoting a circular economy.

Keywords: Date palm ash, earth mortar, lime, durability and mechanical behavior

ملخص

على مر العصور القديمة، كان الملاط الترابي مادة بناء أساسية في مختلف الحضارات نظرًا لتوافره على نطاق واسع وفعاليته من حيث التكلفة وخصائص الراحة الحرارية المفيدة. واليوم، في ظل تزايد المخاوف العالمية مثل تصاعد الطلب على الطاقة وارتفاع انبعاثات ثاني أكسيد الكربون، تكتسب دراسة طرق البناء القائمة على التراب أهمية متجددة. تهدف هذه الدراسة إلى تحسين تركيبة الملاط الترابي، وهو مادة أساسية في البناء الترابي التقليدي، من خلال نهج منهجي. ويتم التركيز بشكل خاص على تقييم الخصائص الميكانيكية والفيزيائية والديمومة لتعزيز أدائها في ممارسات البناء المستدام المعاصرة. ومن الأمور المحورية في هذه المنهجية استكشاف رماد نخيل التمر (DPA) كمادة مضافة محتملة لتحسين أداء الملاط الترابي. للتدقيق في تأثيرات DPA على الملاط الترابي، تضمنت الدراسة إعداد عينات تتضمن تثبيت الجير إلى جانب جرعات متفاوتة منDPA ، تتراوح من 0% إلى 10% من المادة الرابطة.

أظهرت النتائج التجريبية أن دمج DPA يعزز بشكل كبير من خصائص الملاط الترابي. أظهرت التركيبات المثلى التي تحتوي على 6٪ DPA انخفاضًا بنسبة 40٪ في الانكماش الخطي مقارنةً بالخليط المرجعي وزيادة بنسبة 83.4٪ في معامل المرونة. في اختبارات المقاومة الكيميائية، حسّنت تركيبة DPA بنسبة 6% من مقاومة العوامل العدوانية بشكل ملحوظ، حيث كان أداؤها جيدًا ضد كبريتات الصوديوم وحمض الهيدروكلوريك، على الرغم من أن حمض الكبريتيك كان التحدي الأكبر. في ظل الكربنة المتسارعة، أظهرت تركيبة DPA بنسبة 6% قوة انضغاطية متزايدة وزيادة كتلة ثابتة. وعلاوة على ذلك، حسنت DPA الخصائص الفيزيائية وخصائص المتانة (الديمومة) . كشفت تحليلات SEM وEDX أن DPA يقلل من الفراغات بين جزيئات الملاط الترابي ويزيد من محتوى C-S-H، مما يدعم التحسينات الملحوظة في الأداء العام.

توفر الخصائص المميزة للملاط الترابي المحسنة مع إضافات DPA مزايا ملحوظة للبناء المستدام. يخفف DPA من الانكماش ويحسِّن القوة الميكانيكية. وبالإضافة إلى ذلك، يساعد هذا النهج على دعم الاستدامة البيئية من خلال إعادة استخدام المنتجات الثانوية لزراعة نخيل التمر بشكل فعال، والحد من النفايات، وتعزيز الاقتصاد الدائري.

الكلمات المفتاحية: رماد النخيل، الملاط الترابي، الجير، الديمومة، السلوك الميكانيكي.

Résumé

Dans l'Antiquité, le mortier de terre était un matériau de construction fondamental dans diverses civilisations en raison de sa grande disponibilité, de sa rentabilité et de ses caractéristiques avantageuses en matière de confort thermique. Aujourd'hui, face aux préoccupations mondiales croissantes telles que l'escalade de la demande énergétique et l'augmentation des émissions de dioxyde de carbone, l'étude des méthodes de construction en terre gagne en pertinence. Cette étude vise à optimiser la formulation du mortier de terre, un matériau fondamental dans la construction traditionnelle en terre, par le biais d'une approche systématique. L'accent est mis sur l'évaluation de ses propriétés mécaniques, physiques et de durabilité afin d'améliorer ses performances dans les pratiques contemporaines de construction durable. L'exploration de la cendre de palmier dattier (DPA) en tant qu'additif potentiel pour améliorer les performances du mortier de terre est au cœur de cette méthodologie. Pour examiner les effets de la DPA sur le mortier de terre, l'étude a consisté à préparer des échantillons incorporant une stabilisation à la chaux avec des doses variables de DPA, allant de 0 % à 10 % du liant.

Les résultats expérimentaux démontrent que l'incorporation de DPA améliore de manière significative les propriétés des mortiers de terre. Les formulations optimales contenant 6 % de DPA présentent une réduction de 40 % du retrait linéaire par rapport au mélange de référence et une augmentation de 83,4 % du module d'élasticité. Dans les essais de durabilité chimique, cette formulation à 6 % de DPA a notablement amélioré la résistance aux agents agressifs, se comportant bien contre le sulfate de sodium et l'acide chlorhydrique, bien que l'acide sulfurique ait présenté le défi le plus important. Lors d'une carbonatation accélérée, la formulation à 6 % de DPA a présenté une résistance accrue à la compression et un gain de masse stable. En outre, le DPA a amélioré les propriétés physiques et de durabilité. Les analyses SEM et EDX ont révélé que le DPA réduit les vides entre les particules de mortier de terre et augmente la teneur en C-S-H, ce qui confirme les améliorations observées au niveau des performances globales.

Les propriétés améliorées du mortier de terre avec l'ajout de DPA offrent des avantages notables pour la construction durable. Le DPA atténue le retrait et renforce la résistance mécanique. En outre, cette approche soutient la durabilité environnementale en réutilisant efficacement les sous-produits de la culture du palmier-dattier, en réduisant les déchets et en promouvant une économie circulaire.

Mots clés : Cendre de palmier dattier, Mortier de terre, Chaux, Durabilité, Comportement mécanique.

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GENERAL INTRODUCTION

GENERAL INTRODUCTION

The quest for sustainable building materials has led to a resurgence of interest in earth construction, particularly in the use of stabilized earth mortars. These materials offer a promising alternative to conventional construction materials, which are often resource-intensive and environmentally damaging. This dissertation delves into the durability of stabilized earth mortars, assessing their potential to withstand various environmental and mechanical stresses. By exploring the factors that influence their performance, this research aims to provide a comprehensive understanding of how to enhance the longevity and resilience of earth-based construction materials.

1. Research background

Earth construction has a long and storied history, with evidence of its use dating back thousands of years. Ancient civilizations, such as those in Mesopotamia, Shibam Hadramout,Egypt, and China, utilized earth materials to construct homes, fortifications, and monumental structures. The enduring nature of these ancient buildings demonstrates the potential longevity of earth materials. In modern times, the principles of earth construction have been refined and adapted to meet contemporary building standards and environmental concerns.

Since the inception of human activity in construction, raw earth materials have remained fundamental components used worldwide [1]. The use of earth-based materials in construction is time and energy efficient, reducing environmental pollution and transportation and manufacturing costs [2]. Earthen buildings are constructed from bricks (adobe, compressed earth blocks, etc.) and earth mortar made from clay soil, sand, water, and other stabilizers like lime, cement, gypsum, and vegetable fibers [3-5].

One of the main challenges with traditional earth mortars is their susceptibility to environmental degradation, such as erosion, water damage, and freeze-thaw cycles. To address these issues, various stabilization techniques have been developed. Stabilizers such as lime, cement, and natural fibers can significantly enhance the mechanical properties and durability of earth mortars. However, the effectiveness of these stabilizers can vary widely depending on factors such as soil composition, stabilizer type and concentration, and environmental conditions.

Studies have shown that it is preferable to use earth mortar with adobe or compressed earth blocks instead of traditional cement mortar. Using earth construction materials with a significant amount of cement can lead to structural problems if the mortar's resistance is greater than the resistance of the clay blocks [6-8]. Moreover, earth mortars are often stabilized with small amounts of lime or cement by incorporating them into the mix as stabilizers [9].

Historical Perspective

Earth construction has been a cornerstone of human habitation for millennia. Ancient structures, such as the ziggurats of Mesopotamia, the Great Wall of China, and the adobe villages of Africa and the Americas, showcase the durability and versatility of earth as a building material. These structures were built using techniques like rammed earth, cob, adobe, and wattle and daub, each adapted to local conditions and resources. The historical perspective highlights the ingenuity and adaptability of ancient builders, who used readily available materials to create durable and functional structures.

Earth mortars are used either to bind bricks together or as wall coverings. In binding, the mortar is fundamental to the stability of earthen buildings. As wall coverings, it protects against moisture, rare water seepage, and erosion from wind and fluctuations in groundwater levels [10]. It is also used as plaster, recognized as one of the most environmentally efficient materials due to its technical and environmental benefits. Among these technical benefits, earth mortar contributes to thermal comfort through internal thermal equilibrium, a crucial feature for occupant health [11-14].

Contemporary Earth Construction

Modern earth construction has seen a resurgence, driven by a growing awareness of environmental sustainability and the need for cost-effective building solutions. Contemporary techniques often integrate traditional knowledge with modern innovations. Rammed earth, for example, involves compacting soil in layers to form solid walls, while adobe bricks are made from a mixture of soil, water, and organic materials, then dried in the sun. Earthbag construction, where soil-filled bags are stacked and tamped down, is another modern adaptation.

In contemporary times, many in the construction field turn to using lime as a soil stabilizer instead of cement for several reasons. Lime production results in lower CO_2 emissions and energy consumption compared to cement. Additionally, significant amounts of CO_2 are reabsorbed from the atmosphere during carbonation, and earth mortar is soft and flexible, allowing thermal exchange between the internal and external environments of the walls [15].

While there have been significant advancements in the stabilization of earth mortars, a comprehensive understanding of their long-term durability remains elusive. Existing research often focuses on short-term performance or specific aspects of durability, leaving a gap in the holistic understanding of how stabilized earth mortars behave over extended periods. This dissertation aims to address this gap by conducting a thorough investigation into the durability of various stabilized earth mortars. It seeks to identify the key factors that influence their performance and to provide insights into optimizing stabilization techniques for enhanced durability.

2. Problem Statement

Despite the multiple advantages of earth mortar, it has some drawbacks, such as low mechanical resistance and durability in humid environments. Researchers, residents, and authorities strive to improve its properties through surface treatments or using additives, considering economic, environmental, social, and cultural factors [16-18]. This research focuses on using ash as a mineral additive in the preparation of earth mortar, particularly palm ash.

Numerous studies indicate that additives used in mixtures, such as fly ash, slag, silica fume, natural pozzolans, and fibers, are crucial factors affecting the properties and durability of unfired earthen materials [19-24]. Fly ash has been extensively studied to demonstrate its potential as a stabilizer for raw earth bricks and rammed earth construction. According to a study by Sadique and Berto [25] , SEM images showed the formation of new products around fly ash particles, reducing soil porosity and increasing bonding between soil particles and stabilizers. Additionally, experiments by Zhu and Chen [26] on the use of fly ash with cement and phosphogypsum waste to stabilize loess soil for self-compacting rammed earth construction demonstrated that fly ash helps form C-A-H and C-S-H gels in an alkaline environment, filling material voids and justifying the mechanical and water resistance results.

While there have been significant advancements in the stabilization of earth mortars, a comprehensive understanding of their long-term durability remains elusive. Existing research often focuses on short-term performance or specific aspects of durability, leaving a gap in the holistic understanding of how stabilized earth mortars behave over extended periods.

3. Aims of the thesis

Other types of ash, such as rice husk ash and sugarcane ash, have been used in research to stabilize raw earth materials, yielding satisfactory results. This was demonstrated by a study conducted by Behak et al. [27] on the physical and mechanical properties of a mixture of sand, soil, lime, and rice husk ash. They found that the compressive strength of the mixture containing rice husk ash was five times higher than the basic mixture. They also recorded improvements in the mixture's behavior in wet/dry tests. Similar results were obtained in other studies, such as Alves-Ramirez et al.'s [20] research on stabilizing compressed earth blocks using lime and sugarcane ash, and Patagarawa et al.'s [28] study on the thermal properties of rammed earth using corn husk ash as a stabilizer.

The primary objective of this dissertation is to evaluate the durability of stabilized earth mortars under various environmental and mechanical conditions. The specific objectives are as follows:

1 Literature Review: To conduct a comprehensive review of existing literature on earth construction and stabilization techniques, identifying gaps and areas for further research.

2 Material Characterization: To identify and characterize the materials used in stabilized earth mortars, including soil types, stabilizers, and additives.

3 Experimental Design: To develop and apply robust experimental methods for testing the durability of stabilized earth mortars, encompassing a range of environmental and mechanical tests.

4 Data Analysis:To analyze the results of the experiments, comparing the performance of different stabilization methods and identifying the most effective approaches.

5 Practical Recommendations: To provide practical recommendations for the use of stabilized earth mortars in construction, based on the findings of the research.

4. Benefits and Challenges

Ash derived from burning palm tree waste has also been used in construction research. Studies by Al-Kuti et al. [29, 30] on the performance of cementitious materials (paste, mortar, and concrete) have shown improvements in the overall quality of these materials. Shibam Hadramout city and other cities in Yemen were built using materials containing ash, dating back thousands of years. Builders obtained ash from burning wood and waste residues. Significant improvements were recorded in physical and mechanical properties and durability, especially in wall covering and restoration [31-36]. However, no research papers have documented the use of palm ash in earth construction.

Earth construction offers numerous benefits, including low environmental impact, energy efficiency, and affordability. The use of local materials reduces transportation costs and emissions, while the thermal mass of earth walls provides natural insulation, reducing energy consumption for heating and cooling. Additionally, earth construction is often more affordable than conventional methods, making it accessible to low-income communities.

However, there are challenges associated with earth construction. The variability of soil properties can lead to inconsistent performance, while susceptibility to moisture and weathering can compromise the durability of earth structures. Stabilization techniques can mitigate some of these issues, but they also introduce complexities, such as the need for careful selection and testing of materials.

5. Significance of the Study

Algeria is one of the largest date-producing countries in the world, with over 18 million palm trees. Annual farm cleaning operations produce about 200,000 tons of waste, some of which are used for fibers and craft products, while the rest is discarded or burned [37, 38]. To utilize the large amounts of palm waste, date palm ash was used in this study as a partial replacement for lime in the preparation of earth mortar. This research aims to investigate the potential of this eco-friendly material in the preparation of earth mortar, focusing on its mechanical performance and durability.

The significance of this study lies in its potential to revolutionize sustainable construction practices. By demonstrating the effectiveness of stabilized earth mortars, this research could pave the way for more widespread adoption of earth-based construction materials. This would contribute to reducing the environmental footprint of the construction industry, promoting the use of local and renewable resources, and enhancing the resilience of buildings in various climates

This study has significant implications for the field of sustainable construction. By improving the understanding of the durability of stabilized earth mortars, this research can contribute to the development of more resilient and environmentally friendly building practices. The findings can guide architects, engineers, and policymakers in adopting earth-based materials, thus promoting sustainability and reducing the environmental footprint of the construction industry. Furthermore, this research can help in the preservation and enhancement of traditional building techniques, bridging the gap between ancient wisdom and modern science.

6. **Structure of the thesis**

This thesis comprises five chapters focused on enhancing the properties of earth mortar using date palm ash. Here's a summary of its contents:

- ✓ GENERAL INTRODUCTION: This introduces the research background, highlighting the significance of the study and its aims. It outlines the thesis structure and discusses the benefits and challenges associated with the topic.
- ✓ CHAPTER 1: Beginning with an introduction, this chapter delves into the historical evolution of earth construction globally. It details various earth construction techniques such as adobe, rammed earth, cob, and others, while also exploring soil types, their classifications, and their chemical and physical properties. The chapter concludes with a focus on durability, including mechanisms of deterioration like sulfuric attack and carbonation.
- ✓ CHAPTER 2: This chapter starts with an introduction to the materials used in the study, including soil, water, lime, and DPA. It then outlines the experimental methods employed, covering mix proportions, physical tests (porosity, shrinkage, density), mechanical tests (compressive strength, flexural strength), durability tests (absorption, swelling, drying-wetting), Chemical durability tests and Accelerated Carbonation Tests.
- ✓ CHAPTER 3: Focused on presenting findings, this chapter discusses the effects of DPA on physical (porosity, shrinkage, density) and mechanical properties (compressive strength, flexural strength) of materials. It also analyzes DPA's impact on durability properties such as absorption, swelling, and resistance to environmental conditions, culminating in conclusive remarks on the experimental outcomes.
- ✓ GENERAL CONCLUSION AND PERSPECTIVES: This offers a summary of the thesis findings, reiterating key conclusions drawn from the research. It also provides insights into potential future research directions and applications based on the study's outcomes.

CHAPTER 1

AN OVERVIEW ON EARTH CONSTRUCTION

1. CHAPTER 1: AN OVERVIEW ON EARTH CONSTRUCTION

1.1. Introduction

Many of the advantages of earth building are underused in the industrialized world, where earth is frequently used as a low-embodied material [39]. Earth has long been the most well-known and frequently utilized building material in history, and it is perhaps the most essential of all building materials [40]. According to recent statistics, almost half of the world's population still lives in earthen structures [41]. Around 25% of all urban dwelling units globally do not comply with construction rules, while 18% are deemed non-permanent buildings [41]. Earth as a construction material is widely accessible and comes in a variety of compositions. In underdeveloped nations, it is most effectively employed to shelter the maximum number of people with the least amount of demand. It is important to highlight that earth structures are not simply a phenomena in Third World nations, but also in wealthy countries [39]. Earthen structures have several advantages. Earth buildings, for example, are fully recyclable, so sun-dried bricks may be returned to the earth without harming the environment [42]. Using earth for such environmentally friendly structures will be a significant part of humanity's future [43, 44]. In fact, most emerging countries are suffering from a severe housing shortage [45]. As a result, there is a pressing need to develop and build more lasting dwellings at a lower cost. Clay masonry has a long and distinguished history of producing long-lasting and aesthetic structures in this area. Traditional earth construction technology has recently experienced significant advancements that have improved earth's resilience and quality as a construction material for low-cost structures [46].

1.2. History of earth materials and traditional clay buildings

1.2.1. Chronologies of earthen construction

Subsoil building is one of the oldest methods of construction, offering basic shelter with readily available materials. Soil-based structures may be found in many regions of the world, in various shapes, and are occasionally combined with other traditional building materials like wood or stone, or with more contemporary technologies like cement and steel. Earth-building techniques are likely to have originated independently in different regions of the world and expanded with the movement of people. Early man was always on the move, following the hunter-gatherer patterns prescribed by the environment. The first shelters may have been expansions of natural features such as mounds of earth at cave openings or pits dug into the ground, and the first earth structures may have been extensions of natural features such as mounds of earth at cave entrances or pits dug into the ground. Agriculture began in rich river valleys, where silt and clays make good building materials for earthen structures. Wattle and daub was most likely the earliest technique to emerge, which involves building a façade or roof out of wood or grasses and then covering it with dirt. Later, a rammed earth method may have evolved, in which earth was poured against or between timber-framed walls and compressed into place, resulting in a thicker wall. A unit building might have evolved later; first, units were created by hand, and subsequently, the formwork was used to create more cuboid blocks. When dry, they may be moved, allowing material production to be isolated from the location of the structure, for example, appropriate soil might be collected from a flood-prone river valley and utilized to construct buildings at a higher height. In the primary cradles of civilisation, agriculture and earth building developed independently. People began to assemble in communities for the first time when agriculture developed beside important rivers. These rich river valley civilisations offered the ideal soil for earthen construction, and evidence of their evolution may be seen in the valleys of the Tigris and Euphrates, as well as the Nile, Indus, Jordan, Murghab, and Yellow Rivers. Despite the fact that these societies stayed separate, they appear to have evolved quite comparable earth-building skills. Techniques were polished and updated as civilisation and commerce progressed. Because earth construction techniques change from settlement to settlement and year to year, this is difficult to trace. However, it appears that the shift from hand-moulded to cuboid bricks took place approximately 5000 BC in Mesopotamia, and that rammed earth was not present in South America prior to European immigration. When other building materials are available, earth is commonly used in conjunction with them. Where stone is scarce, earth is used as a plaster or mortar, like in Malton in the north of England, or grass is used to build roofs and walls, as in the Western Isles of Scotland [47].

1.2.2. Contemporary earthen construction in the world

1.2.2.1. Earth building techniques in Asia

In China, the establishment of advanced civilization began around 2300 BC, coinciding with the time when nomadic groups settled into the fertile alluvial plains of the Yellow River, leading to the rise of the Lungshan civilisation. The region's rich and pliable soils were particularly advantageous, allowing inhabitants to construct pit homes by digging into the soft earth and to mound the soil for creating robust rammed earth walls. This innovative technique facilitated the emergence of fortified towns and settlements, with notable examples found at key sites like Lianyungang, Jiangsu, Taosi, Erlitou, and Longwan. An archaeological discovery at Pingliantai revealed formwork boards and specialized ramming tools, indicating the construction methods of that time. During the Warring States Period (475–221 BC), this method of employing rammed earth evolved further, leading to the establishment of more complex and sophisticated rammed earth walls in major community centers such as Langya, Anyang, Linzi, and Xiadu. Following this period, the Qin dynasty (221–206 BC) marked a significant milestone as it became the first dynasty in western China to erect rammed earth defensive walls along its northern frontiers to protect against invasions. The subsequent Han dynasty (206 BC–202 AD) and Jin dynasty (265 AD–420 AD) continued this practice, restoring and expanding these formidable walls as part of their military strategy and construction endeavors [47].

The Tang dynasty (618–907 AD) marked another significant chapter in the history of Chinese fortifications, as it expanded the boundaries of the empire and pursued the enhancement of commerce. However, it was also a time of turmoil, with frequent incursions from various nomadic tribes to the north. In response to these threats, fortified towns were constructed across northwestern China, particularly along the eastern stretch of the legendary Silk Route. Major cities such as Jiahoe, Gaochang, and Xi'an were encircled by expansive rammed earth walls designed for defense and protection. The city of Kashgar, located in the far western part of China, is notable for its entire structure being built from adobe. Similarly, the military stronghold known as Baishui, located at the western terminus of the Great Wall, was constructed exclusively from rammed earth materials, showcasing the ingenuity of ancient building practices [47].

Around 7000 BC, settled civilization emerged in the Indus Valley, with Mehrgarh, a modest adobe village in what is now Pakistan, serving as a precursor to the more extensive Indus Valley civilization that blossomed around 3000 BC. By approximately 2600 BC, this civilization had matured along the banks of the Indus River, leading to the development of impressive urban centers, most notably Harrapa and Mohendjaro. In both of these significant settlements, adobe homes were meticulously planned around an organized grid layout, with individual streets enhancing the communal living experience. Furthermore, the Bactria-Margiana archaeological complex, comprising nearly 300 unique walled adobe brick enclosures dated between 2200 and 1700 BC at various sites including Namazga-, Altyn-, and Gonur-Depe, is intrinsically linked to the practices of settled agriculture and the genesis of monumental architecture in Central Asia [47].

Though the dominant peoples of Central Asia were chiefly nomadic, traces of some adobe forts or Qalas can be found in western Uzbekistan, with some estimates suggesting their origin around 300 BC. However, due to the changing patterns of trade, the movements of conquering armies, and the alterations of river routes, only a handful of these ancient settlements have managed to withstand the test of time into the modern era. Many of those that persevered, such as Balkh and Merv, evolved over centuries into prominent commercial hubs. Due to its rich history, Balkh (known historically as Bactria) in contemporary Afghanistan is often referred to as Umm Al-Belaad, or "Mother of Cities." Among Balkh's existing structures, the ancient rammed earth edifices of Takht-e Rustam and Top-Rustam, attributed to either Buddhist or Zoroastrian traditions, stand as some of the city's earliest surviving buildings, believed to date back to approximately 2000 BC. Over time, Balkh emerged as the preeminent city in the region, establishing itself as a cornerstone of trade and commerce.

Turkmenistan's Merv city presents a unique phenomenon among ancient sites. Archaeological research indicates that because multiple communities were built adjacent to one another rather than directly atop the previous ones, earlier structures can often be uncovered without the complete demolition of more recent additions. Merv’s buildings, predominantly constructed from earth, saw their earliest settlements emerge as far back as 2000 BC and remained almost continuously inhabited until 1787 AD, when it was ultimately abandoned and fell into ruins before being rediscovered as an important archaeological site. Both Merv and Balkh found themselves strategically situated on principal trade routes traversing Central Asia, leading to the establishment of numerous additional earthen towns along the Silk Route. Panjakent, an adobe city located in western Tajikistan, was first documented around 500 BC and was likely a central hub along the Silk Road until its decline in the 8th century. Since the onset of intensive archaeological excavations in the 1940s, the site has seen a surge in interest and is now a popular tourist destination.

Farther east, the rammed earth defensive walls and structures of Ordu-Baliqin, which was the capital of the Uyghur empire and a prominent Silk Route metropolis, were excavated. Established in 745 AD, this city was eventually abandoned by 840 AD. Throughout history, many clay villages faced devastation at the hands of invading armies, including those of Alexander the Great in 330 BC, the Muslim conquests in 720 AD, and the marauding forces of Genghis Khan in 1220 AD, resulting in a significant number of sites across Central Asia reduced to mere echoes of their former grandeur. From around 9000 BC onward in the Euphrates and Tigris river valleys emerged the early nomadic civilizations that transitioned to permanent agriculture and settlement.

These early civilizations are known for constructing circular edifices using hand-moulded oval bricks, examples of which can be observed at significant sites such as Djade al-Mughara in Syria and Tappeh Ozbaki and Ganj Durrell in Iran. At locations like Jericho and Netiv Hagdud, these oval bricks persisted in use until approximately 6000 BC. In Iraq, the Tell Hassuna site introduced square bricks around the same time, which signified a shift in building style as the structures in Jericho evolved from circular forms to rectangular designs. As the cities along the Euphrates and Tigris rivers flourished, growing in both dimension and intricacy, the metropolis of Uruk, distinguished by its rammed earth establishments and adobe temples, emerged as the largest settlement within the region around 3500 BC. The Assyrian cities of Elba and Mari, situated nearer to the Arabian Gulf, vied for regional influence, and excavations at these locations reveal the presence of earthen city walls and elaborate adobe palaces within their bounds.

The technological advancements achieved by the time of the Ziggurat of Ur's completion around 2100 BC are particularly noteworthy, as it featured a central core built from adobe bricks, fully encased in a fired brick facade set within bitumen. Although many Ziggurats may have been originally constructed from adobe, they have likely succumbed to erosion and decay over centuries, with many remaining anonymous today. On the alluvial plains of the Arşamba river in central Turkey, the village of Çatalhöyük emerged, likely being the largest settlement of its time, supporting a population of around 5000 individuals between 7300 and 6800 BC. This city was architecturally composed entirely of adobe, characterized by irregularly shaped homes packed tightly together, encouraging rooftop access as the primary means of entering dwellings. Throughout history, many villages in this area have experienced restorative efforts, often rebuilding their earthen cores over the years.

The Iranian cities of Yazd and Isfahan showcase a wealth of ancient adobe architecture that has survived throughout history, with some of these buildings standing as testaments to the architectural prowess of their times. Moreover, the city of Tousis boasts rammed earth walls, while the remarkable Bam Citadel, which dates back to around the 7th century AD and was once hailed as the largest adobe structure in the world, was regrettably compromised during an earthquake in 2003. Shibam in Yemen is particularly renowned for its strikingly tall adobe buildings. The local inhabitants began construction around 1700 AD, and there are presently more than 500 adobe 'skyscrapers' that ascend to heights of up to 30 meters. The Tarim region is home to the fascinating Muhdhar Mosque, within which resides a clay minaret, completed in 1914, that proudly holds the title of the world's tallest earthen structure, soaring to an impressive height of 53 meters, symbolizing the enduring legacy of adobe architecture across diverse cultures and epochs [47].

Fig.1. 1. Rammed earth section of Kyichu Lhakhang Monastery, Bhutan [48].

Fig.1. 2. . Rammed earth fort at Basgo, India [49].

Fig.1. 3. Minaret of Al-Muhdhar Mosque, Tarim, Yemen [50].

Fig.1. 4. Mud-brick Seiyun Palace in Yemen [51] .

1.2.2.2. Earth building techniques in Africa

Despite the widely recognized fact that Africa stands as the origin of mankind, the field of archaeology has yet to uncover a rich and detailed history of earth construction across the vast expanse of the continent. The traditional African mud house, typically crafted from woven reeds or wood and then plastered with a mixture of soil, may have remarkably remained relatively unaltered for millennia, reflecting its enduring design. The oldest known earth-covered settlements, which prominently feature woven reeds and branches, such as Mermid and Fayum located in the lush Nile Delta region, have been dated as far back as 5000 BC, showcasing an early ingenuity in construction techniques.

Fast forward to around 2900 BC, significant Egyptian dynasties began to emerge notably in the Nile Valley, and during this period, the artisans of the land crafted hand-shaped adobe bricks utilizing a mixture of clay river silt combined with desert sand and straw sourced from cultivated grains. Adobe, a natural building material, was prominently employed as a construction method for monumental structures long before the erection of the more iconic stone edifices that we often associate with ancient Egypt, a fact illustrated by the impressive independent adobe constructions found at Shumet el-Zebib and nekhen, which have been dated to 2750 BC. Even within the realms of Egypt, adobe continued to be utilized actively as a vernacular building material among the community.

Remarkably, the masons of the Valley of the Kings resided in settlement known as Deir el Medina from around 1550 to 1080 BC, where they created square, single-room adobe homes arranged in a structured grid system that exemplified early urban planning. The notable Pharaoh Akhenaten, during his reign, erected Tel el-Amarna as a new capital city in 1353 BC; however, this ambitious project was soon abandoned shortly after its establishment. In this city, one could observe single-story, rectangular adobe houses featuring exterior steps that led up to flat roofs, embodying the architectural aesthetics of that era. Rameses II, who ruled from 1279 to 1213 BC, undertook a multitude of construction projects, and his royal seal was notably stamped on adobe bricks originating from his major building endeavors. Despite these developments, Egypt remained somewhat isolated from the broader landscape of Africa, which ultimately resulted in a minimal exchange of construction practices with the continent at large.

The Phoenician culture remarkably expanded over the vast regions of North Africa, originating from the eastern Mediterranean. They established numerous thriving colonies along the north coast of Africa, which facilitated trade and cultural exchange. One of their most significant cities, Carthage, located in what is now modern-day Tunisia, was founded in 814 BC. Archaeological excavations have revealed the presence of rammed earth walls, which were commonly utilized in the construction of dwellings within the city. This ancient technique can also be observed in both Marrakesh and Fes, where impressive rammed earth city walls have stood the test of time. This building method is not limited to these cities; it played a pivotal role in monumental Muslim construction across the region. A prime example is the stunning El Badi Palace in Marrakesh, which was completed in the year 1578 and showcases the enduring legacy of rammed earth construction in creating magnificent architectural masterpieces.

Although complex and diverse cultures have existed in West Africa since approximately 1500 BC, the notable Ghanaian empire, which governed a significant region of the continent from roughly 830 AD onwards, is recognized as the first to be documented in historical records. While much of the monumental architecture from that era is composed of durable stone, it is thought that ancient vernacular architecture heavily relied on existing earth building techniques, utilizing materials such as adobe and cob construction methods for their residential and communal structures. The decline and eventual fall of the powerful Ghanaian dynasty in 1235 AD created an opportunity for the rise of Mali's expansive empire, which encompassed the illustrious clay towns of Djenne and Timbuktu, both of which hold immense historical significance. Djenne's first grand mosque, an architectural marvel, was most likely erected around 1200 AD, but unfortunately, it fell into disrepair until it was skillfully rebuilt in 1907, showcasing the resilience and dedication of the local community to preserve their cultural heritage.

Other notable West African buildings with comparable architectural styles include the famous Sankore and Djinguereber Mosques located in Timbuktu, Mali, which were impressively constructed around the year 1320. They stand out not only for their historical significance but also for their unique design elements. Additionally, the Grand Mosque of Agadez in Niger, erected around 1515, showcases similar stylistic features. These remarkable structures are particularly distinctive due to the bundles of palm stalks that gracefully protrude from their walls. These palm stalks are not merely decorative; they serve an essential function as scaffolding during the buildings' annual replastering, a crucial maintenance practice that helps preserve their integrity and appearance.

Furthermore, inverted catenary dome structures built from earth can be found as far east as Cameroon. In this region, the houses of the Musgum people exemplify these inverted catenary dome forms, constructed from local earth materials, showcasing the ingenuity and adaptability of local architecture. On the contrary, monumental earth structures are considerably rare in the wooded and more humid parts of central and southern Africa. While such large-scale earth constructions may be absent in these regions, various forms of earth construction are still actively utilized across the continent. These alternative earth-building techniques reflect the diverse cultural heritage and architectural practices that continue to thrive in Africa today [47].

Fig.1. 5. Hypostyle column in the Temple of Amun [52].

Fig.1. 6. Approach and first pylon - Temple of Horus [53].

Fig.1. 7. El Badi Palace in Marrakesh [54].

1.2.2.3. Earth building techniques in Europe

The earth is utilized skillfully in combination with timber through various construction techniques such as wattle and daub, as well as the half-timbered methods commonly seen in northern Europe. In contrast, in southern Europe, adobe and rammed earth techniques are predominantly employed to create sturdy structures. The small dwellings that were built on solid stone foundations represent the earliest usage of adobe in the European continent, with evidence dating back to approximately 5300 BC at the prehistoric village of Sesklo in Greece. This early use of adobe highlights the ingenuity of ancient peoples in adapting local materials for shelter. However, in northern Europe, the prevalent use of earth along with wood in construction has led to the degradation of many ancient sites over time. As a result, only the foundations of these structures remain today, making it a challenging task to accurately estimate the original building materials and techniques that were employed. Additionally, intriguing remnants of adobe structures have been uncovered further east in the region of Hattua, located in central Turkey, with these findings dating back to around 1600 BC, showcasing the diverse architectural practices of ancient cultures across Europe.

The Phoenicians, who were originally from the eastern Mediterranean region, migrated and established several towns in Spain, including notable locations such as Morro de Mequitta. This significant movement could have led to the introduction of rammed earth construction techniques to Europe, showcasing the exchange of architectural knowledge. Roman historian Pliny the Elder recounts the impressive structures constructed by Hannibal, particularly his famous rammed earth towers that stood as a testament to engineering prowess. Additionally, the prominent architect Vitruvius illustrates how this ancient building method was employed in the development of the French city of Marseilles and also in the historic Greek city of Athens, where structures were described as being fully made of adobe. Despite the prevalence of monumental architecture during the Greek and Roman empires, largely built from durable stone, it is highly probable that traditional building methods such as half-timbered and wattle and daub construction continued to be widely utilized in vernacular architecture across Europe long after the decline of these great empires. This adaptation and persistence of indigenous building techniques reflect the resilience and ingenuity of local communities in response to changing circumstances and architectural trends.

In the year of our Lord 711 AD, the transformative religion of Islam made its grand entrance into southern Europe, bringing along with it a wealth of architectural techniques and knowledge that had been developed in North Africa. This period witnessed the construction of numerous rammed earth and adobe fortresses, strategically built during a time characterized by significant conflict and territorial disputes. Among the notable fortifications that were discovered are those of Calatayud and Plad'Almalain in Spain, which were unearthed in the year 884 AD, illustrating the importance of these structures in that era. The Muslim defense walls of medieval towns, such as the magnificent cities of Cordoba, Seville, and Granada, were primarily constructed using rammed earth, showcasing the durability and strength of this ancient building method. Around the year 1238, the Alhambra Palace in Granada, which is now celebrated as a UNESCO World Heritage Site, was constructed predominantly out of this same rammed earth, serving as a glorious testament to the architectural ingenuity of the time. While earth continued to be a popular construction material well into the 16th century, it gradually began to fade from regular use, especially as the demand for more durable and resilient burned brick began to rise significantly. In northern Europe, the emergence of wattle and daub techniques marked a new chapter in vernacular architecture, demonstrating the adaptability and creativity of builders in that region. Furthermore, cob buildings, which date back to approximately 1400 AD, have been discovered scattered throughout the United Kingdom, and this particular building technology was successfully utilized as a vernacular technique well into the nineteenth century, attesting to its enduring practicality and appeal in various architectural contexts.

The political atmosphere in Europe was dramatically shifting toward freedom and autonomy for the common man, alongside a growing sense of revolt against the established governing classes, especially toward the end of the 18th century. In this dynamic and transformative climate, the Frenchman Francois Cointeraux emerged as a pivotal figure who rediscovered and fervently championed the ancient technique of rammed earth construction. In the year of 1791, Cointeraux took significant steps to promote this innovative building method by issuing a series of comprehensive rammed earth booklets in the city of Lyon. These booklets were meticulously translated into several languages, including English, French, German, and Italian, which greatly facilitated the dissemination of this valuable technology across Europe and even into the emerging landscape of the United States. This widespread circulation of ideas played a crucial role in the adoption of rammed earth as an alternative and sustainable building technique in various regions.

Earthen construction experienced a significant decline in popularity with the onset of the industrial revolution in Europe, primarily due to the increased availability and widespread use of burnt brick, which became the preferred building material of the time. However, this ancient technique saw a notable resurgence after the devastating impacts of the two world wars. In the aftermath of the First World War, a series of innovative experiments involving rammed earth and chalk-based structures were conducted in the United Kingdom, paving the way for a renewed interest in these sustainable practices. Subsequently, East Germany adopted rammed earth construction methods after the conclusion of the Second World War, which ultimately resulted in the establishment of the first comprehensive building standards specifically tailored for this resilient and eco-friendly material. As a consequence, the versatility and adaptability of earthen construction gained recognition once more, promoting a blending of traditional techniques with modern standards in the realm of architecture [47].

Fig.1. 8. Alhambra Palace, Granada in Spain [55].

1.2.2.4. Earth building techniques in North America

Native Americans residing in modern-day Mexico and the southern regions of the United States made extensive use of earth as a vital building material for their structures. The Aztec civilization, known for its significant contributions to architecture, constructed major monumental buildings using cut stone throughout Mexico. However, it is believed that the vernacular homes and everyday dwellings of many indigenous communities were primarily made from adobe, a natural building material composed of clay, sand, and straw. In Arizona, the Hohokam civilization showcased remarkable architectural skills by building adobe dwellings, which featured somewhat sunken floors that were meticulously carved into the alluvial soils of the region. These impressive adobe constructions, particularly those located at the Casa Grande National Monument, have been dated to approximately 750 A.D., providing invaluable insights into the architectural practices of that time. Meanwhile, the Pueblo peoples of present-day Mexico exhibited their architectural prowess by constructing sizable adobe houses designed to accommodate several families, with some of these structures reaching impressive heights of multiple stories. The most renowned example of such architecture is the Taos Pueblo, which has a rich history that dates back to roughly 1000 A.D. and remains an important cultural landmark that reflects the resilience and ingenuity of Native American communities.

Many missions and frontier forts that were erected by Europeans in North America extensively utilized adobe as a primary building material, including notable examples such as the adobe Tamacacori, Guevavi, and Calabazas Jesuit missions located in Arizona, which were all constructed in the year 1691. Furthermore, the governor's mansion situated in Albuquerque was also built using adobe, dating back to 1706. As European colonization continued its relentless spread westward, there was an increasing need for security among settlers, which prompted the construction of various forts designed to provide safety and protection against potential assaults from Native American tribes. The adobe structures of Fort Union, established in 1851, and Fort Selden, which came later in 1865, serve as enduring testaments to the US Army's remarkable capability to construct resilient defenses utilizing whatever materials were readily available in the environment. These forts not only played a crucial role in safeguarding the settlers but also represent the architectural ingenuity of that era, making noteworthy contributions to the historical landscape of North America.

Many cities located on the vibrant west coast, particularly notable ones such as San Jose and Los Angeles, may have originally been constructed using adobe, a traditional building material, at some point in their rich histories. However, due to relentless urban growth and constant rebuilding efforts over the years, very few of the original adobe structures remain intact or have survived the test of time. A solitary yet significant adobe wall, which was skillfully erected in 1822 and forms part of the original lodges around which the esteemed university was organized, still stands proud and resilient at Santa Clara University in San Jose. Another fascinating example is Casa de Estudillo, a prominent historic site situated in the lively Old Town area of San Diego, which was built from adobe in 1829 and has recently undergone a thoughtful renovation to preserve its historical significance. The influx of Chinese immigration to the west coast of the United States was notably encouraged by the allure of gold mining opportunities, and these immigrants brought with them unique construction techniques, such as rammed earth, which were previously unheard of in the region. Juana Briones, an enterprising businesswoman from Palo Alto, California, made a significant contribution to the building landscape by erecting a rammed earth and cob house in the year 1845. Furthermore, Chinese immigrants constructed a rammed earth herb shop in 1855 in Palo Alto, known as the Chew Kee Store located in Fiddletown, California. The approximately 150 rammed earth dwellings that are concentrated in the picturesque San Antonio Valley within Monterey County, which were built around the year 1896, are most likely attributable to the skilled efforts of later European immigrants who brought their craftsmanship to the area. The rammed earth construction method was eventually introduced to the eastern coast of the United States by industrious German immigrants who had honed the technique in their homeland. An interesting historical structure, Hilltop House, located in Washington, D.C., was constructed using the rammed earth method in the year 1773. Furthermore, in 1812, Bushrod Washington (the nephew of George Washington) took the initiative to erect rammed earth cabins situated on his Mount Vernon estate, which is located in Alexandria, Virginia. Although the future US president and prominent architect Thomas Jefferson was certainly aware of this innovative construction technique, it remains unclear whether he personally utilized it to construct any of his buildings. However, it is worth noting that the slave quarters at the Bremo plantation in Virginia, a design by Jefferson, were constructed using this rammed earth material by his friend General John Hartwell Cocke around the year 1819, showcasing the technique's application during that era.

S.W. Johnson, an innovative thinker, created a remarkable rammed earth home in Trenton, New Jersey, where he drew inspiration from the pioneering work of Francois Cointeraux in Europe. To further educate and inspire, Johnson released a comprehensive brochure in 1806, specifically detailing the process of rammed earth construction. His hope was to provide a valuable blueprint for newly arrived Europeans who were eager to settle and cultivate farmland in America. This innovative construction style was championed in the 19th century by John Stuart Skinner, who was the influential publisher of The American Farmer magazine. Skinner dedicated considerable effort to promote this fascinating technique, publishing numerous insightful articles focused on the unique properties and advantages of rammed earth construction.

As the interest in this building method grew, many others started to experiment with rammed earth, leading to a flurry of articles appearing in various publications of the time discussing these advancements. One notable figure was William Anderson, a professor from South Carolina who strongly advocated for rammed earth construction. In 1850, he boldly erected the Church of the Holy Cross in Stateburg, South Carolina, showcasing the potential of this material to create sturdy and attractive buildings. Following this, in 1867, a new Marine hospital was planned for New Orleans with the specific objective of experimenting with new and innovative construction techniques. The architectural design involved an iron frame complemented by rammed earth panels used as infill, which was quite avant-garde for its time. Unfortunately, this ambitious project experienced significant budget overruns, was never completed, and ultimately faced destruction.

Rammed earth was also utilized effectively in the construction of St. Thomas Church located in Shanty Bay in 1838, as well as in the building of various residences across Greensville in 1868, both situated in Ontario, Canada. As the 19th century drew to a close, the advent of railways revolutionized the construction industry by making it much more convenient to transport heavy building materials across the nation. Consequently, this development led to a decline in the popularity of locally sourced building materials such as rammed earth and adobe, as builders began to favor other options. However, the interest in rammed earth experienced a resurgence in the 1920s, greatly influenced by the well-known English architect Clough Williams-Ellis, who recognized its potential.

In 1924, the architect Karl Ellington published an enlightening book that contributed to this renewed interest, and just two years later, in 1926, the Department of Agriculture issued Bulletin No. 1500. This important publication provided detailed guidance on the various methods involved in rammed earth building. The challenges presented by the Great Depression and the subsequent New Deal programs in the early 1930s inspired a number of labor-intensive construction techniques to be explored. Notably, in 1935, Thomas Hibben successfully erected seven rammed earth houses in Gardendale, Alabama, illustrating the continued relevance and adaptability of this ancient technique. Around the same period, Dr. Ralph Patty and his colleagues conducted thorough erosion testing, which they published at South Dakota Community College. Their findings led to the development of essential technical documentation for those interested in pursuing rammed earth building, ensuring that this traditional method would continue to evolve and find new applications in modern construction.

Around the year 1940, a passionate advocate for rammed earth construction, David Miller, undertook the ambitious project of constructing his own rammed earth home in the historic area of Greely, Colorado. This significant endeavor not only led to the establishment of the Rammed Earth Institute but also served to inspire and motivate a new generation of modern earth builders. Among these influenced individuals were notable figures such as David Easton, Paul Graham McHenry, and Bruce King, who carried forward the valuable principles of sustainable building practices [47].

1.2.2.5. Earth building techniques in South America

The richest and most significant place for archaeological evidence of earth construction in South America is without a doubt the coastal districts of northern Peru. A recently uncovered temple at northern Peru's intriguing Ventarron site is believed to have been meticulously built using pieces cut straight from the river mud and it dates back to roughly the year 2000 BC. The earliest known earth bricks can be traced back to the Moche civilization, which thrived between the years 100 and 800 AD in the fertile lands of Northern Peru. The city of Cerro Blanco was recognized as the epicenter of this remarkable civilization, featuring two breathtaking pyramids that were devoted to the sun and the moon. Among these architectural wonders, the Huacas del Sol and de la Luna rise to an impressive height of 50 meters, showcasing extraordinary adobe-core construction. The distinct markings found on the individual adobe bricks strongly indicate that these monumental constructions were built by numerous separate and skilled groups working collaboratively. Meanwhile, the Lima civilization, which flourished between 100 and 650 AD in central coastal Peru, was contemporaneous with the Moche culture and also made significant contributions to earth construction. The Huaca Pucllana and the Huaca Juliana, both reaching an impressive height of 25 meters, serve as prime examples of this culture's adobe pyramids, showcasing their unique technique of placing adobes vertically. Notably, the Nazca culture, best known for its enigmatic Nazca lines, established its capital in adobe at the significant site of Cahuachi, which is still the subject of ongoing excavations today in the southern region of Peru. Although there exists limited archaeological evidence of vernacular building practices, it is highly likely that adobe bricks were utilized extensively in both monumental and vernacular structures across various cultures. Following the decline and eventual fall of the Moche culture around 750 AD, the Lambayeque culture began to flourish, continuing the esteemed tradition of constructing adobe pyramids at significant archaeological sites such as Batan Grande, Tucume, and Apurlec. The Chimu civilization, which emerged around 900 AD and established their capital of Chan Chan near what is now the bustling city of Trujillo in northern Peru, represents the largest civilization to arise after the decline of the Moche.

Chan Chan, boasting a vibrant population of up to 26,000 people and impressive adobe walls soaring to about 15 meters in height, was likely the largest city on the entire continent during that historical period. This remarkable urban center features ten 'royal' enclosures, each one surrounded by sturdy adobe walls that rise an impressive 9 meters high and are adorned with intricate relief designs that showcase the artistic talent of its builders. The Incas, a powerful empire, eventually conquered the Chimu civilization renowned for its monumental architecture constructed with expertly cut stone. Despite this conquest, it is highly likely that the traditional vernacular architecture continued to thrive, with buildings still being constructed from adobe, reflecting the enduring legacy and cultural practices of the Chimu people.

When European settlers made their arrival in the New World, they brought along with them a variety of new construction methods that had been developed and refined in Europe. These techniques were essential for establishing missions and constructing villages that would serve as the first footholds for their new societies. In a notable instance related to the construction of the Colego da Campanhia in São Paulo, a Jesuit missionary issued an urgent plea to Europe in the year 1549, specifically calling for 'artisans qualified to handle loam, and carpenters, for the construction of a rammed earth wall.' This appeal highlights the challenges faced by early settlers in employing skilled labor for their ambitious building projects. Over time, as a result of the influx of European architectural styles and methods, São Paulo emerged as a significant center for rammed earth construction, showcasing a blend of both monumental and vernacular building styles.

One of the impressive landmarks that arose from this period is Taubate's remarkable rammed earth Cathedral, which was successfully completed in 1645, showcasing the architectural prowess of its time. Additionally, the Church of Our Lady of the Rosary, which stands as a testament to the era's craftsmanship, was completed in 1720. Furthermore, the rammed earth House of the Chamber, constructed in 1776, reflects a style that is remarkably comparable to that prevalent in Portugal during the same period, mirroring a similar architectural aesthetic that was evident in various regions of southern Europe.

However, the architectural landscape of São Paulo faced significant challenges when major flooding occurred in 1850, which rendered many structures unstable and posed a risk to public safety. This environmental catastrophe sparked a widespread public outcry against earthen architecture, prompting authorities and citizens alike to call for the removal of the majority of the city's traditional earthen structures. Despite these challenges faced by urban centers, it is worth noting that in many Andean sections of South America, adobe construction remains a prevalent and culturally significant building technique that continues to endure through generations. This enduring legacy of adobe architecture serves as a reminder of the rich cultural heritage and the adaptability of building methods in response to the diverse landscapes of the continent [47].

1.2.2.6. Earth building techniques in Australasia

The nomadic aboriginal inhabitants of Australia are known for their traditional lifestyle, during which they did not utilize earth construction methods. In contrast, European immigrants, seeking to establish their presence in this vast and diverse land, attempted to employ a variety of building styles that were typical of their home countries. An early and significant mention of rammed earth in the region can be traced back to the Hobart municipal gazette of May 1823. In a resolution made at that time, it stated that the pise, or rammed earth, construction method appeared to this Society to be both an inexpensive and expedient solution suitable for the needs of the time. Consequently, the Society strongly recommended its adoption throughout Van Diemen's Land, highlighting its practical benefits.

Fast forward to 1839, a newspaper based in South Australia documented the construction of no less than 30 rammed earth dwellings, emphasizing this technique's growing popularity. Rammed earth was frequently utilized as a rapid construction method during the two notable gold rushes and in the frontier settlements, where the demand for quick and sustainable housing surged, exemplified by places like Penrith in New South Wales and Rushworth in Victoria. Interestingly, various types of buildings were employed by European immigrants in New Zealand, where both rammed earth and adobe were experimented with in hopes of discovering effective building solutions. However, the unfortunate earthquakes that struck in 1846 and 1855 led to a decline in the favor of all types of masonry construction, as the safety concerns overshadowed previous enthusiasm.

One of the most well-known historic earth monuments in New Zealand is Pompallier House located in Russell, which was erected in 1841. This structure stands as a testament to the early architectural endeavors of that era. G.F. Middleton, an English-trained architect, played a significant role in promoting rammed earth construction during his work at the Commonwealth Experimental Building Station. His dedication to the method led him to conduct numerous rigorous tests, all of which were meticulously documented in the well-regarded Bulletin No. 5 of 1953. This publication became the recognized standard reference for rammed earth construction in both Australia and New Zealand, holding considerable influence in the building community until relatively recently [47].

1.2.3. Earth construction in Algeria

In the year of 1969, a notable construction effort led to the building of 136 mud-brick houses in the agricultural village of Bouhlilet, which is located in the Batna region. Following this, in 1971, a collaborative Franco-Belgian team undertook the interesting task of creating an experimental set of rural dwellings in the town of Zeralda. The projects continued to flourish, as in 1973, 30 houses were constructed out of a total of 300 mud houses within the village of Mustapha Ben Brahim, situated in the Sidi Bel Abbès Province. In a significant advancement in construction techniques, the village of Abadla was established in 1975 utilizing the innovative earth-filling process, and the following year, a commendable achievement was marked by the completion of 100 housing units in the agricultural village of Felliache located in Biskra, which were constructed using the Toub method. The year 1980 saw the establishment of 120 homes in the agricultural village of Madher, in Boussaada, all fashioned out of compressed earth blocks (CEB), which contributed significantly to sustainable building practices. The trend continued in 1981 when 40 additional homes were constructed in Cheraga, which is situated near Algiers, also utilizing the efficient CEB method. In 1984, a fascinating bioclimatic prototype was developed in Tamanrasset, constructed with CEB materials, alongside another prototype built at CNERIB showcasing the same materials. By 1986, the trend persisted with the construction of 10 properties in Adrar, also utilizing CEB materials, as well as another set of 10 properties built in Reggane using the same CEB method. Fast forwarding to 1994, a total of 24 housing units were constructed in Tamanrasset by the property development and management office using CEB, while an impressive 44 additional housing units were similarly established by the Tamanrasset ETR, also utilizing the CEB method. Moving into 1996, an innovative adobe prototype was executed at CNERIB, showcasing yet another method in sustainable housing. Most notably, in 2006, an ambitious project titled "Energy-efficient rural housing development" was launched at CNERIB, backed by funding from the European Union, highlighting the continuous evolution of eco-friendly housing initiatives in the region [56].

Fig.1. 9. Clay building, M'Zab Valley in Algeria [57].

Fig.1. 10. The village of Felliache in Biskra

1.3. Main advantages and disadvantages of mud constructions

Advantages

- A large number is available locally.

- It just takes a small amount of energy to manufacture.
- The material is completely recyclable.
- There is a lot of calorific inertia.
- Hygrometric control is excellent.
- Sound and thermal insulation are excellent.
- Relative humidity control in the house Thermal inertia is aided by it.
- It is breathable, healthful, and free of toxic gases.
- Contributes to the home's acoustic and aesthetic comfort.
- The pricing is unbeatable.
- Excellent long-term durability.
- It requires little upkeep.
- Fire resistance is excellent.
- Xylophagous insect resistance.
- Mold and fungus resistance.

Disadvantages

- The biggest flaw is the length of time it takes to apply building processes.
- There are no professional norms governing these constructive methods.
- It necessitates specialized knowledge, both in terms of application and technique selection.
- The earth's makeup varies significantly.
- It is necessary to have some background knowledge in the field.
- Raw earth is more or less vulnerable to poor weather depending on its makeup.
- Between clay soils, there is a lot of chemical dispersion (Dispersion in water and flocculation: fine particles remain in suspension)

1.4. Earthen construction techniques

The installation technique used is determined by the masons' or carpenters' culture and expertise. Unlike terracotta, raw earth is found immediately on the ground. The building procedures differ depending on the earth's flexibility and granular texture. The primary mud building techniques were identified by Houben and Guillaud [58] as shown in Fig.1. 11.

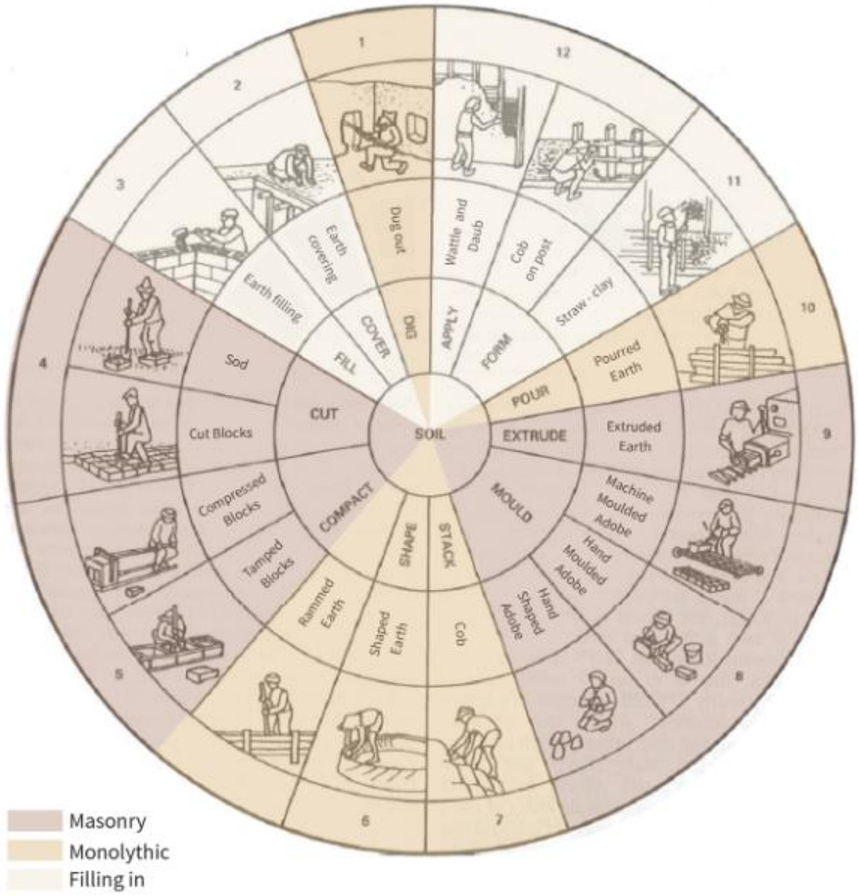


Fig.1. 11. Wheel of different earth construction techniques CRATerre [59].

The following is a list of the key characteristics of the various earth-building methods:

1.4.1. Adobe

Adobe is a mud-brick hand-shaped or molded then dried for a few days in the open air. Clays, silts and sands are mixed with water to achieve a plastic state, and sometimes with fibers to reduce cracking during drying [60, 61] (see Fig.1. 12).

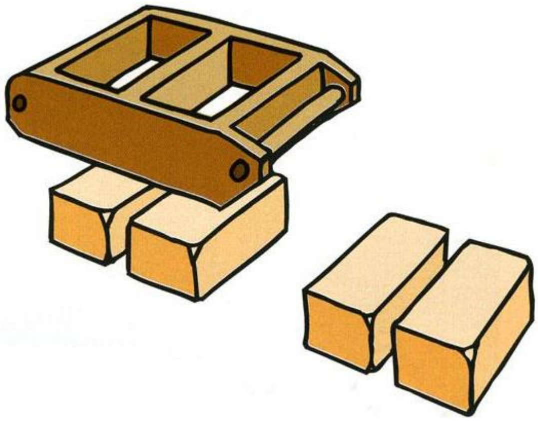


Fig.1. 12. Adobe construction.

Shibam in Yemen is one of the twenty medieval adobe sites designated as UNESCO World Heritage Sites (Fig.1. 13).

Fig.1. 13. Mud skyscraper city of Shibam Hadramout in Yemen, co-named "Manhattan of the desert" the world's first skyscrapers classified by UNESCO [62].

Characteristics:

- Density from 1.4 to 1.8 T / m^3
- Sandy clay soil, with a good proportion of sand

Implementation:

- The earth, cleared of stones, crumbled, sifted, is watered until it almost becomes mud. It rests 48 hours before being mixed with the feet, fork, rake ... Vegetable or animal fibers are added to it, for 30% of the total volume.
- The mold is a simple wooden frame without bottom made to the chosen dimensions. It allows one or more blocks to be molded at a time.The classic dimensions are those of bricks: 22cm long, 10.5cm wide and 5cm high (or 28 x 13.5 x 5, or 40 x 20 x 8.5, etc.).
- The mold is wet, a portion of the mixture is thrown in (expelling air bubbles), leveled and removed from the mold on the ground sprinkled with sand or straw. The block is turned over several times to homogenize the drying, which takes 3 to 4 weeks.
- The earth can also be shaped by hand, or cut with a wire or a spade. The bricks are made near the deposit, dried, and then transported to the site.
- The blocks are sprinkled before being assembled, with earth mortar or lime, on a stone or brick base, at a rate of 80-100 cm per day so that the mortar settles. The thickness of the wall depends on the positioning of the header (width) or panneresse (length) of the blocks. The traditional work is generally plastered.

1.4.2. Rammed earth

Rammed earth makes it possible to build massive walls, which can be load-bearing, by tamping between the formwork of thin layers of powdery earth. As the mixture is barely wet, form stripping is immediate as shown in Fig.1. 14.The compacted strata remain visible, with a texture rich in grain and color.

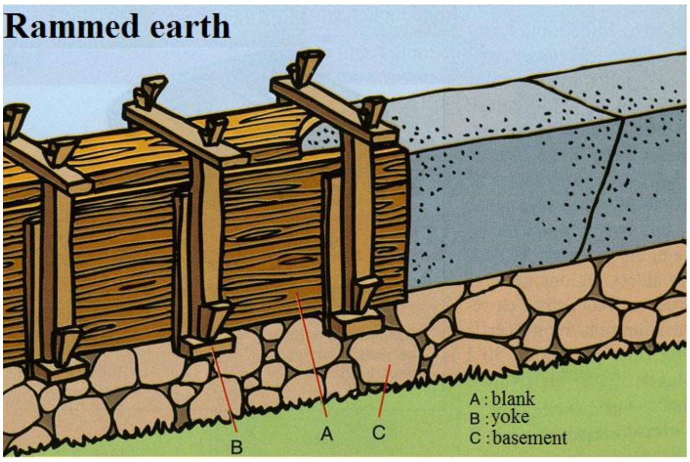


Fig.1. 14. Rammed earth construction

Because of the technicality associated with the use of formwork, rammed earth is more recent than adobe. Very old remains (9th century BC) have been spotted in Tunisia, and many UNESCO World Heritage sites bear witness to this sections of the Great Wall of China and in Algeria the Mansourah monument in Tlemcen (as shown in Fig.1. 15 and Fig.1. 16).

Fig.1. 15. The Great Wall of China at Mutianyu [63].

Fig.1. 16. Minaret of Mansourah in Tlemcen, Algeria [64].

Characteristics:

- Density: 1.7 to 1.9 T / m^3.
- Soil with a sandy or sandy-gravel texture. Loamy or clay-sandy soil is also suitable.

Implementation:

- The earth, mixed and moistened (8 to 12% water) is stored nearby. It is not amended. Currently, the transport of the earth and its mixing are mechanized. Rammed earth is sometimes stabilized with natural hydraulic lime. The setting, faster, is then carried out under plastic sheeting, in 2 or 3 weeks.
- The earth is compacted in a formwork. The formwork (shuttering) is made up of 2 wooden panels (currently in plywood or chipboard) 2 to 4 m long and 80 to 100 cm high. It is placed astride the base, maintained by through keys which will be embedded in the masonry and then removed during stripping. With a bunker bottom (board closing one end), the banchée is stopped vertically. With a single plank, the plenum connection will be at an angle (at the equillade).
- The first layer of soil of about 12 cm is thrown into the wall, compacted at the feet and then tamped in 2 passages, by one or two scavengers.
- A new soil bed is added, and so on. The ditch is completed when the compacted earth reaches the top level of the formwork. This is immediately dismantled and reassembled. Each new ditch is placed astride two others of the lower layer.
- The first lift (layer of earth) is completed when the tour of the building is completed.
- The next lift is asked when the withdrawal of the previous one is completed (in 1 or more days).

- The openings are made gradually, by wooden frames placed in the formwork, or drilled in the finished wall, at the location defined by the frames and lintels embedded in the rammed earth during its construction. In order to limit erosion and/or "hang" a lime plaster, a weather-resistant lime mortar is often associated, 5 to 12 cm thick, with rammed earth.
- After restoration, a new weakly hydraulic lime plaster is, most of the time, applied to standardize the appearance, insulate and protect the rammed earth. It is applied with a trowel, in 2 or 3 coats, respecting the setting times.

1.4.3. Cob

A rarely utilized method, scarcely adopted in modern construction due to time constraints, is the lesser-known technique. A cob wall, comparable to rammed earth in its solid, monolithic structure, possesses a thickness ranging from 40 to 60 cm, or potentially even more. This wall typically comprises clusters of earth balls, occasionally fortified with plant fibers and occasionally mineral components like flint fragments or crushed terracotta. Throughout most regions of the world, these walls are meticulously shaped by hand, resembling immense sculptures. This technique was extensively employed in Europe, referred to as "Cob" in England and "Bauge" in France. It bears resemblance to adobe construction, although in certain instances, a larger proportion of straw fibers is incorporated. Noteworthy instances can be found in places like Saudi Arabia (depicted in Fig.1. 17) and the historical structures of Shibam, Yemen, where a combination of cob and rammed earth techniques is employed.

Fig.1. 17. Al Masmak Palace in Riyadh, Saudi Arabia [65].

Characteristics:

- Density: 1.7 T/m^3
- Clayey to clayey-sandy soil

Implementation:

- The earth extracted is stoned, spread and wetted. It is kneaded until it becomes a paste to which fibers are mixed (rigid straw, hay, rush, horsehair, etc.). The mixture is trodden several times before being brought to the chosen place.
- After equipping a base 30 to 90 cm high, forks of mix are placed on it, flat or obliquely, with an overhang of about 5 cm. Lifting is complete when the entire length of the wall is filled, 60 to 90 cm high. The overhangs are packed down with a cudgel (single stick). After one to four weeks of drying, shrinkage, 2 to 3 cm, is carried out.
- Standing on the levee, the mason installs a line or a plank directly above the base. He removes the excess soil by cutting the side of the wall using a paroir (flat spade with a long handle, with a sharp edge), or, nowadays, a hay cutter, a long metal blade. As with

rammed earth, openings are made during construction or cut out after drying, depending on the region.

1.4.4. Wattle and daub (WAD)

Different methods for wattle and daub construction are employed based on the geographical area. In the conventional approach, a framework of wooden components supports the packed earth. This method has been utilized for nearly six millennia [66]. In this context, the earth does not serve a structural purpose; it may incorporate straw, resulting in a predominantly clay-rich mixture. Conversely, the load-bearing capacity is provided by the timber framework. This technique is suitable for non-load-bearing walls and is applicable for both exterior and partition walls up to a thickness of 20 cm. Fig.1. 18 portrays two residences constructed using wattle and daub in Germany and France, respectively.

Fig.1. 18. (a) A structure crafted from wattle and daub in the heart of Bad Langensalza, Germany (Image: Captured by Sebastian Wallroth); (b) A residence located in Alsace, France (Image: Courtesy of the Auroville Earth Institute) [66].

Characteristics: Clayey-loamy soil.

Implementation

- The earth is trodden, wetted and spread on the ground. Made into a soft paste (20 to 35% water), it is amended with chopped vegetable fiber (barley, rye, wheat, flax, etc.)

but also animal. Kneaded and re-wet, the mixture (usually straw torches bound to clay or a straw-earth-water mixture) rests for 1 to 2 days.

- The nature of the WAD depends on the type of half-timbering it will fill: the base receives doves whose section determines the thickness of the WAD wall. The spacing of these posts (10 to 80 cm), the presence and number of scarves (pieces of bracing placed at an angle) define the implementation.
- Three types of WAD support are found, sometimes simultaneously in the same locality: half-timbered splints (splinters of wood inserted at an angle), structures fitted with girders (vertical pieces of wood) and those with wattlework , or covered with laths.
- The splints serve as an attachment structure for straw torches coated with WAD.
- The yokes are interlaced with rods (grid). They serve as a trellis for the mixture.
- The lattice is installed on the 2 sides of the wall and serves as WAD formwork. After drying, the lattice is covered with the same mixture.
- The trellis is wetted before being filled, by hand or with a trowel, from bottom to top and from the ends towards the center. Everything is smoothed.
- The first lined side dries for a day, before the second side of the wall is filled with WAD, which clings to the first layer.
- Several passages make it possible to plug the cracks due to shrinkage.
- Streaks are made on the outside before drying, in order to hang the subsequent coating, applied one to two months later. This lime plaster is applied with a trowel, after wetting the support. It can cover the wood, thus protected.
- The long implementation times mean traditional WAD is only used for rehabilitation. There are ready-to-use daubs on laths, which can be projected by machine. New insulation techniques are similar to WAD, with plants prepared and bound with hydraulic lime, such as hemp mortar. The walls, 40-50 cm thick, dry in 3 to 4 weeks. Hemp mortar is also increasingly used as interior insulation.

1.4.5. Compressed earth bricks

Compressed earth blocks (CEB) are made in manual or mechanized presses with moist earth (Fig.1. 19), composed of a balanced proportion of clays, silts, sands and small gravels. The addition of cement or lime is common to increase the mechanical characteristics and the resistance to water [67, 68].

Compressed earth blocks (CEB) have only recently appeared. Around 1950, the first manual press produced 300 to 800 bricks daily, it conquered the international market by its simplicity and lightness. After several refinements, the technique took off within the framework of economic housing programs in Africa, for example Algeria (Fig.1. 20).

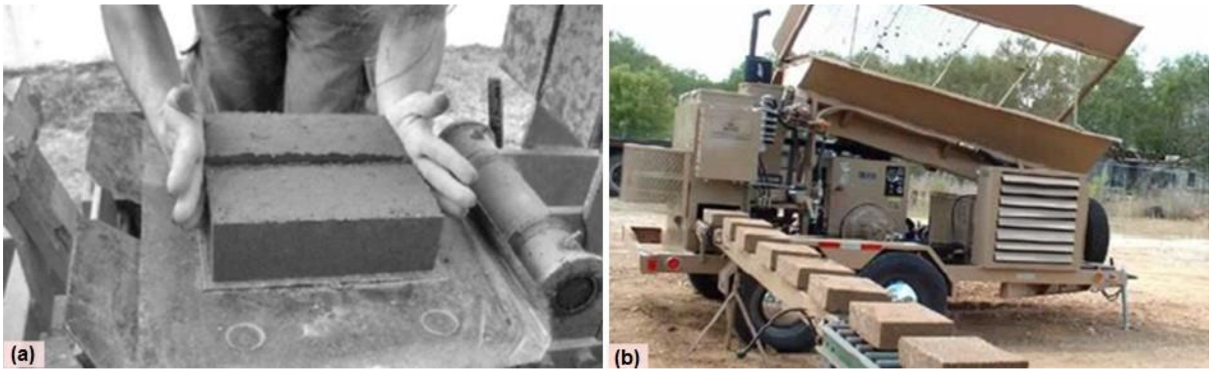


Fig.1. 19. CEBs manufactured by means of (a) a manual press (b) a hydraulic press [69]

Fig.1. 20. House from CEB in Algeria [70].

Advantages:

- CEB is an eco-friendly material.
- CEB provides excellent thermal and acoustic comfort.
- CEB offers great resistance to compression.

- The CEB presents an architectural and aesthetic interest.
- The CEB allows a wealth of forms and varied patterns in its use.
- CEB is simple to implement.

Disadvantages:

- Brick-making is slow.
- Fragility: at the slightest shock the brick breaks or crumbles.
- Deteriorates under the effect of frost.

1.5. Soil and classification

To effectively detail the diverse materials encountered during ground investigations, it becomes imperative to adopt a structured framework for describing and categorizing soils. Such a framework should encompass a wide range of deposits, remain relevant to engineering contexts (thus facilitating comprehension and interpretation by engineers), and yet maintain a degree of conciseness. It is crucial to differentiate between the processes of description and classification:

Soil description involves articulating the physical attributes and condition of the soil. This description can pertain to either a soil sample or the soil in its original location. It is derived from visual examination, basic tests, on-site observations, knowledge of geological history, among other factors.

On the other hand, soil classification entails segregating soils into distinct classes or groups based on shared characteristics and potentially similar behaviors. A classification designed for engineering applications should predominantly rely on mechanical properties, such as permeability, stiffness, and strength. The designated classification of a soil can then be incorporated into its descriptive account.

1.5.1. Mineral compositions of soil (Clay Mineral Composition)

This section provides a concise overview of the composition and structure of clay. The significance of clay lies in its role as a cohesive element in soil, where its cohesive properties are crucial for the strength characteristics of earth mortar. Notably, clay particles are visible only under a microscope, each enveloped by a water film due to surface tension. This water film is responsible for binding the particles together [71].

According to Ogunye (1997)[72], the atomic structure of clay minerals involves two fundamental building blocks: tetrahedral units composed of silica and octahedral units composed of alumina, as shown in Fig.1. 22. The ([SiO_4]4-) tetrahedron contains a silicon atom equidistant from oxygen or hydroxyls. This tetrahedral arrangement forms a sheet with a hexagonal structure, where oxygen atoms at the base corners are coplanar, shared between adjacent tetrahedral. These sheets possess varying chemical compositions based on clay type, hydration level, and spacing. The inter-sheet spacing ranges from 7 to 20 Angström [59].

In general, certain sheets consist of silica (silicon surrounded by oxygen atoms), while others comprise alumina (aluminium atoms surrounded by oxygen atoms and OH groups). In some instances, the base may consist of Si and Mg or Si and Fe, expanding beyond just Si or Al. Thus, the silica tetrahedron sheet can be visualized as a layer of oxygen at the base and a layer of hydroxyls at the tetrahedral tips. Within the octahedral unit, aluminium, iron, or magnesium atoms are equidistant from six oxygen or hydroxyls. The bonding of two or more molecular sheets leads to the formation of distinct clay mineral groups, as depicted in Fig.1. 21. The primary clay minerals are kaolinites, illites, and montmorillonites. Although the soils under investigation are discussed in section 3.2.1, a brief examination of the other two primary soil minerals is deemed necessary for comparative purposes.

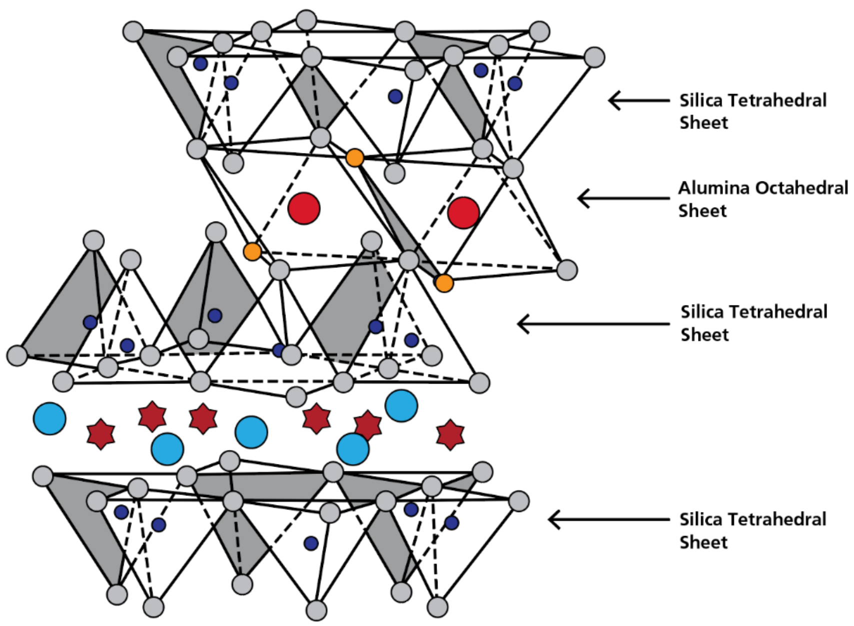


Fig.1. 21. Various units in clay raw materials in Tetrahedral Sheet (silica sheet) and Octahedral Sheet 1[73].

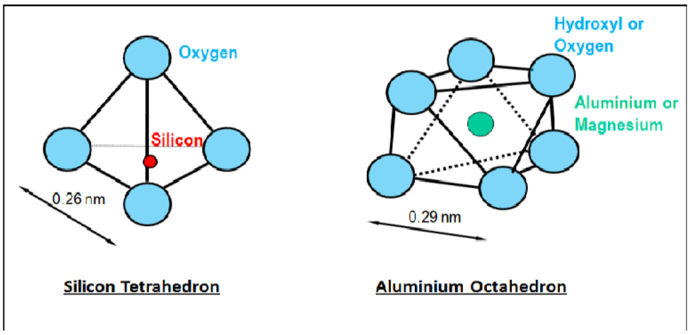


Fig.1. 22. Types of layer structure of clay minerals [74].

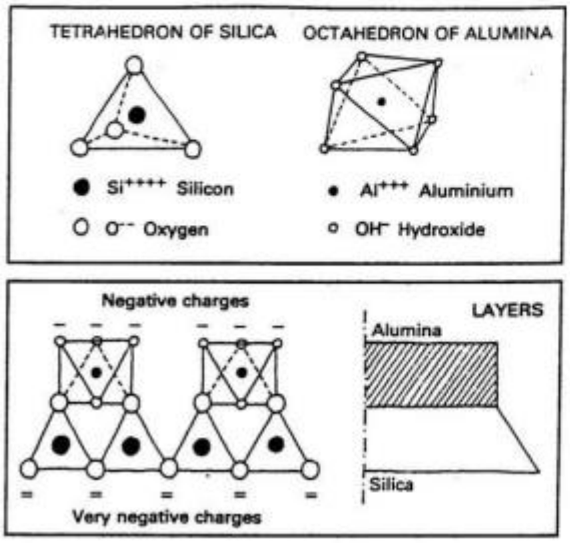


Fig.1. 23. Layer arrangement [74].

Kaolinite

Primarily originating as a product of alteration resulting from the weathering of feldspar and muscovite under acidic conditions, kaolinite possesses a non-expansive nature. It is commonly found in extensively weathered, well-drained soils, particularly prevalent in tropical regions. Structurally, kaolinite comprises a singular silica tetrahedral sheet and a sole alumina sheet, combined into a repeating unit with the generalized formula [$Al_2Si_2O_5(OH)_4$], as seen in Fig.1. 22, Fig.1. 23 and Fig.1. 24.a. The spacing between these sheets remains constant at 7 Angstrom, and the crystal's thickness ranges from 0.005 to 2μm, as depicted in Fig.1. 22. Notably, the arrangement of charges within the structure is in equilibrium, meaning no excess charges exist within the lattices due to substitutions within the lattice. From an engineering perspective, kaolinites are regarded as the most stable among clay types [72].

The hydrogen bonds linking these elemental sheets exhibit sufficient strength to prevent the penetration of water molecules and other ions, rendering the lattice non-expansive. As a result, the accessible surface area to which water molecules can be attracted is limited to the outer surfaces.

Illite

Illite has a basic three sheet structure consisting of gibbsite combined with two sheets of silica as seen in Fig.1. 24.b. In the silica sheet there is partial substitution of silicon by aluminium. The combined sheets are linked together by relatively weak bonding due to non-exchangeable potassium ions held between them [75]. According to Powrie [76], illites have the same basic structure as the non-clay mineral muscovite mica. Illite differs from these minerals in that fewer of the silica Si^{4+} positions are taken by aluminium Al^{3+}, so there is less potassium between the layers. Illite particles are smaller than mica particles and the layers are more randomly stacked. Illite may also contain magnesium and iron in the gibbsite sheet. For example, iron-rich Illite which has a characteristic green hue is known as glauconite. Illites occur as small, flaky particles mixed with other clay and non-clay minerals. The distance between sheets is 10 Angström and the thickness of the crystal is between 0.005 and 0.05µm. Unlike Kaolinite and Montmorillonite, their occurrence in high-purity deposits is unknown [76].

Montmorillonite

Montmorillonite has the same basic structure as illite, a three sheet structure comprising of a sheet of gibbsite between two silica sheets as seen in Fig.1. 24.c. Montmorillonites have a similar basic structure to the non-clay mineral group known as pyrophyllites [76]. According to Craig [77], there is a partial substitution of aluminium by magnesium and iron in the gibbsite sheet. In the silica sheet there is partial substitution of silicon by aluminium. The space between the combined sheets is occupied by water molecules and exchangeable cations other than potassium, resulting in very weak bonds which are easily separated by the adsorption of water. For these reasons, Powrie [76] states that the Montmorillonite particles are very small and can swell significantly by the adsorption of water. Therefore, soils which contain Montmorillonites exhibit a substantial potential for volume change and are sometimes termed expansive soils. The distance between the sheets ranges from 14 to 20 Angström. Thickness of the crystal layers lies between 0.001 and 0.12 µm.

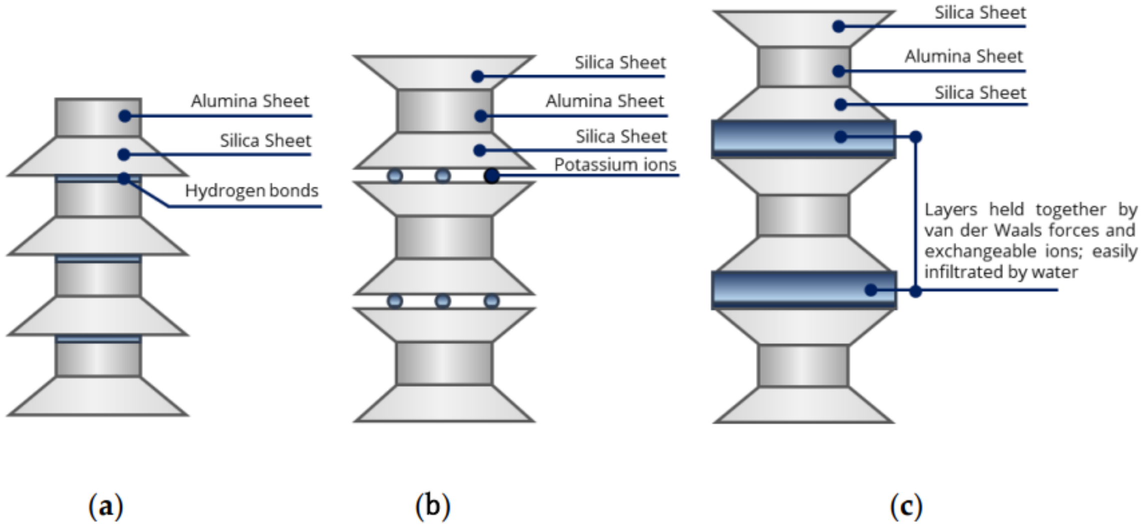


Fig.1. 24. Main groups of clay minerals. (a) Kaolinite (1:1 non-expanding); (b) illite (2:1 non-expanding); (c) montmorillonite (2:1 expanding).[78]

1.5.2. Types of soil

Soil, in its broadest sense, encompasses the layer of loose material covering the Earth's surface. This material is a complex mixture that includes disintegrated rock, humus (organic matter), as well as inorganic substances. The formation of soil from rock is a slow process, typically taking 500 years or more to complete. It occurs as a result of various natural forces acting on rocks, causing them to break down into their constituent components. These forces encompass a range of factors, including the mechanical impact of wind, erosion by water, and chemical reactions involving salts.

Soil development can be divided into three distinct stages:

- **Solid Soil**: In the initial stage, the soil consists primarily of solid particles, with little or no air or water present in the pores between these particles.
- **Soil with Air in the Pores**: As soil matures, it begins to incorporate air into the spaces between the solid particles, allowing for improved aeration and root respiration.
- **Soil with Water in the Pores**: The final stage sees the infiltration of water into the soil, filling the spaces between the particles. This water provides the necessary moisture for plant growth.

Soil types vary widely in response to environmental factors. The classification of soil primarily depends on its texture, which is determined by the relative proportions of sand, silt, and clay, as well as the presence of different forms of organic and mineral compositions.

There are four main types of soil:

- **Sandy Soil**: Sandy soil is composed of small particles derived from weathered rocks. It is characterized by low nutrient content and poor water retention capacity, making it challenging for plant growth. However, it excels in drainage capabilities, making it ideal for applications where good drainage is necessary.
- **Silt Soil**: Silt consists of smaller particles than sand but larger than clay. It has a fine texture that retains water better than sandy soil. Silt is often found near water bodies like rivers and lakes and is relatively fertile, making it suitable for agriculture.
- **Clay Soil**: Clay soil comprises the smallest particles among the three soil types. Its tightly packed particles result in excellent water retention but poor drainage, making it difficult for plant roots to thrive. When wet, clay soil is sticky and compact, but it becomes smooth when dry.
- **Loamy Soil**: Loam is a well-balanced combination of sand, silt, and clay, incorporating the best qualities of each. It retains moisture and nutrients effectively, making it highly suitable for farming. Loam is often referred to as agricultural soil and is rich in organic matter (humus). It also typically has higher calcium and pH levels due to its diverse mineral composition.

1.5.3. Chemical and Physical properties of soils

Before undertaking any significant construction or earth-based projects, it is imperative to thoroughly assess the properties and characteristics of the soil. Previous research has established the critical importance of understanding soil's physical attributes in such endeavors [46].

One of the primary considerations for soil suitability in earth building walls is its composition. Ideally, the soil should have a high sand content with just enough clay to act as a binder. Excessive clay can lead to issues like cracking due to shrinkage effects [79]. An essential characteristic to evaluate is the particle size distribution or texture of the soil, which plays a pivotal role in determining its suitability. Soils that are highly plastic clay, clean gravels, sandy, or organic in nature are generally considered unsuitable and not recommended for earth construction [80-83].

The swell capacity and soil shrinkage are closely related to the clay content within the soil. A basic formula often employed is to have 30% clay with the rest comprising loam and a small amount of aggregate [84]. The silt content should be less than one-third of the soil composition. The California Uniform Building Code Specification recommends a range of 55% to 75% sand and 25% to 45% clay and silt [85], as indicated in Table.1. 1.

Table.1. 1 A suggested mixture that works well for most blocks.[86]

Specifications	Percentage
Sand	65
Clay	20
Silt	15

Various proposals have been put forth, suggesting a balance between clay/silt and sand within the range of 30% to 70% [87]. Some research has indicated that the upper limit for clay/silt should not exceed 45% [85]. According to Burroughs (2001), the ideal range for clay/silt is 36% to 45%, while sand should be present at levels between 66% to 75%. Additionally, the minimum sand content should fall between 50% to 55%, while the maximum should be in the range of 70% to 75%.

Moving on to soil characteristics, it is crucial to understand that engineering behaviors vary significantly between clays and sands [88, 89]. The principal distinctions lie in particle size distribution, shape, and plasticity, with secondary characteristics encompassing factors like texture and color [90, 91]. Moreover, chemical properties are influenced by the composition of soil components and the quantities of elements like magnesium, calcium, carbonate, iron oxides, and sulphates. Soil pH, in general, tends to remain close to neutral [92].

1.6. Current Practices in Earth Mortar Stabilization

Earth offers an affordable and sustainable option for creating eco-friendly building materials. However, ensuring resistance to water damage and achieving adequate mechanical strength are key challenges in earthen construction. To address these issues, chemical stabilizers like lime, cement, or a combination of both are commonly incorporated. The primary methods for stabilizing earth materials include physical, and chemical stabilization techniques [93].

1.6.1. Chemical Stabilizers

Stabilizing earth mortar is essential for enhancing its strength, durability, and resistance to weathering. Several techniques are commonly employed in construction to achieve this stabilization, ensuring that earth mortar can withstand the test of time. Here are some of the key techniques:

1.6.1.1. Lime Stabilization

Lime stabilization is a well-established technique in earth construction. It leverages lime's properties to improve the performance and durability of earth mortars. This method combines traditional knowledge with modern sustainable construction practices, highlighting lime's enduring strength and eco-friendly nature [8, 91, 94].

- Chemical Reactions and Binding

Lime stabilization relies on chemical reactions to enhance soil properties. When lime, either as quicklime or hydrated lime, is mixed into earth mortar, it undergoes hydration, forming calcium hydroxide. The calcium hydroxide then reacts with soil particles to produce calcium silicates and calcium aluminates. These compounds act as effective binding agents, increasing the cohesion between soil particles [95].

- Enhanced Workability and Durability

Lime stabilization improves both the workability and durability of earth mortar. Earth mortars of sand, silt, clay, and organic matter can challenge their variable plasticity. Lime acts as a plasticizer, making the mortar more malleable and easier to handle, thus reducing the risk of cracks and inconsistencies [95]. Furthermore, lime-stabilized mortar exhibits greater water infiltration, weathering, and mechanical stress resistance. This enhanced durability makes it suitable for load-bearing walls, foundations, and other structural elements requiring long-lasting performance [96].

- Environmental Benefits

Lime stabilization offers significant environmental advantages. Lime is derived from limestone, a plentiful natural resource, making it a sustainable choice. Its production requires less energy than cement, aligning with sustainability goals. Additionally, the carbonation process, where lime absorbs carbon dioxide from the atmosphere during curing, helps reduce greenhouse gas emissions, enhancing its environmental profile [97].

- Versatile Applications

Lime stabilization is versatile and can be applied in various construction projects, from traditional homes to modern buildings and historic preservation. It is particularly valuable in heritage conservation for preserving historical authenticity. Its compatibility with modern architectural trends prioritizing eco-friendliness positions lime stabilization as a technique that bridges traditional practices with contemporary environmental needs [98].

1.6.1.2. Cement Stabilization

Cement stabilization is a prominent technique in earth construction that leverages the properties of Portland cement to substantially enhance the performance and durability of earth mortars. This transformative process significantly improves the strength and resilience of earth-based building materials, rendering them suitable for a broader range of structural applications. The effectiveness of cement stabilization hinges on the intricate chemical interactions of Portland cement when introduced to earth mortar; the cement undergoes hydration, wherein cement particles react with water to form calcium silicate hydrates (C-S-H) and calcium hydroxide [99]. These compounds serve as potent binding agents, creating a robust matrix interlocking with soil particles, enhancing cohesion and compressive strength. This newfound strength enables structures to withstand substantial loads more effectively, ensuring structural integrity and longevity. Beyond strength enhancement, cement stabilization provides dual benefits to earth mortar: improved workability and enhanced durability [100]. Earth mortars, often composed of a heterogeneous blend of sand, silt, clay, and organic matter, can present challenges during construction due to inconsistent plasticity. However, the addition of cement acts as a plasticizer, increasing the mortar's malleability and ease of handling, thus simplifying construction processes and reducing the likelihood of undesirable cracks or irregularities. Furthermore, cement-stabilized mortar demonstrates markedly improved resistance to water infiltration, weathering, and mechanical stress. This enhanced durability makes cement stabilization ideal for demanding long-lasting performance, such as foundations, walls, and pavements, ensuring the creation of robust and resilient structures [101].

1.6.1.3. Mineral and geopolymer additives

Additives can be incorporated to stabilize earth mortar and enhance its properties and performance. These materials address specific challenges and improve the adaptability of earth mortar in construction. Admixtures, such as pozzolans (e.g., fly ash or silica fume) and superplasticizers, modify mortar properties. Pozzolans enhance workability, reduce water demand, and improve durability and resistance to chemical attack. Superplasticizers enhance flow and workability without increasing water content, improving handling during construction. Polymer-based additives like latex or acrylic polymers improve bonding and adhesion, which is useful in applications like plastering or rendering where strong adhesion is crucial.

Emerging chemical stabilizers, such as geopolymers and biopolymers, offer environmentally friendly alternatives to traditional methods. Geopolymers, synthesized at low temperatures from alumino-silicate materials like metakaolin, enhance mechanical performance and thermal properties, offering comparable stability to Portland cement-stabilized materials while reducing the carbon footprint [102]. Biopolymers, derived from living organisms, have been used for soil stabilization since prehistoric times, with examples including proteins, plant extracts, and even termite saliva . While natural polymers like pine resins can improve strength, they may also increase water absorption . Bio-cementation, an environmentally friendly biochemical process that improves soil properties through the precipitation of calcium carbonate, is also an area of growing interest, although its high cost and reliance on specific conditions pose challenges for practical application

1.6.2. Physical Stabilizers

Physical stabilization of earth mortar involves modifying the soil's inherent properties through texture correction and incorporating diverse additives and reinforcements. Texture correction entails adjusting the soil composition, where Guettala et al. [103] demonstrated that adding sand can reduce ductility and improve stability. The strategic addition of fibers, such as straw, mesh, bamboo, jute, or hemp, strengthens the material's structure, effectively preventing cracking as the clay component shrinks during the drying process, and creating a reinforcing network that enhances tensile strength [104]. Bouhicha et al.[105] suggested that plant fibers reduce shrinkage in soil. This is crucial in regions susceptible to structural movement or seismic activity.

Furthermore, expanded materials like glass beads or perlite can be incorporated to reduce the density of the mortar, yielding lightweight earth mortar that is particularly advantageous for insulating layers or applications where minimizing structural weight is a priority [106]. Eco-friendly alternatives, such as natural reinforcements like rice husks or coconut fibers, offer a sustainable approach to enhancing mechanical properties and promoting green building principles. Finally, geogrids and mesh reinforcement systems provide robust structural reinforcement, especially against lateral forces, making them ideal for earthbag construction and similar techniques, ensuring the creation of durable and resilient earth-based structures [107, 108]. Millogo et al. [109] observed that short kenaf fibers reduce crack propagation in earth blocks.

1.7. Curing Earth Mortar: Nurturing Strength and Durability

Curing is critical in stabilizing earth mortar, ensuring maximum strength and durability by maintaining optimal moisture and temperature during drying and setting [110]. This process is essential for properly hydrating stabilizing agents like lime or cement, preventing incomplete chemical reactions that can weaken the mortar [111]. Curing begins immediately post-compaction to prevent premature moisture loss, requiring temperature control to avoid cracking from high temperatures or slowed reactions from low temperatures [112-114]. The duration varies, necessitating techniques like covering surfaces with wet burlap or applying curing compounds to retain moisture, alongside regular monitoring with moisture meters for precise adjustments [115]. Ultimately, curing is integral to quality control, ensuring effective stabilization through regular cracks and moisture content inspections.

1.7.1. Testing and Quality Control in Earth Mortar Construction

Testing and Quality Control in Earth Mortar Construction

Testing and quality control are indispensable practices in earth construction, ensuring the integrity, safety, and reliability of structures built with earth mortar [116]. These processes involve various assessments and checks to verify that earth mortar meets specified standards and performance requirements [4].

- Key Testing Methods

Compressive strength tests determine the ability of earth mortar to withstand axial loads, measuring the maximum load samples can withstand before failure in a controlled laboratory setting [117]. Moisture content checks assess moisture levels, crucial for preventing weakening from excessive moisture or hindered curing from inadequate moisture, typically measured with specialized equipment [117]. Consistency tests, such as slump and flow table tests, evaluate workability and consistency to ensure suitability for applications like rendering or masonry. Durability assessments subject cured earth mortar to environmental stressors like freeze-thaw cycles to evaluate long-term resilience [118].

- Material Analysis, Field Testing, and Non-Destructive Testing

Material analysis examines components like soil types and stabilizing agents to ensure they meet project specifications, often involving laboratory testing. Field testing assesses performance on-site, including in-situ tests of structural elements to verify strength and stability [119]. Non-destructive testing (NDT) methods, such as ultrasound or ground-penetrating radar, assess the integrity of structures without causing damage, valuable for evaluating existing structures or detecting hidden defects [120]. Quality control documentation ensures proper recording of all tests and inspections, providing a comprehensive record of quality and performance, while compliance with established industry standards and guidelines ensures that earth mortar meets regulatory and safety requirements, assuring durability and safety [121].

1.7.2. Mix Design for Earth Mortar: Crafting Strength and Versatility

Mix design in earth mortar construction hinges on carefully selecting and proportioning ingredients to achieve the desired properties and performance, guided by the principles of soil science and material behavior [103, 122, 123]. A critical aspect involves soil selection, characterized by evaluating particle size distribution and plasticity to ensure suitability for the intended application, such as adobe, rammed earth, or compressed earth blocks; according to Fig.1. 25, the granularity nomograms illustrate recommended areas for particle size distribution, showing variations based on the construction method [122]. While specific percentages may vary, the general principle emphasizes a balance of clay, silt, and sand fractions to optimize strength, workability, and durability. Stabilizing agents, such as lime or cement, are chosen based on structural requirements and environmental considerations, while additives like fibers or pozzolans can further enhance specific properties [124]. This iterative mix design process requires careful consideration of material availability, cost-effectiveness, and environmental impact, often involving laboratory testing and field trials to validate performance and ensure compliance with relevant building codes and standards.

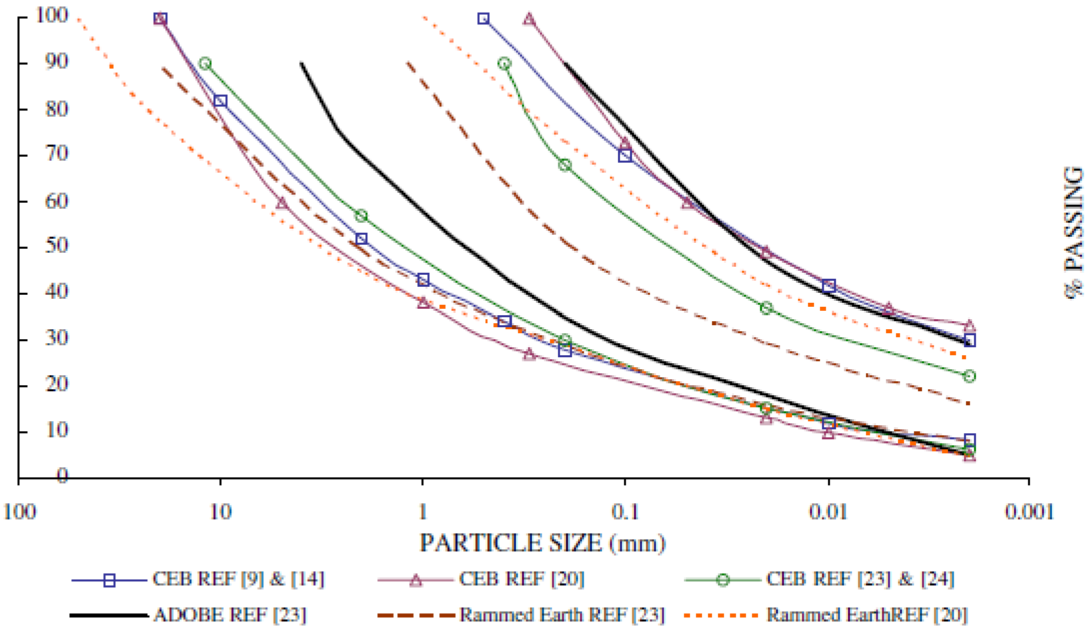


Fig.1. 25 Granulometric nomograms indicating optimal particle size distributions for adobe, rammed earth, and compressed earth blocks [122].

1.7.3. Protecting Earth Mortar Structures from Environmental Factors

Protecting earth mortar structures from environmental factors is crucial for ensuring their long-term integrity and durability, as it is susceptible to moisture, freeze-thaw cycles, UV radiation, pests, and seismic activity [125]. To protect against moisture, roofing with proper overhangs and sloping, water-repellent surface treatments, and efficient drainage systems is essential to prevent weakening and erosion [126]. In cold climates, adequate insulation and exterior cladding can minimize the effects of freeze-thaw cycles, which cause cracks and deterioration. Integrated finishes or natural coatings can shield earth mortar from UV radiation, preventing fading and surface degradation [127]. Regular inspections are necessary to deter pests like termites, and physical or chemical barriers around the foundation [128]. In earthquake-prone regions, seismic retrofitting and engineered designs adhering to earthquake resistance standards are vital [129]. Finally, routine maintenance, including crack repairs and reapplication of protective coatings, is crucial for addressing wear and ensuring the continued performance of protective measures [4].

1.8. Durability

1.8.1. Durability Concepts

The capacity to withstand wear and aging over time defines durability across all construction materials and structures. The term "durability" originates from the Latin word "Durabilis," meaning "durable," and has been interpreted in diverse ways by different authors. According to the sustainability guide BS 7543:1992 [130], durability, as defined, is the capability of a structure and its components to fulfill required functions over time, influenced by external factors. BSI CP3 1950 offers a contrasting perspective, viewing durability as a measure inversely related to the rate of material or component deterioration. In alternative literature [131], authors characterize durability as a gauge of a material's ability to sustain distinctive traits and weather resistance during the operational lifespan of the associated structure. All materials within a building envelope must execute their functions and endure with minimal upkeep throughout their designated service life. Failure to meet these performance criteria suggests exceeding the threshold of weather resistance, leading to deterioration when environmental forces are potent enough to disrupt the bond between constituent particles [132].

In the context of earthworks, durability is contingent on resistance, dimensional stability, and responsiveness to weather conditions. These characteristics are predominantly influenced by the selection of constituent materials and the quality of the manufacturing process applied in their creation. Variations may occur due to structural changes under exposure conditions, potentially resulting in a decline in performance. The durability limit marks the juncture where a decline in performance signals the conclusion of the material's service life (BS 7543, 1992) [130].

1.8.2. Mechanisms of Earth Mortar Deterioration

1.8.2.1. Water-Related Deterioration

Water plays a crucial role as a common factor in various deterioration mechanisms affecting blocks. The primary sources of water contributing to these mechanisms include rainfall, increasing humidity, and condensation. Water's impact can take on either a solvent or abrasive form.

Solvent Action

The solvent action of water is widely recognized as a prevalent deterioration mechanism, especially in construction materials like earth mortar. Leaching, the process of washing away soluble substances, is a key aspect. In blocks stabilized with cementitious binders, the main sources of solubles are calcium hydroxide and the clay fraction potentially present in the non-stabilized or partially stabilized residual matrix.

Dissolved calcium hydroxide may be carried away by surface runoff during rain or expelled to the block's surface through evaporation in high temperatures, resulting in efflorescence. Swelling clays, such as illite and montmorillonite, promote dispersion, with the dispersed particles being washed away during rainy seasons.

The irreversible dissolution process is favored by high temperatures and the presence of carbon dioxide. Prolonged leaching increases material porosity and causes flaking. The use of lime and pozzolans in conjunction with cement during stabilization can minimize the effects of leaching. Pozzolans and lime have the ability to fix both the calcium hydroxide in hydrated cement paste and any excess clay, respectively.

Abrasive Action

Abrasive action of water occurs in areas with frequent and intense precipitation, like humid tropics. Surface erosion in blocks happens when rainwater directly impacts an exposed surface, creating a spray that removes loose particles. Detached particles, usually from the non-stabilized fraction, are easily carried away by surface runoff. Surface abrasion results in decreased strength, stiffness loss, and increased permeability.

Beyond erosive effects, water can induce swelling or act catalytically, triggering harmful chemical reactions.

1.8.2.2. Freezing/Thawing-Related Deterioration

In regions where freezing and thawing phenomena occur due to environmental conditions, blocks may experience deterioration. Ice growth in pores, accompanied by hydraulic and osmotic pressures, leads to the accumulation of expansion forces within the pore system. These disturbances tend to alter particle distribution [133].

1.8.3. Temperature-Related Deterioration

When stabilized earth mortars are exposed to elevated ambient temperatures, they may undergo dimensional variations leading to cracks, warping, or fissures. Diurnal and nocturnal temperature variations, influenced by geographical location, serve as parameters for deterioration. These temperature fluctuations have the potential to impact the physical, mechanical, and chemical properties of blocks, influencing durability in diverse ways [131].

Swelling and Shrinkage

Swelling and contraction are continuous, cyclical phenomena inducing significant internal compression and traction stresses, resulting in degradation. Blocks with low tensile strength may develop surface cracks, not only impacting aesthetics but also facilitating moisture ingress [134].

Shrinkage and Drying of Clay

Two distinct shrinkage mechanisms may occur in a block. One involves the expulsion of water from capillary pores through evaporation, while the other is the shrinkage of water within the clay and hardened cement paste. The former is considered a reversible process, contrasting with the irreversible nature of the latter. High ambient temperatures facilitate moisture loss through evaporation. Although the expulsion of capillary water is reported as non-harmful to block structure, some water remains in the form of strongly adsorbed water in unstabilized clay plates and the gel of hardened cement. Gradual removal of this water occurs only at high temperatures and over extended periods, making the shrinkage process slow but significantly irreversible in the case of hardened cement paste.

Catalytic Action

The catalytic action of temperature variations in initiating chemical reactions is well-known. Chemical reactions unlikely at low ambient temperatures become more probable at higher temperatures. Temperature fluctuations influence moisture movement within a block. The combination of moisture and elevated temperatures forms a catalytic framework, reactivating dormant chemical activity. Reactions favored by this mechanism include the crystallization of soluble salts, oxidation, leaching, among others. A mere 10°C temperature increase is reported to double the speed of chemical reactions.

1.8.4. Biological Deterioration

Biological deterioration of earth mortar primarily stems from high moisture content. Fungal stains or molds may proliferate on masonry walls, and harmful crawling plants can develop when dust and dirt accumulate in cavities, wall cracks, and mortar joints, creating a conducive environment for seed growth. Roots can penetrate deeply, leading to additional cracks and water infiltration [135].

1.8.5. Chemical Deterioration

Earth mortars contain potentially reactive chemicals from the soil and binder. The soil, making up the majority of a block's mass, contains minerals and contaminants. Some of these substances may stay dormant until actively in contact with environmental elements like rainwater, high temperatures, relative humidity, and gases. The binder also holds potentially unstable chemical components, even in its hardened paste phase. Exposure to environmental agents can trigger chemical reactions [131]. The harmful mechanisms of chemical action can be outlined as follows:

Direct Cement Decomposition: This can occur due to acid attack. Neville (1995) [136] states that no cement is known to resist acid attack.

Formation of Expansive Products: The three main reaction categories likely to impact the durability of stabilized earth blocks through expansive product formation are:

Crystallization of Soluble Salts: This can occur in block pores, especially in sandy soils where salts are commonly found. Quantities as low as 6% of the sand mass can initiate these reactions. The most common salts are sulfates and chlorides, which become reactive only in solution. Alternating wetting and drying of block surfaces provide an ideal setting for these reactions, and rising moisture contributes to salt transport from groundwater [137].

The crystallization process involves salts in solution infiltrating capillary pores. Due to high temperatures leading to evaporation, moisture is removed, causing salt crystallization in the pores. The volume of crystals increases, and the block structure cannot withstand further expansion, leading to significant stresses, cracks, and disintegration on the block surface. Eventually, salts dislodge soil particles, washed away during rains [131, 138].

Alkali-Aggregate Reactions (involving silica and carbonates): These reactions occur between material constituents with the risk of forming expansive products. The reaction can take place between active silica and carbonates in soils, as well as alkalis present in trace amounts in cements or soils.

Direct Lime Decomposition: In contrast to potential issues with cement, Direct Lime Decomposition involves intentionally subjecting limestone to controlled thermal treatment to produce lime without generating carbon dioxide. Unlike cement's susceptibility to acid attacks, where no cement is resistant, lime production focuses on avoiding carbon dioxide release and minimizing environmental impact.

1.9. Sulfuric Attack

Sulfates exist in diverse forms in the natural environment, appearing in solutions within seawater, groundwater, industrial wastewater, acid rain, and even in the air as gaseous SO_2 or attached to particles like micrometric fly ash and nanometric soot. They are most commonly found in soils, often identified by brackish efflorescences observed on arid surfaces [138]. Clayey soils tend to have higher sulfate concentrations compared to sandy soils [131].

In the Earth's crust, prevalent sulfate types include calcium sulfate (gypsum or selenite $CaSO_4.2H_2O$), sodium sulfate (thenardite Na_2SO_4), potassium sulfate (arcanite K_2SO_4), and magnesium sulfate (epsomite $MgSO_4.7H_2O$). These sulfates are commonly found in regions with limited rainfall, such as parts of Australia, Africa, the UK, and Gulf regions, but notable concentrations can also be present in wetter areas. Sulfates may already exist in the soil or result from the oxidation of sulfide minerals, primarily pyrite FeS_2. Groundwater activation facilitates this oxidation and the transfer of sulfates to other locations. Despite the sulfate anion being represented as SO_4, soil tests typically express sulfate as SO_3 [139].

Origin Of Sulfates

Sulfates in soil form through the evaporation of saline waters or the processes of dissolution, transportation, and reprecipitation during cyclical drying/wetting periods. Dissolution is a form of water interaction with evaporable minerals in rocks. The geochemical transition between sulfate minerals and water is another interaction form. For instance, depending on geochemical changes, anhydrite can hydrate into gypsum, or gypsum can dehydrate into anhydrite. The transition phase is controlled by temperature, pressure, and liquid salinity.

Seawater contains around 3.6% gypsum-anhydrite. Upon evaporation, gypsum and anhydrite precipitate. Interpreting laboratory studies, Blatt et al (1980) [140] propose gypsum as the original mineral. However, even if anhydrite were the original mineral, it could convert to a more stable form, either calcium sulfate or gypsum, after precipitation. The gypsum-anhydrite transition (eq. 1) is a reversible reaction, influenced by the water activity a^2 H_2O. Processes decreasing water activity, like increased temperature or electrolyte concentration, lead to the reaction proceeding in the forward direction (dehydration) proportionally to the square of water activity [141].

$$\underset{gypsum}{CaSO_4.2H_2O} \rightleftarrows \underset{anhydrite}{CaSO_4 + 2H_2O} \qquad K_{eq} = a^2_{H_2O} \tag{1}$$

Gypsum and anhydrite, being soluble, can chemically react with compounds in cement, causing damage. The literature lacks absolute dissolution rates for these minerals. Liu and Nancollas (1971) [42] demonstrated small gypsum crystals dissolve in first-order kinetics (eq. 2).

$$dM / dt = KA(c_S - c) \quad (2)$$

Fabuss et al (1969) [142], on the other hand, illustrated that anhydrite dissolves following second-order kinetics (eq. 3).

$$dM / dt = KA(c_s - c)^2 \quad (3)$$

Where:

M: mass of dissolved calcium sulfate at time t.

cs: concentration of the substance in a saturated solution.

c: concentration of the substance in a solution at time t.

A: area of the mineral surface exposed to the aqueous solution.

K: constant.

Sulfate plays a crucial role in regulating the setting of cements during their application. However, under specific conditions, the uncontrolled development of sulfur compounds poses a threat to the durability of cementitious materials, a phenomenon known as "sulfuric attack."

Cements resistant to sulfates, with low C_3A content (less than 8%), and cements incorporating additional cementitious materials that consume portlandite (CH) in pozzolanic reactions generally display high resistance but are not completely impervious to this form of attack [143].

1.9.1.1. Case of Concrete and Cements

In the context of cements, mortars, and concretes, the formation of hydrated calcium aluminate (C-A-H) through the hydration of C_3A results in immediate stiffening of the paste, known as "flash set." To mitigate this effect, sulfates (gypsum, anhydrite, or hemihydrate) are introduced, reacting with C_3A to produce the primary ettringite. The development of this mineral is not problematic during this stage as cement setting has not yet occurred, and ettringite can form within the suspension [144].

In the case of gypsum ($CaSO_4$, $2H_2O$), the continuation of the C_3A hydration reaction is detailed as follows (eq. 4&5):

- Dissolution of tricalcium aluminate and gypsum:

$$Ca_3Al_2O_6 + 2H_2O \rightarrow 3Ca^{2+} + 2AlO^{2-} + 4OH^- \quad (4)$$

$$CaSO_4\ 2H_2O \rightarrow Ca^{2+} + SO_4^{2-} + 2H_2O \quad (5)$$

- Combination of ions in solution to form primary ettringite (hydrated calcium trisulfate) which crystallizes (eq. 6):

$$32H_2O + 6Ca^{+2} + 2AlO^{2-} + 3SO_4^{2-} \rightarrow 3CaO.Al_2O_3.3CaSO_4.32H_2O \quad (6)$$

With decreasing sulfate content, ettringite dissolves, releasing sulfate ions that then reassociate with aluminate, this time forming hydrated calcium monosulfate (eq. 7). Unlike ettringite, this mineral does not exhibit expansive properties.

$$3CaO.Al_2O_3.3CaSO_4.32\ H_2O + 6Ca^{2+} + 4Al(OH)_4^- + 8OH^- \rightarrow 3CaO.Al_2O_3.CaSO_4.12\ H_2O + 8H_2O \quad (7)$$

To permit partial or total decomposition of ettringite into monosulfate, the gypsum content in cements is restricted to a ratio of less than 3 moles of sulfate to 1 mole of C_3A [144].

Secondary ettringite emerges as a consequence of sulfuric attack, presenting a challenge as its development occurs several months or years after cement setting. This reaction, known as delayed ettringite formation, occurs as ettringite develops from a sulfur source at the expense of cement hydrates. Sulfur sources can be internal, involving the remobilization of sulfates from the cement matrix or alteration of minerals in aggregates (such as pyrite), or external, involving the transfer of sulfates from groundwater or soils.

In addition to ettringite formation, various sulfur compounds can arise from sulfides and sulfates, including sodium and magnesium, through reactions with cement hydrates (eq. 8, 9&10) [144]. For instance:

- Gypsum formation with portlandite:

$$Ca(OH)_2 + Na_2SO_4 10H_2O \rightarrow CaSO_4 2H_2O + 2NaOH + 8H_2O \quad (8)$$

- Gypsum formation with C-S-H:

$$3CaO 2SiO_2 aq + MgSO_4 7H_2O \rightarrow CaSO_4 2H_2O + Mg(OH)_2 + SiO_2 aq \quad (9)$$

- Ettringite formation with C-A-H:

$$3CaOAl_2O_3 6H_2O + 3(CaSO_4.2H_2O) + 20H_2O \rightarrow$$

$$CaO.Al_2O_3.3CaSO_4.32H_2O + 2Ca(OH)_2 \quad (10)$$

- Thaumasite formation from ettringite (eq. 11):

$$CaO.Al_2O_3.3CaSO_4.32H_2O + CO^{2-} + H_2SiO_4^{2-} \rightarrow$$

$$CaCO_3.CaSiO_3.CaSO_4.15H_2O + 12H_2O + Ca(OH)_2 + 2Al(OH)_4^{-} \quad (11)$$

Thaumasite, a mineral resulting from the transformation of ettringite by the substitution of alumina with silica and carbonate, is particularly influenced by the source of silica derived from C-S-H. Thaumasite formation is linked to a weakening of the cement [144].

Reports suggest that the presence of alkali metal sulfate (Na_2SO_4) can prevent ettringite formation due to aluminate hydrolysis, while gypsum hydrolysis ($CaSO_4$) leads to ettringite formation without hindrance [145].

Concerning ammonium sulfate $(NH_4)2SO_4$, the attack is more detrimental to concrete compared to sodium and magnesium sulfates. The mechanism of ammonium sulfate attack involves a combined acid/sulfate nature (eq.12, 31&14). In addition to sulfate ion attack, ammonium ions cause deleterious effects. Portlandite is highly soluble in ammonium solutions, and the ammonium cations neutralize due to acid hydrolysis with the cement matrix [143].

$$NH_4^+ + OH^- \rightarrow NH_3 + H_2O \quad (12)$$

$$Ca(OH)_2 + (NH_4\ )\ SO_4 \rightarrow CaSO_4.2H_2O + 2NH_3 \quad (13)$$

$$xCa.SiO_2.aq + x(NH_4)\ SO_4 + xH_2O \rightarrow SiO_2.aq + xCaSO_4.2H_2O + 2xNH_3 \quad (14)$$

The consumption of calcium hydroxide (CH) in equation (21) to form gypsum results in a pH reduction and increased porosity of the concrete surface in contact with the ammonium sulfate solution. Before or after the complete depletion of CH, decalcification of calcium silicate hydrate (C-S-H) occurs in equation (22) due to pH reduction in the pore solution. This reaction initiates a reduction in the calcium/silicate (C/S) ratio of the C-S-H structure, leading to a progressive loss of cohesion and eventual disintegration. Additionally, the formation of gypsum and, to a lesser extent, ettringite with gaseous NH3 in the pore structure can result in volume expansion [143].

1.9.1.2. Case of Stabilized Soils

Drawing parallels, considering that the elements engaged in the earlier reactions mirror those relevant in soils stabilized with cement or lime, these reactions prove apt for elucidating the sulfate attack mechanism on stabilized soils. During soil treatment, when sulfates are present, they can engage with portlandite and cement hydrates, resulting in cracks due to the swelling caused by expansive phases or the destruction of the binding phase, consequently compromising the acquired rigidity. Sulfate attack manifests a few days to weeks after the treatment. Predominantly, ettringite and thaumasite emerge as the compounds formed in soil scenarios, with the necessary chemical elements originating from either the soil or the binding agents [144].

1.9.1.2.1. Sulfate Assault on Cement-Stabilized Soils

The reactions governing sulfate attack and ettringite formation adhere to two mechanisms [146]:

- In the first mechanism, when sulfate-containing soil interfaces with cement, the cement acts as a source of supplementary calcium and alumina essential for ettringite formation, while the soil, containing pyrite or gypsum, serves as the sulfate source.

- In the second mechanism, cement serves as a source of additional calcium, while the soil itself provides the necessary alumina and sulfate for further ettringite formation.

Mehta (1983) [147] noted that sulfate adsorption on C-S-H surfaces diminishes strength due to a reduction in cementation capacity. Nonetheless, positive effects associated with ettringite formation have been documented, such as enhanced initial strength, delayed C3A hydration, and the capability to stabilize water-rich soils. The improved strength is ascribed to the removal of water by ettringite, leading to increased soil dry density [148].

1.9.1.2.2. Sulfate Assault on Lime-Stabilized Soils

In the case of sodium sulfate (eq.15), lime in the stabilized soil undergoes partial depletion via the following reaction:

$$Ca(OH)_2 + Na_2SO_4 \rightleftarrows CaSO_4 + NaOH \quad (15)$$

The presence of sodium sulfate transforms lime into insoluble gypsum and sodium hydroxide in the soil system. Consequently, the amount of lime interacting with the soil diminishes. However, the presence of sodium hydroxide elevates the pH, thereby augmenting the dissolution of soil silica [149]. The formation of sodium hydroxide as a by-product ensures the sustained high alkalinity crucial for C-S-H stability [131].

If the pH reaches 12.4, clay minerals (silicates SiO_2 and aluminates Al_2O_3) may dissolve or exist in an amorphous form. These compounds can then combine with the calcium and sulfates in the soil to generate ettringite [141]. The colloidal gel, from which ettringite crystallizes, possesses the capacity to attract numerous water molecules, causing repulsion between clay particles and an overall system expansion. Thus, if water is available, ettringite formation is accompanied by significant expansion [138].

Moreover, the presence of gypsum in lime-stabilized clayey soil introduces additional Ca^{2+} cations and SO_4^{2-} anions. The Ca^{2+} cations contribute to an increased total number of cations drawn to the surfaces of clay particles. The heightened concentration of Ca^{2+} ions in the soil system, due to gypsum addition, accelerates soil-lime reactions, culminating in improved cementation compound formation [149].

For other sulfate types, Ca^{2+} cations remain those supplied by added lime. In this scenario, cation exchange hinges on the position of the cation associated with sulfate in accordance with the lyotropic series order $Li^{+} < Na^{+} < K^{+} < Mg^{2+} < Ca^{2+} < Ba^{2+} < Al^{3+} < H^{+}$, relative to other cations already present in the clay-lime system [138]. Kinuthia et al. (1999) [150] observed that calcium and magnesium sulfates generated a substantial amount of ettringite, whereas sodium and potassium sulfates produced minimal ettringite.

Alterations in the properties of lime-stabilized soils in the presence of sulfates hinge on sulfate concentration, metal cation type, available calcium oxide and alumina quantity, and consequently lime amount, as well as the quantity and size of clay particles, temperature, and humidity [138].

1.9.2. Origins of Elements Leading to Ettringite Formation

1.9.2.1. Sulfates

Sulfate ions within the stabilized soil matrix may originate internally or externally. Internal sulfate attack is immediate, while external sulfate attack relies on the penetrability of sulfate ions [141]. Sulfur compounds may exist in the soil or its surroundings, such as gypsum deposits, sebkra, alteration of pyritic marls, metamorphic schists, soils contaminated by plaster waste, soils polluted with pesticides, seleniferous waters (groundwater), and runoff waters [144].

1.9.2.2. Calcium

Calcium can derive from lime dissolution or cement hydration. The elevated concentration of Ca^{2+} and high pH can impact clay particles, leading to the dissolution of silicates and aluminates. These dissolved components then combine with calcium, forming secondary cementitious materials and resulting in the stabilization of clay treated with lime.

1.9.2.3. Alumina

Ettringite consists of alumina produced from the dissolution of clay minerals [141]. Alumina can also originate from hydraulic binders. Two types of sulfate attack are identified based on alumina origin:

- Type I attack: Alumina comes from hydraulic binders.

- Type II attack: Alumina comes from clays through pozzolanic reactions.

Clays represent a potentially significant source of alumina for pozzolanic reactions. The contribution of alumina from clays introduces a risk of disturbances during stabilization with hydraulic binders. The nature and clay content are crucial parameters in ettringite formation. The proportion of releasable alumina depends on the clay type, with, for instance, smectite releasing more alumina than kaolinite. Even a low clay proportion of around ten percent is adequate to facilitate ettringite formation [144].

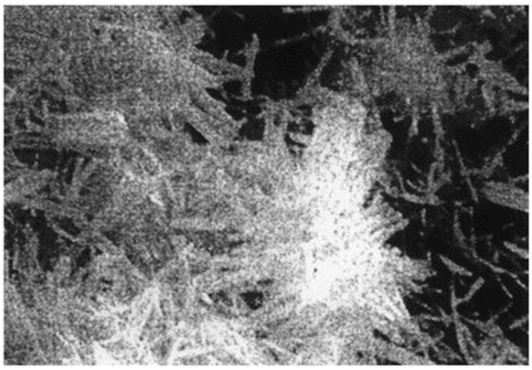


Fig.1. 26. Formation of ettringite in lime-treated kaolinite (after 6 months of curing) [149]

Tsatsos & Dermatas [151] endeavored to assess the mechanisms accountable for ettringite formation in lime-treated kaolinite and montmorillonite systems, in the presence of sodium sulfate. The results indicated substantial swelling in the kaolinite-lime-sulfate system due to the considerable availability of alumina for ettringite formation. However, in the montmorillonite-lime-sulfate system, the swelling effect was minimal due to the gradual release of alumina and a higher Si/Al ratio inducing the formation of calcium silicate hydrates (C-S-H).

1.9.3. Characteristics of Ettringitic Expansion

The expansion stemming from the formation of ettringite stands out as the most widely acknowledged mechanism in sulfate attack. Key properties associated with ettringite are intricately tied to its expansion. Extensive research by multiple investigators [152-154] has delved into the mechanisms driving this expansion. Theories and computational models have emerged to elucidate the process of ettringite formation and its subsequent expansion. Two primary conceptual frameworks underpin these explanations: the crystalline growth theory and the swelling theory [155].

According to the first theory, expansion occurs due to the growth of ettringite crystals, which emerge on the surfaces of expansive particles or within the solution. The growth of these crystals, referred to as crystalline growth, generates crystallization pressure and, consequently, the force driving expansion.

In contrast, the second theory posits that expansion results from the swelling of minute ettringite particles (1µm) possessing colloidal properties. These particles, known as gel, boast a substantial specific surface area and retain water, inducing expansion through swelling and leading to repulsion between clay particles. The formation of this gel is a product of the reaction between expansive particles and the surrounding solution. When the solution contains $Ca(OH)_2$, ettringite particles assume colloidal dimensions. In the absence of $Ca(OH)_2$, ettringite particles exhibit larger sizes. Expansion occurs only in the former scenario characterized by colloidal-sized ettringite crystals and the presence of $Ca(OH)_2$ [141, 155].

Ettringite formation persists as long as the necessary ingredients for the reaction remain abundant. Wild et al. [138] have reported that the ability to absorb water is confined to the stages of ettringite formation and is nonexistent upon its complete crystallization. On a contrasting note, ettringite can develop within void spaces, orchestrating its growth without significant expansion. Alternatively, it may form within a dense matrix, making it challenging for the soil matrix to accommodate the growth of these crystals. Fig.1. 27 elucidates the structural configuration of calcium, alumina, and water ions in ettringite.

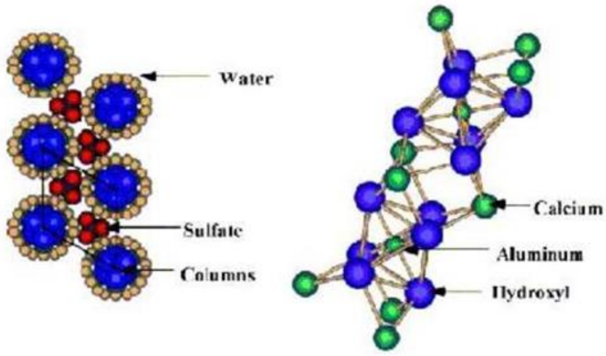


Fig.1. 27. Ettringite Structure [146]

The process of expansion during hydration has been scrutinized by several researchers. As per Lafuma [152], Mikhailov [153], Mehta [154], and Hunter [156], the hydration of ettringite triggers a volume increase of 200% from the initial volume. Notably, ettringite crystals are observed to grow in the void spaces of soils or massive rocks unrestrictedly, while those forming within dense soil or rock matrices exert a substantial pressure of approximately 241 MPa [146].

1.9.4. Factors Affecting Expansion

1.9.4.1. Impact of Sulfate Quantity

The level of sulfate content is a crucial factor indicating the formation of ettringite [141]. Wild et al. [138] examined the expansion of mixtures comprising kaolinite, lime, gypsum, and slag. Lime concentrations varied from 2 to 6%, and slag concentrations ranged from 0 to 4% (Fig.1. 28). Initially, the samples were kept at 30°C and 100% relative humidity, then immersed in water at 30°C. For a kaolinite-gypsum mixture treated with 6% lime, volumetric swelling increased fivefold as the sulfate concentration rose from 1.8% to 3.8%.

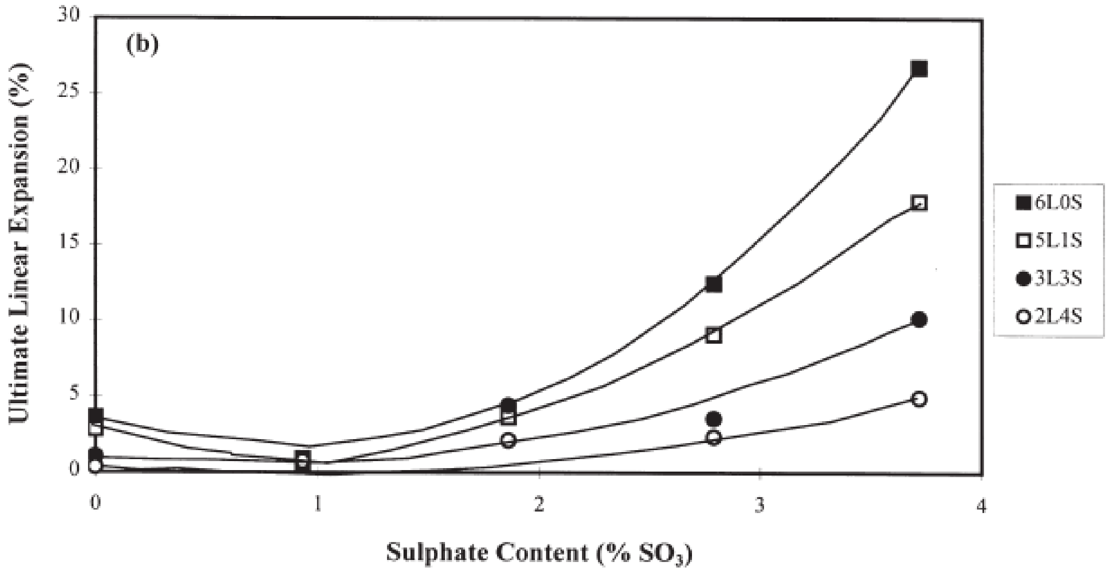


Fig.1. 28. Swelling after immersion based on sulfate content in a blend of Kaolinite, lime (L), slag (S), and gypsum at different concentrations. 6L0S = 6% lime and 0% slag [138].

When sulfur is present in minerals, considerations must be given to their solubility and dissolution kinetics, linked to grain size and specific surface area [144]. Table.1. 2 provides solubility values for certain sulfate compounds, and Fig.1. 29 illustrates the solubilization kinetics for calcium sulfate based on mineral size [141].

Table.1. 2. Solubility rates of specific sulfate compounds [144]

Chemical Compounds	Solubility Rate (g/L at 20°C)
Gypsum ($CaSO_4 \cdot 2H_2O$)	2.4
Hemihydrate ($CaSO_4 \cdot H_2O$)	9.0
Anhydrite ($CaSO_4$)	2.0
Sodium Sulfate (Na_2SO_4)	100.0

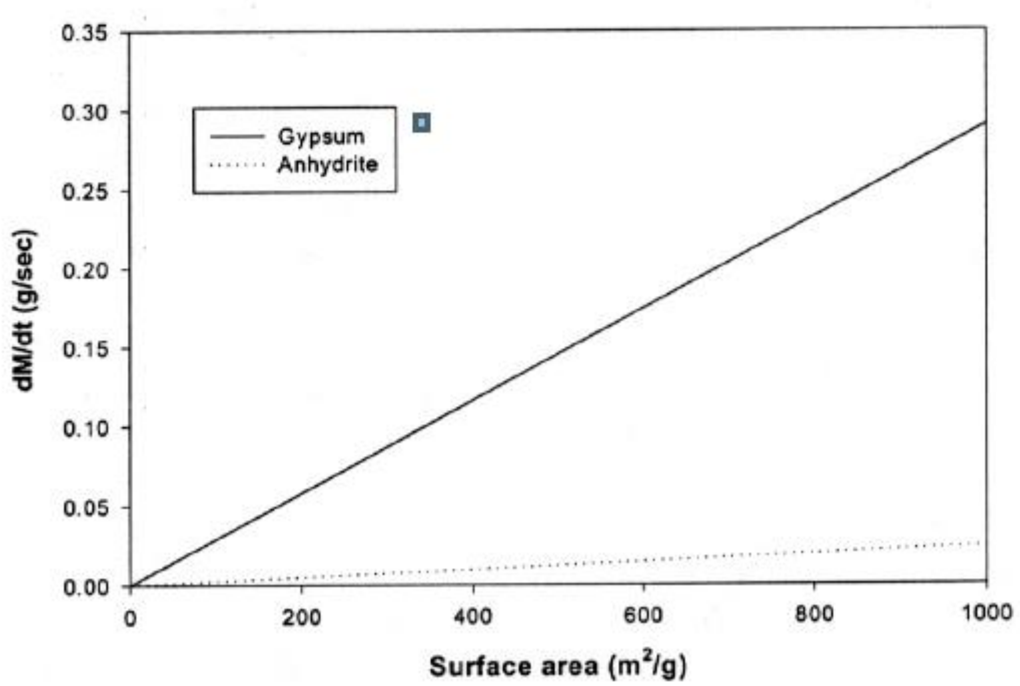


Fig.1. 29. Solubilization kinetics of calcium sulfate in relation to mineral size [141].

1.9.4.2. Impact of Soil Characteristics

The impact of sulfates on soil varies depending on its nature and mineral composition. Sherwood [157] demonstrated that the compressive strength of sand treated with 10% hydraulic binder (cement) remains unaffected by a 3% sulfate content, whereas soil containing clay experiences a 50% reduction in mechanical strength with a 0.2% sulfate presence.

1.9.4.3. Impact of Hydraulic Binder Type

During soil treatment with cement, the formation of cement hydrates creates a network that resists the soil-cement matrix, limiting ettringite expansion. Conversely, lime treatment does not immediately supply cementitious materials. The pozzolanic reaction between calcium hydroxide and clay minerals gradually produces additional cementitious materials, allowing ettringite to expand more freely [141].

1.9.4.4. Impact of Humidity

Swelling phenomena during curing vary based on sample storage conditions (air drying, at relative humidity, or immersion) [158]. According to Cabane [144], issues related to sulfur become apparent during immersion curing. Wang et al. [159] assessed the axial swelling of sulfur-containing soil treated with Portland cement under diverse curing conditions (from low humidity to total saturation). When specimens are stored in open air, axial swelling remains negligible over time, irrespective of the cement percentage used (Fig.1. 30).

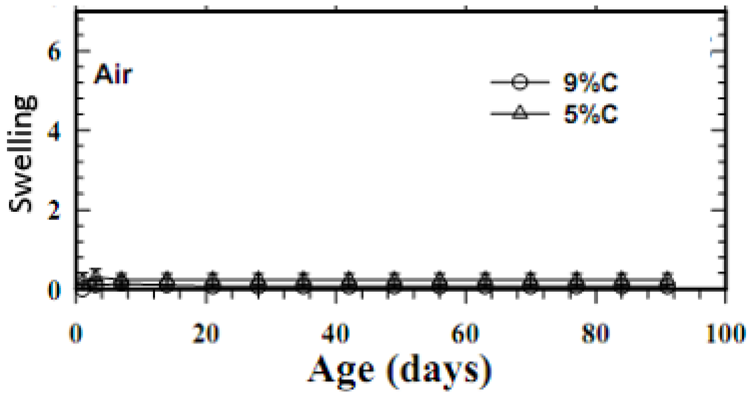


Fig.1. 30. Swelling over time for specimens stored in open air [159].

When specimens are stored at a constant water content in a sealed bag, axial swelling increases, especially with higher cement content (Fig.1. 31).

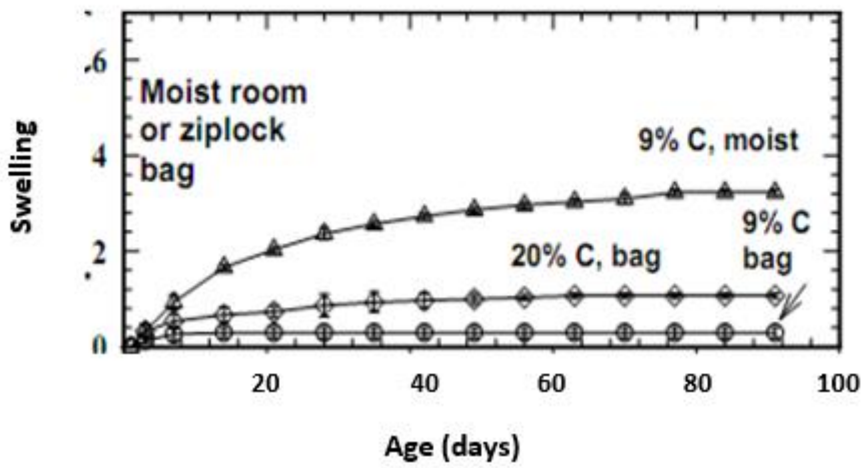


Fig.1. 31. Swelling over time for specimens in a humid chamber (moist) or at a constant water content (bag) [159].

The more humid the curing conditions, the more pronounced the ettringitic swellings. Swelling is also proportionate to the amount of hydraulic binder used for soil treatment.

1.9.4.5. Impact of Temperature

Wang et al. [159] investigated the temperature's effect on the swelling of sulfate-containing soil. Specimens were maintained at a constant water content and 40°C. An increase in temperature resulted in increased swelling, regardless of the added binder content (Fig.1. 32).

Elevating the temperature during specimen curing accelerates soil setting, leading to higher compression strengths for the same curing duration. In sulfur-containing soil, temperature expedites the expansive ettringite formation process, causing swelling [158].

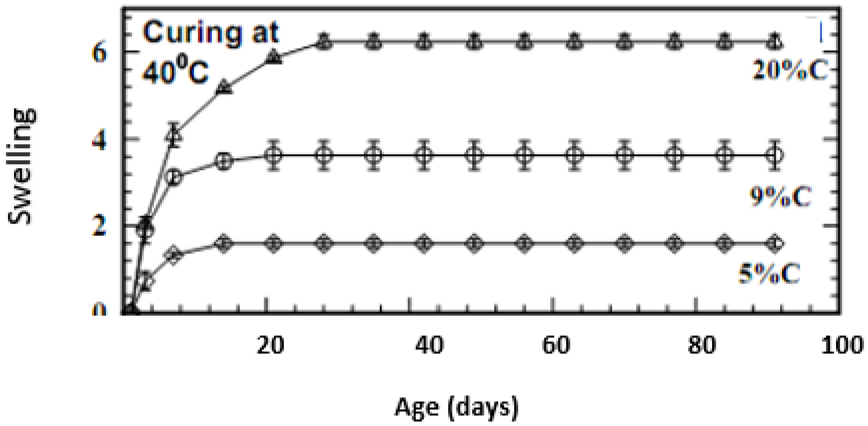


Fig.1. 32. Swelling over time for specimens stored at constant water content at 40°C [159].

1.9.4.6. pH Value Influence

The pH value plays a role in both the formation and expansion of ettringite. The use of cement and lime for stabilization raises the pH above 12. When the pH surpasses 9, there is a potential increase in the solubility of aluminates and silicates. This not only allows clay particles to engage in pozzolanic reactions but also contributes to ettringite formation due to the presence of aluminates. In a study on ettringite stability, Gabrisova et al. [160] demonstrated that the pH threshold for the disappearance of ettringite is 10.7. Below a pH of 10, only gypsum and aluminum sulfate remain stable.

1.9.4.7. Stress Rate Influence

The degree to which ettringite forms diminishes during the medium-term expansion process. This may be because a nucleus does not develop in areas where pressure needs to be exerted if it can develop more effectively in areas where pressure is not necessary. This suggests that ettringite crystals prefer growth in voids and cavities that contribute less to expansion. It has been explained that the extent of expansion caused by the development of a solid phase in a porous material depends on the stress rate generated and the material's ability to resist them. Very small ettringite crystals growing in small pores can exert strong pressure, while large ettringite needles growing in open fissures or other cavities produce less pressure.

1.9.5. Ettringite Identification

There is no satisfactory method for accurately identifying ettringite quantitatively. Kota [161] observed that expansive minerals like ettringite in lime-treated soil cannot be identified through X-ray diffraction (XRD). However, scanning electron microscopy (SEM) observations revealed the presence of needle-shaped crystals. Ludwig [162] found that less than 1% of ettringite could be identified using XRD. Additionally, Wang [141] pointed out that ettringite crystals in lime-treated soil, existing in small size and a weakly crystalline state or as a gel, cannot be identified through XRD.

1.9.6. Perturbation Thresholds

The concept of a threshold can be defined in various ways: it can be the concentration of a disruptive chemical element from which an impact on the soil is detected, while the material may still exhibit certain mechanical performance. Alternatively, it can be the concentration at which the mechanical performance of the material becomes inadequate. The threshold concept is considered an average value representing a homogenized material. However, geological variations within an excavation can result in undetected local enrichments.

In soil stabilization, disturbance thresholds related to sulfates vary among different authors depending on the treatment, soil characteristics, and sulfur form. Several authors have proposed sulfur content thresholds to proactively prevent these disturbances. Table.1. 3 provides a summary of various threshold values found in the literature:

Table.1. 3. Disturbance thresholds for soil treatment with sulfur compounds [158]

Stabilizers	Soil Types	Clay Minerals	Form of Sulfate Compound	Threshold
Lime	Different % of clay	-	-	<0.25%
Cement	-	-	-	<0.9%
Cement and gypsum	Loam	Illite and chlorite	Gypsum	<0.15%
Lime (4.5%)	Loam and clays	Sepiolite, montmorillonite, kaolinite	Gypsum	<1%
Lime (5%)	Calcium bentonite	-	Sodium, potassium, magnesium, aluminum sulfate	<0.05%
Lime	Clays	Smectite, kaolinite, montmorillonite	-	<0.03%
Lime	Sand + 30% clays	Kaolinite	Sodium sulfate and gypsum	<0.3%
Lime (3%)	Marine clays	Montmorillonite, kaolinite, sepiolite	Calcium and sodium sulfate	<0.2%
Lime	-	-	-	<1%
Lime (6%)	Black cotton	Montmorillonite	Sodium sulfate	<0.5%
Lime (3%)	Marine clays	Montmorillonite, chlorite, kaolinite, vermiculite	Sodium, potassium, magnesium, calcium sulfate	<0.05%
Lime	-	-	-	0.01% to 0.5% low to moderate risk0.5% to 1.2% moderate to serious risk>1.2% very serious risk
Lime (6%)	Kaolinite	-	Gypsum	1%
Lime (6%) + slag	Kaolinite	Kaolinite, micas	Gypsum	1%
Lime (1%) + hydraulic road binder (8%)	Marne (A2)	-	Pyrite and gypsum	<0.1% suitable0.1% to 1% doubtful>1% unsuitable

1.9.7. Strategies to Prevent Or Alleviate The Problem

Preventing the formation of ettringite or problematic minerals involves disrupting the availability of elements crucial to their development, specifically: calcium, alumina, water, and sulfates. Stabilizers rich in calcium, such as lime, cement, and fly ash, are highly advantageous for soil stabilization. However, it is evident that in soil treatments involving sulfates, the stabilizer supplying the most calcium will result in increased ettringite formation. Since lime has a higher calcium content compared to cement and fly ash, the expectation is that more ettringite would form when stabilizing sulfate-containing soil with lime. Possibly, combinations of pozzolans with Portland cement or lime can be utilized to counteract ettringite formation, similar to approaches used in cement concretes. Pozzolans encompass materials like silica fume, fly ash, slag, and others [163].

Moreover, in soil stabilization amidst sulfates, the optimal strategy for stabilizing clayey soil with lime would involve prompting the formation of harmful minerals before compaction. If these minerals emerge during the loosening phase, prior to placement and compaction, no harm will occur. This can be achieved by establishing an appropriate retention time (the duration between the application of the stabilizer and the compaction of the stabilized soil). To attain maximum density during compaction, an adequate amount of water (3 to 5 points above the optimum) is essential. Excessive water application should occur during the loosening phase.

1.10. Carbonation of Earth Mortar

1.10.1. Importance of Carbonation in Construction

Carbonation is a critical chemical process that significantly influences the hardening and durability of earth mortars. This process involves the reaction of carbon dioxide (CO_2) from the atmosphere with the components of the mortar, leading to the formation of stable carbonate compounds such as calcium carbonate ($CaCO_3$). Carbonation enhances the physical and mechanical properties of earth mortars, making them more durable and resistant to environmental degradation. Understanding the carbonation process is essential for optimizing the performance of earth mortars in construction, ensuring they meet modern standards of strength and longevity while maintaining their sustainable characteristics [164].

1.10.2. Chemical Process of Carbonation

1.10.2.1. Definition and Explanation of Carbonation

Carbonation refers to the chemical reaction between carbon dioxide (CO_2) in the atmosphere and calcium hydroxide ($Ca(OH)_2$) present in the mortar, resulting in the formation of calcium carbonate ($CaCO_3$). This reaction is crucial in the setting and hardening process of earth mortars. As CO_2 diffuses into the mortar, it reacts with the calcium hydroxide to form calcium carbonate and water. This transformation not only increases the strength of the mortar but also reduces its solubility, making it more resistant to weathering and other environmental factors.

1.10.2.2. Chemical Reactions Involved

The primary chemical reaction involved in the carbonation process can be represented as (eq.16):

$$Ca(OH)_2+CO_2 \rightarrow CaCO_3+H_2O \tag{16}$$

In this reaction, calcium hydroxide, a key component of the mortar, reacts with carbon dioxide from the air to form calcium carbonate and water. This process gradually converts the free lime into stable calcium carbonate, which enhances the mortar's durability. The rate and extent of carbonation depend on factors such as the concentration of CO_2, the availability of calcium hydroxide, and environmental conditions like humidity and temperature.

1.10.2.3. Role of Carbon Dioxide

Carbon dioxide plays a pivotal role in the carbonation process. It diffuses through the pores of the mortar and reacts with calcium hydroxide to form calcium carbonate. The availability of CO_2 in the atmosphere and its diffusion into the mortar are critical for the carbonation process. The rate of diffusion and subsequent reaction are influenced by the mortar's porosity, permeability, and environmental conditions. Effective carbonation requires a balance of CO_2 concentration and moisture content within the mortar, as both too much and too little water can impede the process.

1.10.3. Factors Affecting Carbonation

1.10.3.1. Environmental Conditions (Humidity, Temperature)

Environmental conditions such as humidity and temperature significantly influence the carbonation process. Higher humidity levels facilitate the dissolution of CO_2 in the pore water of the mortar, enhancing the carbonation rate. However, excessive moisture can saturate the pores, hindering CO_2 diffusion. Temperature affects the reaction kinetics, with moderate temperatures being optimal for carbonation. Extremely high or low temperatures can slow down the reaction rates, affecting the overall efficiency of the carbonation process.

1.10.3.2. Composition of Earth Mortar

The composition of earth mortars, particularly the proportions of clay, sand, and silt, affects their carbonation potential. High clay content can hinder CO_2 diffusion due to the dense packing of clay particles, while appropriate sand and silt ratios improve the permeability and facilitate the carbonation process. The presence of organic matter and other impurities can also impact the carbonation rate and the quality of the resulting calcium carbonate.

1.10.3.3. Porosity and Permeability

The porosity and permeability of earth mortars are crucial factors in the carbonation process. Porous mortars allow better penetration of CO_2, leading to more efficient carbonation. The permeability of the mortar determines how easily CO_2 can diffuse into the interior of the material. Mortars with high permeability facilitate rapid carbonation, while those with low permeability may experience slower carbonation rates, potentially affecting their long-term durability.

1.10.3.4. Curing Conditions

Proper curing conditions are essential for optimal carbonation. Adequate moisture and CO_2 exposure during the curing process enhance the carbonation rate and improve the properties of the mortar. Controlled curing environments that balance humidity and CO_2 levels can significantly enhance the carbonation process, leading to mortars with improved mechanical strength and durability.

1.10.4. Role of Carbonation in Durability

1.10.4.1. Enhancement of Mechanical Properties

Carbonation improves the mechanical properties of earth mortars by forming stable carbonate compounds that increase the material's strength and hardness. The conversion of calcium hydroxide to calcium carbonate reduces the solubility and enhances the durability of the mortar. This process also fills the pores and microcracks within the mortar, leading to a denser and more cohesive material structure.

1.10.4.2. Impact on Compressive Strength

The formation of calcium carbonate during carbonation increases the compressive strength of the mortar. This makes the mortar more robust and capable of withstanding greater loads, enhancing its suitability for structural applications. The improved compressive strength also contributes to the overall longevity and resilience of the construction.

1.10.4.3. Resistance to Weathering

Carbonated mortars exhibit enhanced resistance to weathering and environmental degradation. The stable carbonate compounds formed during carbonation protect the mortar from erosion, freeze-thaw cycles, and other weather-related damage. This increased resistance to weathering extends the lifespan of the mortar and reduces the need for frequent maintenance and repairs.

1.10.5. Significance in Construction

1.10.5.1. Benefits of Carbonation in Sustainable Building Practices

Carbonation contributes to the sustainability of earth mortars by enhancing their durability and reducing the need for frequent repairs. This process aligns with eco-friendly construction practices by utilizing natural materials and reducing CO_2 emissions compared to cement-based mortars. The use of carbonated earth mortars supports the development of low-carbon, sustainable building solutions that minimize environmental impact while maintaining high performance.

1.10.5.2. Comparison with Other Building Materials

Earth mortars, when properly carbonated, can compete with conventional building materials such as cement-based mortars and concrete. They offer a low-carbon alternative with similar or superior performance characteristics, including strength, durability, and resistance to weathering. The use of earth mortars can significantly reduce the environmental footprint of construction projects, making them an attractive option for sustainable building practices.

1.10.5.3. Future Perspectives

Research into optimizing the carbonation process and understanding its long-term effects on earth mortars will further enhance their viability as sustainable building materials. Advances in material science and construction techniques will likely expand the applications of carbonated earth mortars. Future studies should focus on developing new formulations of earth mortars, improving the efficiency of the carbonation process, and investigating the long-term performance of carbonated mortars in various environmental conditions.

1.11. Conclusion

This chapter has provided a comprehensive overview of earth construction, highlighting its historical significance, global applications, and the scientific principles governing its performance. From ancient building traditions to contemporary innovations, earth construction stands as a testament to the versatility and sustainability of natural materials. The exploration of various earthen construction techniques, such as adobe, rammed earth, cob, wattle and daub, and compressed earth bricks, reveals the adaptability of earth to diverse climates and construction needs.

A detailed examination of soil composition, chemical properties, and the role of stabilizers underscores the importance of understanding the material science behind earth construction. The chapter also addresses the critical aspects of durability, focusing on the mechanisms of deterioration and strategies to mitigate them, including the impact of sulfuric attacks and the complexities of ettringite formation. Furthermore, the discussion on carbonation highlights its role in enhancing the mechanical properties and durability of earth mortar, emphasizing its significance in sustainable building practices.

This chapter serves as a foundation for understanding earth construction, providing essential knowledge for researchers, practitioners, and anyone interested in sustainable and resilient building solutions. By integrating historical insights with scientific understanding, the chapter paves the way for further exploration and innovation in the field of earth construction.

CHAPTER 2
MATERIALS AND EXPERIMENTAL METHODS

2. CHAPTER 2: MATERIALS AND EXPERIMENTAL METHODS

2.1. Introduction

This chapter details the materials employed and the experimental methodologies implemented to investigate the properties and performance of earth mortar. A comprehensive characterization of the raw materials, including soil, water, lime, and Date Palm Ash (DPA), is presented, encompassing physical, chemical, and mineralogical analyses. The methodologies for preparing component proportions and assessing both physical and mechanical properties are thoroughly described. These include water-accessible porosity, linear shrinkage, dry bulk density, ultrasonic pulse velocity, abrasion resistance, compressive strength (dry and wet), and flexural strength testing [165, 166].

Furthermore, the chapter outlines the durability tests conducted to evaluate the long-term performance of the materials under various environmental conditions. These tests encompass total absorption, capillary absorption, swelling, wetting and drying cycles, chemical durability, and accelerated carbonation. The combination of material characterization and experimental testing provides a robust framework for analyzing the behavior and suitability of the investigated materials for sustainable construction applications.

2.2. Materials

2.2.1. Soil

The soil used in our research study was meticulously collected from the Biskra region, a remarkable area in the southeastern part of Algeria known for its unique soil characteristics. This valuable soil sample has also been subjected to several rigorous laboratory tests and analyses to ensure its quality and suitability for our ongoing experiments. The soil quality is of the utmost importance, as it dramatically impacts the outcomes of our research endeavors.

2.2.1.1. Grain size analysis and sedimentometry (NF P 18-560 and NF P 94-057)

Through this test, we can gain insights into the intricate granular composition of the soil under careful study, as clearly illustrated in Fig.2. 1. This detailed analysis facilitates a deeper understanding of the soil's characteristics and variations.

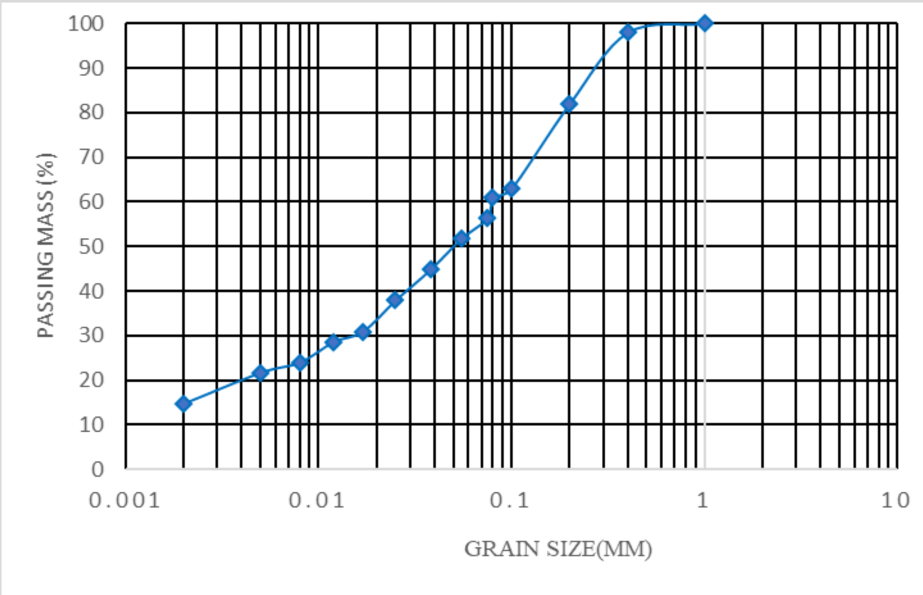


Fig.2. 1. The distribution of grain sizes within the test soil sample.

2.2.1.2. A comprehensive overview of physical characteristics and Atterberg Limits.

The results obtained from the soil analysis indicate that the Atterberg limits indeed fall well within the permissible and acceptable limits set forth by industry standards. This is a positive finding as it suggests the soil has suitable properties for various engineering applications. Additionally, Table.2. 1 presents a comprehensive overview of the detailed physical characteristics of the soil along with the specific Atterberg Limits, which are crucial for understanding the behavior of the soil under different conditions.

Table.2. 1 Physical Properties and Atterberg Limits.

Liquid Limit (LL)	Plasticity Limit (PL)	Plasticity Index (PI)	Absolute density (kg.m^{-3})	Bulk density (kg.m^{-3})
21	16	5	2395	1260

2.2.1.3. Analysis of Chemical Composition and Mineral Structure

Chemical and mineral composition analyses were performed meticulously in the Research Center and Technology Services Industry Building Materials CETIM, which is situated in Boumerdès, Algeria. The chemical composition of the soil samples was comprehensively obtained through advanced X-ray fluorescence techniques, as meticulously demonstrated in Table.2. 2. Meanwhile, the mineral composition of the soil was skillfully identified through X-ray diffraction methods, as clearly indicated in Table.2. 3. This comprehensive analysis provided valuable and insightful information into the characteristics and properties of the soil samples collected from the region, enhancing our understanding of its unique geochemical profile.

Table.2. 2 Soil chemical composition

Component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	TiO_2	LOI
Content	36.84	4.47	2.22	27.78	1.86	0.14	0.72	0.19	0.11	0.29	25.39

Table.2. 3 Composition of minerals in the soil

Minerals	Quartz	Calcite	Dolomite	Illite
Mineralogical composition (%)	45	39	8	7

2.2.2. Water

The water utilized in this study is specifically the drinking water, which is carefully maintained at a temperature of 20 ± 2 °C. Its quality satisfactorily meets and adheres to the requirements and standards specified in the NFP 18-404 standard.

2.2.3. Lime

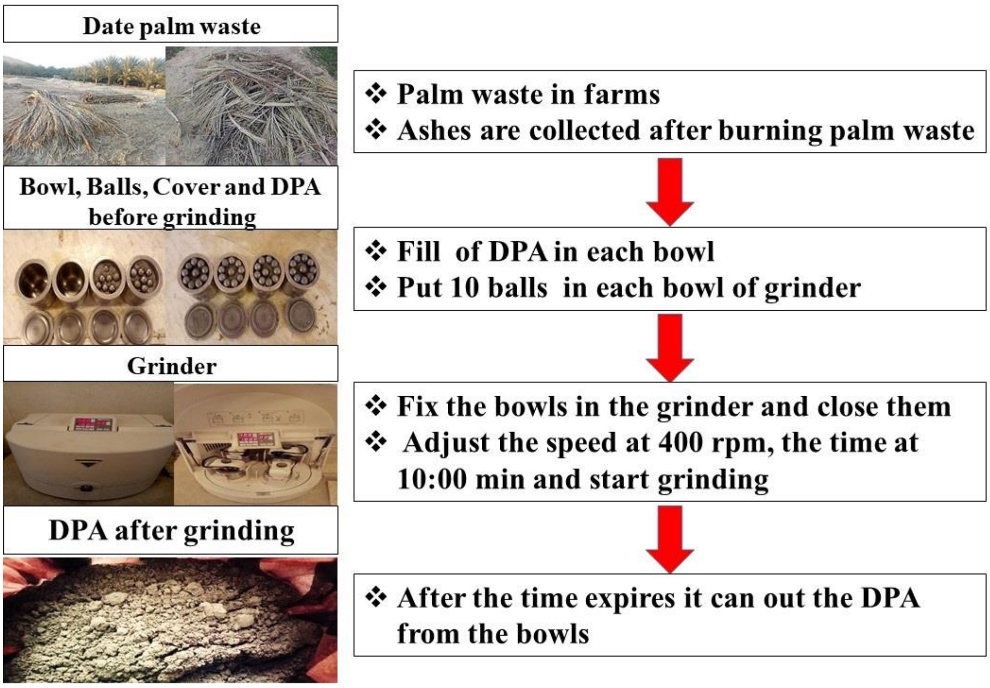
The binder used to prepare the various intricate mixtures is a specific type of quicklime produced at the dedicated lime unit in Saida, Algeria. Table.2. 4 effectively displays and exemplifies the precise chemical composition of this quicklime, allowing for a better understanding of its properties and applications.

Table.2. 4 Chemical composition of lime

Component	CaO	Al_2O_3	Fe_2O_3	MgO	SiO_2	K_2O	SO_3	Na_2O
Content	82.77	10.63	3.27	1.88	1.35	0.15	0.11	0.06

2.2.4. Date Palm Ash (DPA)

In Biskra, we collected and utilized the vegetal ash from palm waste. The dedicated palm farmers in this area meticulously gather all the parts of the palm tree that have reached their end of life, which includes various components such as the bunches, fibrillium, leaflets, petiole, pedicels, rachis, spathe, thorns, and trunks. After gathering these materials, they proceed to burn them to produce ash. This ash is then carefully collected. The preparation of the ash involves grinding it down using a traditional line grinder, as shown in Fig.2.2. This method ensures that the ash is finely processed and ready for its intended applications.



❖ **Physical properties of DPA**

Blaine fineness (cm²/g)	**4179**
Specific gravity (g/cm³)	**2.4**

❖ **The main mineralogical components by using X- ray Powder Diffractometer of DPA shown in Fig.3.3**

❖ **SEM analysis of DPA shown in Fig.3.4**

❖ **Energy-dispersive x-ray spectroscopy (EDX) of DPA shown in Fig.3. 5**

Fig.2.2. Methodology of DPA setup

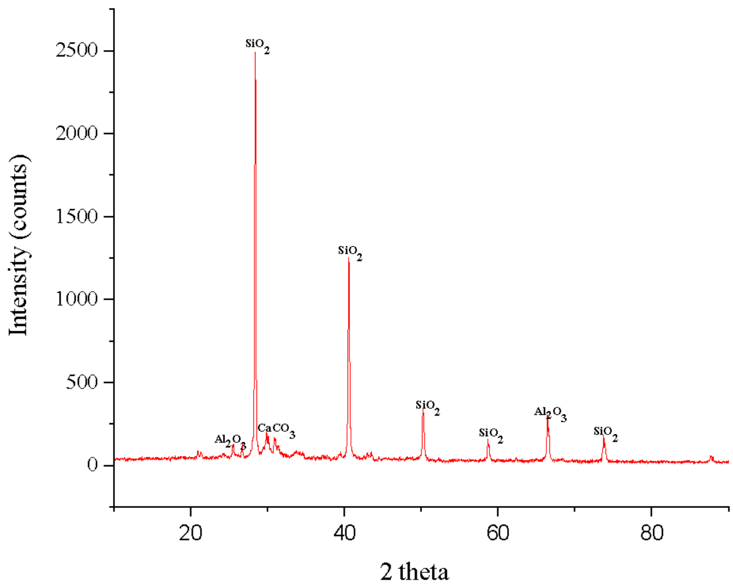


Fig.2.3. X-ray diffraction pattern of DPA.

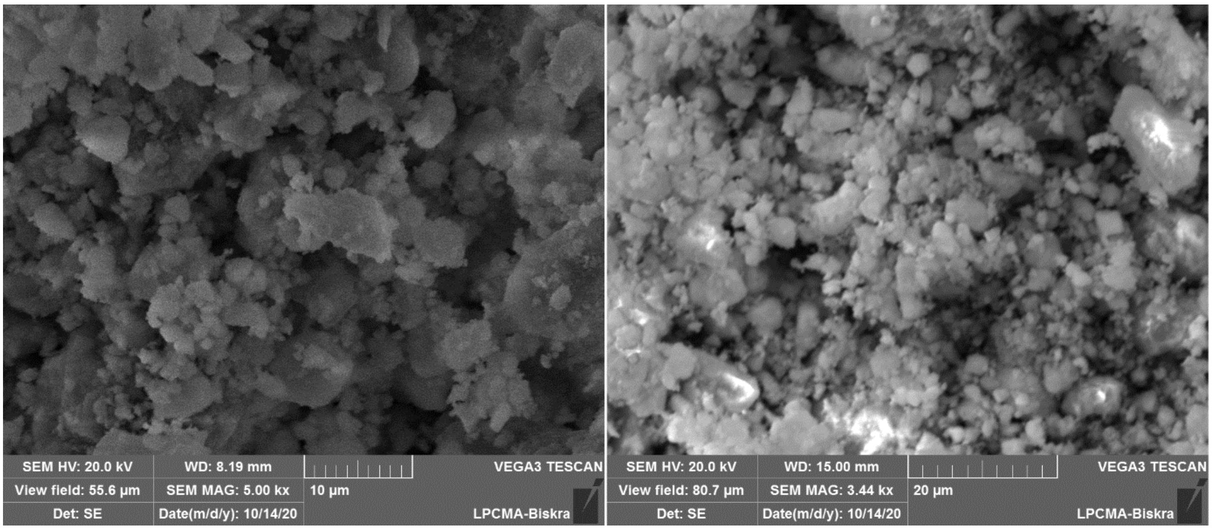


Fig.2.4. Scanning electron microscopy of DPA

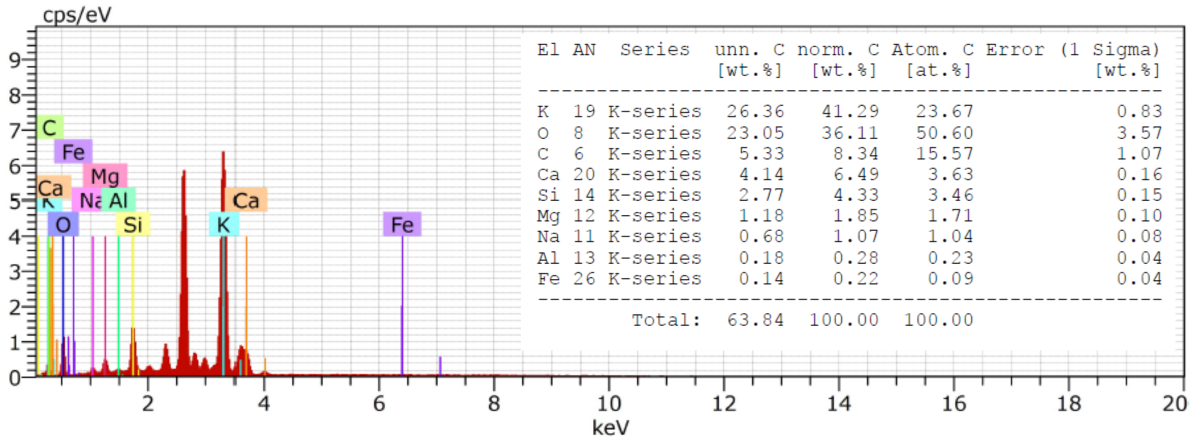


Fig.2. 5. Energy-dispersive x-ray spectroscopy (EDX) of DPA

2.3. Methods

2.3.1. Prepare Proportions of Components

Following the established French standard EN 1015-11, earth mortar samples (EMS) are carefully manufactured in custom moulds that measure 4x4x16 cm^3 to perform all tests comprehensively and accurately. The EMS utilized in these tests were prepared after identifying the optimal amounts of water and stabilizer content in the mixture, which was achieved through assessments of dry compressive strength. According to Hilt and Davidson [167], an ideal point exists concerning the dose of lime for effective soil stabilization that corresponds precisely to the plasticity limit. The specific proportions of the experimental mixtures are thoroughly detailed in Table.2. 5 and illustrated in Fig.2. 6.

The addition of ash was carefully undertaken in varying proportions of 2%, 4%, 6%, 8%, and 10%, and the effects of these additions on the overall mixture were closely studied for their impact on performance. When initiating the mixing process, the global dry weight of the mixture must be consistently maintained across all samples throughout the various stages of the study to ensure comparability and accuracy in results. The soil must be dried in an oven for a precise duration of 24 hours at a temperature of 65± 2 °C, which is vital to ensure that the mixture reaches an optimal state of dryness before experimentation.

Initially, we began by thoroughly mixing the dry soil mixture for a precise duration of two minutes. The binder and additives, including the lime and ash, were added, and this mixture was blended for an additional minute. Then, Water was gradually introduced into the mixture and blended for two minutes to achieve a homogeneous consistency. In the next step, we filled each mould up to the midpoint and then utilized a machine to shake the moulds with 15 strokes, ensuring that the material was compacted effectively. After the first portion was completed, This shaking process was repeated for the second half of the moulds.

After thoroughly mixing and moulding, we covered the moulds with a protective plastic film to prevent moisture loss. We placed them in an oven set to a consistent temperature of 65± 2 °C for 12 hours, which was meant to accelerate the drying process significantly. Following this period, we carefully removed the samples from the moulds and placed them in a tightly closed plastic sack. They were then returned to the oven for another 7 days at the same temperature of 65± 2 °C. After this prolonged drying phase, we extracted the samples from the plastic sack and re-entered them into the oven until they were scorched and achieved a steady weight. Finally, we subjected the samples to rigorous testing, ensuring accuracy by averaging the results from three samples for a reliable outcome.

Table.2. 5 Proportions of experimental mixtures

Soil %	Lime %	Water %	DPA %
100	10	24	0 2 4 6 8 10
		26	
		28	
	12	26	
		28	
		30	
	14	28	
		30	
		32	
	16	30	
		32	
		34	

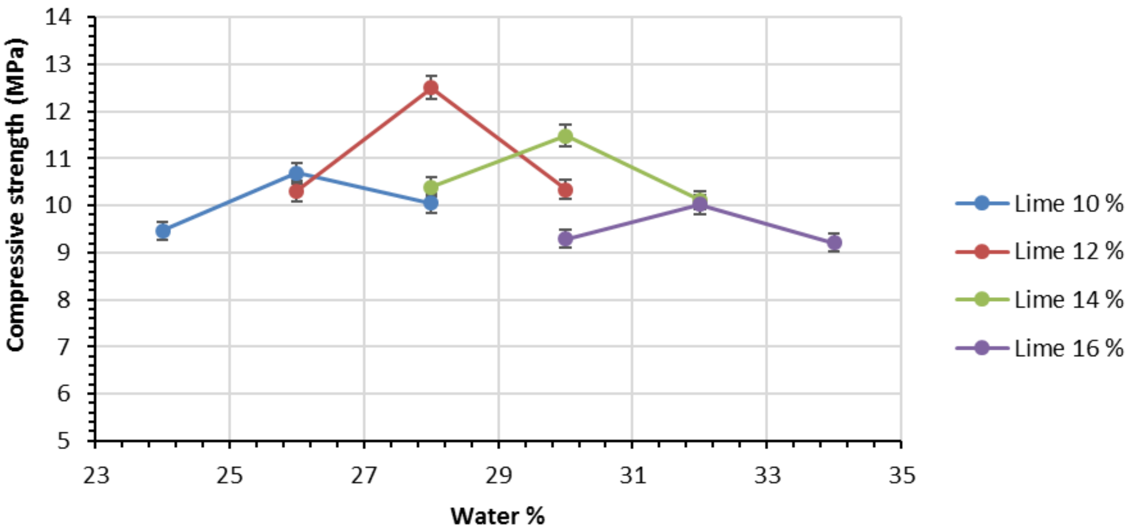


Fig.2. 6. Optimization of lime and water based on dry compressive strength.

2.3.2. Examination of Physical Properties

2.3.2.1. Water-accessible porosity evaluation

Porosity is a significant measure of the void spaces present in a given sample, and it is widely regarded as a key indicator of the material's overall durability. Given its crucial importance, the thorough evaluation of this property becomes essential in the scope of this comprehensive study. The specific test procedure was meticulously conducted in accordance with the established standard "ASTM C642-06" (2006). To accurately calculate the porosity of the sample, the following weights are utilized:

- Dry mass by oven (M1): dry the test specimen at a temperature between 100 to 110 °C, to ensure having a constant mass.
- Saturated mass after immersion (M2): Immerse the test specimen in water at 21 ° C after final drying for at least 48 hours. Dry on the surface by removing moisture from the surface with a towel and identify the mass.
- Saturated mass after boiling (M3): Place the test specimen in a suitable container covered by water and boil for 5 hours. Leave the mass to cool for at least 14 hours. to reach a final temperature of 20 to 25 ° C. Then wipe the surface with a towel and identify the mass.

- Apparent mass immersed (M4): Suspend the sample, after immersion and boiling, by a wire and determine the apparent mass in water by hydrostatic weighing.

The porosity of water (P) can be determined precisely through the equation provided below:

$$P = \frac{M_3 - M_1}{M_3 - M_4} \times 100 \quad (17)$$

2.3.2.2. Conducting a test for linear shrinkage evaluation

Shrinkage measurements were conducted meticulously by the "NF P15-433" standard to ensure accuracy and reliability. To achieve precise results, we meticulously prepared our samples using custom-designed moulds crafted to meet specific requirements. We strategically positioned the studs along the longitudinal faces of each sample to optimize the measurement process and ensure consistency across all tests. The method of measuring shrinkage was carried out periodically, utilizing a sophisticated digital shrinkage apparatus, which allowed for real-time data collection. This continued until we obtained consistent, steady readings that accurately reflected the shrinkage values.

2.3.2.3. Test of dry bulk density

The dry bulk density of EMS was meticulously calculated through the precise utilization of the "EN 1015-10/ A1" standard, which has been specifically applied based on the following detailed and specific relationship:

$$\rho = \frac{M}{V} \quad (18)$$

Where (M) represents the total dry mass that is precisely measured in kilograms (kg) and (V) indicates the volume, which is expressed in cubic meters (m^3).

2.3.2.4. Test of ultrasonic pulse velocity

The TICO Ultrasonic Testing Instrument was utilized with great care and attention to detail in strict accordance with the well-established and recognized "NF EN 12 504-4" standard.

This specific and sophisticated testing method allows us to ascertain the effectiveness and the overall impact of incorporating DPA on the speed of wave transmission while also analyzing the overall duration or time spent by the wave as it travels through the material. The comprehensive testing procedure involves placing two poles on either side of the sample under examination to ensure precise measurements. Before this step, we ensure that the sample is greased effectively to prevent air infiltration. Any air pockets that might form would obstruct accurate readings, which could jeopardize the integrity of the entire testing process. This careful and methodical preparation enables us to obtain precise measurements of both the speed of the wave pulse and the transmission time, ensuring that we achieve reliable and valid results.

2.3.2.5. Test of abrasion resistance

This test is conducted per the XPP 13-901 standard. During the test, the samples are subjected to mechanical corrosion using a wire brush that is no more than 25 mm wide.

First, placing a mass weighing exactly 3 kg on top of the brush is essential. After securing the mass, we will move the brush steadily across the surface of the samples using a consistent back-and-forth motion. This motion should be continuous, with one complete pass every second for one full minute.

Once this comprehensive process is completed, we can proceed to calculate the abrasion coefficient using the specific relationship outlined in the standard:

$$A_R = \frac{S}{M_1 - M_2} \tag{19}$$

Where (S) represents the brushed surface area in cm², and (m1 and m2) refer to the masses of the sample before and after corrosion, respectively, measured in grams.

2.3.3. Mechanical properties

2.3.3.1. Testing of Dry and Wet Compressive Strength

The dry compressive strength test is an essential and critical procedure used to identify, evaluate, and assess the dry resistance of various material specimens. It is very important to note that, in general, wet specimens typically exhibit lower mechanical properties when compared to their dry counterparts. This significant difference between wet and dry states highlights why it can be particularly useful and highly informative to conduct tests on specimens that are in a wet state. By doing so, we can thoroughly determine their minimum characteristics and performance levels under the most unfavourable and challenging conditions. This is crucial for understanding how these materials will actually perform in real-world scenarios where moisture levels can significantly impact their structural integrity and durability. This specific approach is in accordance with the established standard NF EN 196-1, which meticulously outlines the necessary procedures, protocols, and guidelines for ensuring accurate, consistent, and reliable testing results that are essential for proper evaluation.

2.3.3.2. Test flexural strength

The three-point bending test is methodically and rigorously performed on a sample sized 40mm × 40mm × 160mm, per the widely established standard "NF EN 196-1." During this comprehensive testing process, the flexural strength Rf and the elastic modulus (E) are meticulously calculated based on specific and well-defined relationships that govern material behavior under various forms of stress. These calculations are essential for thoroughly understanding how the material will perform in real-world applications, ensuring accurate predictions of its performance and reliability.

$$R_f = \frac{1.5 \ . \ P_f \ . \ L}{W^3} \quad (20)$$

$$E = \frac{P_f L^3}{48 I \delta} \quad (21)$$

Where (Pf) represents the peak load in N, (S) denotes the support span in mm, (W) indicates the specimen depth in mm, (δ) is the deflection caused by the load F, and (I) is the moment of inertia.

Fig.2. 7. Experimental setup for the flexural strength test.

2.3.3.3. The SEM–EDX analytical method

SEM–EDX analysis was meticulously conducted within the esteemed Laboratory of Physics of Thin Films and Applications (LPCMA), which can be found at the University of Biskra in Algeria. The main objective of this comprehensive and detailed analysis is to systematically identify and thoroughly characterize the distinctive features of the EMS. This includes not only assessing the strength of cohesion between various elements but also evaluating the presence and impact of the voids located within the material itself.

2.3.4. Durability tests

2.3.4.1. Test of total absorption

This test determines the samples' total absorption (TA) capacity. It was systematically conducted using two methods to ensure accurate and reliable results. According to the standards outlined in BS 3921 (1985), various methods can be used to carry out this test effectively. From these, two particular methods have been carefully selected for our purposes. The first method employed is the boiling test, which precisely measures the total absorption porosity of the materials being tested. In this particular procedure, the samples are initially placed in a water bath set at room temperature, and the water temperature is gradually increased until it reaches the boiling point within one hour. Once the boiling point is achieved, the samples are maintained at an elevated temperature of 100°C for 5 hours. Following this heating phase, a slow cooling process lasts approximately 16 to 19 hours until the samples return to room temperature. After this cooling phase, the wet samples are thoroughly weighed, and the total absorption capacity is subsequently calculated by applying the equation.

Additionally, the test was conducted using an alternative method to comprehensively assess the water resistance and overall durability of the samples being examined. In this alternative approach, the samples were immersed in a water bath for varying durations of 1, 2, 3, and 4 days. After their specified immersion times, the samples were carefully removed from the water, and their wet weights were measured accurately. Subsequently, the percentage of water absorption was determined by calculating the relative difference between the weights of the dry samples and their corresponding wet weights, using the following formula for clarity and precision.

$$TA\ (\%) = \frac{(M_W - M_D)}{M_D} \times 100 \qquad (22)$$

Where (MW) represents the wet mass in grams and (MD) represents the dry mass in grams.

2.3.4.2. Test of capillary absorption

According to the “NF XP 13-901” standard, water absorption is measured through capillaries by partially submerging the sample to a specific depth of 5 mm. For consistency, it is important to ensure that the submerged portion maintains the same height across all the test samples. The water absorption coefficient, denoted as Ca, corresponds to the specific absorption rate measured after a precise duration of 10 minutes. This process adheres to the following formula, which is essential for calculating the absorption properties accurately:

$$Ca = \frac{100 \times W}{S\sqrt{t}} \qquad (23)$$

Where (W) represents the water mass in grams absorbed by the sample during the test, (S) denotes the surface area immersed in square centimeters, and (t) indicates the immersion time of the sample in minutes.

2.3.4.3. Swelling test

According to the “XP P 13-901” standard, this test is conducted using installing two screws into each sample. Initially, we take the samples and submerge them in a water bath for 96 hours, allowing them to absorb moisture. After this period, we remove them from the water and dry them thoroughly for approximately 10 minutes. Finally, we proceed to measure the distance between the two screws carefully and accurately, which will help us calculate the swelling of the samples utilizing the relationship described below:

$$\Delta l_s = \frac{l_1 - l_0}{l_0} \quad (mm/m) \tag{24}$$

Where: (l_1 and l_0) represent the distance between the two screws after and before immersion, respectively.

2.3.4.4. Drying and wetting test

This test aims to verify the sample's ability to withstand by exposing them to several harsh cycles and observing their effect according to the standard ASTM D 559-57. During this study, we applied 12 moisturizing and drying cycles, and in each cycle, we immersed the samples in water for 5 hours and then dried them in the oven at 71 ° C for 42 hours. After each cycle, we remove the damaged part in the samples. In the last cycle, we calculate the percentage loss of mass compared to the original mass of each sample.

2.3.4.5. Chemical durability tests

To evaluate the chemical durability of EMS, they were subjected to different chemical solutions with a concentration of 5% as an accelerated procedure: sodium sulfate (Na_2SO_4), sulfuric acid (H_2SO_4), and hydrochloric acid (HCl), all of which were kept at room temperature. This testing procedure was based on the method proposed by Bezerra et al. [168]. The initial step involved drying the EMS in an oven at a temperature of 110°C for 24 hours to remove any moisture content, after which their dry weights were recorded.

Following the drying process, the EMS were placed in plastic containers, elevated on support bars to ensure they did not touch the bottom. The prepared chemical solution was then carefully poured into the container until it reached a height of 2 cm from the bottom surface of the EMS. This setup was designed to ensure consistent exposure of the EMS to the chemicals. The chemical solution level was maintained at 2 cm by replacing it at regular intervals to ensure the EMS were continuously exposed to the solution. After 20 days of exposure, the EMS were removed from the solution and weighed again to record any changes in mass.

The containers were then emptied, and the EMS were left in an oven at a temperature of 110°C for 24 hours before being weighed once more. This process of soaking in the chemical solution followed by air drying constituted one cycle. In this study, the EMS underwent ten such cycles. The average mass variation for a minimum of three samples was calculated and reported.

After completing the wetting-drying cycles, the EMS underwent further testing to determine the extent of mass loss and changes in compressive strength, which provided a measure of their chemical durability. These tests allowed for the assessment of the EMS' resistance to chemical degradation and provided insights into their long-term performance when exposed to harsh chemical environments.

2.3.4.6. Accelerated Carbonation Tests

In the accelerated carbonation tests, the EMS are placed in a sealed chamber designed specifically for this purpose, as illustrated in Fig.2. 8. Inside this chamber, the gas mixture is composed of 50% carbon dioxide (CO2) and 50% air, a concentration that effectively simulates accelerated carbonation conditions [169]. To optimize the carbonation process, the relative humidity within the chamber is meticulously maintained between 60% and 70%. This is achieved by using a saturated sodium nitrite solution, which ensures that the environment remains within the ideal humidity range for carbonation. Research has shown that the carbonation rate is maximized within this humidity range, facilitating a more efficient testing process.

The temperature inside the chamber is kept constant at approximately 20 ± 2°C, which is close to room temperature and provides a stable environment for the test. To determine the depth of carbonation in the concrete samples, a specific method is employed. This involves splitting the EMS to expose a fresh fracture surface, which is then treated with an alcoholic phenolphthalein solution. This solution acts as a pH indicator, changing color to reveal the extent of carbonation. According to Nedeljković et al. [170], the phenolphthalein solution creates a distinct visual difference on the fracture surface:

- ✓ An internal pink zone indicates the non-carbonated area, where the concrete remains alkaline.
- ✓ An external colorless zone indicates the carbonated area, where the pH has dropped due to the reaction between CO2 and the concrete components.

After applying the phenolphthalein solution, the depth of the carbonation front is measured. This is done by taking measurements on both faces of the specimen at intervals of 2 cm. The measurements are taken after allowing approximately 15 minutes for the color change to fully develop. The average of these measurements provides an accurate representation of the carbonation depth. To monitor the progression of carbonation over time, these measurements are repeated for each specimen after 14, 28, and 42 days of exposure in the CO2 chamber. This detailed process ensures that the results are reliable and reflect the carbonation behavior of the EMS under accelerated conditions.

Fig.2. 8 Airtight enclosure (CO_2 chamber).

2.4. Conclusion

This chapter has provided a detailed account of the materials and experimental methods used to assess the properties and performance of earth-based construction materials. The comprehensive suite of tests, ranging from material characterization to mechanical and durability assessments, offers a robust framework for evaluating the suitability and long-term viability of the investigated mixtures. The data obtained through these methods will be presented and analyzed in subsequent chapters, providing insights into the effects of different material compositions and environmental factors on the performance of earth mortars.

By adhering to established standards and rigorous experimental protocols, this study aims to contribute reliable and reproducible findings to the field of sustainable construction materials. The methodologies outlined herein serve as a foundation for future research and development efforts aimed at optimizing the use of earth-based materials in the built environment.

CHAPTER 3
RESULTS AND DISCUSSION

3. Chapter 3: Results and discussion

3.1. Introduction

This chapter presents and discusses the results obtained from the experimental investigations outlined in Chapter 2. The primary focus is on elucidating the effects of Date Palm Ash (DPA) addition on the physical, mechanical, and durability properties of earth mortar. The results are systematically presented, beginning with the impact of DPA on physical properties such as water-accessible porosity, linear shrinkage, bulk density, ultrasonic pulse velocity, and abrasion resistance.

Subsequently, the chapter examines the influence of DPA on mechanical properties, including dry and wet compressive strength, flexural strength, and overall mechanical behavior. Finally, the effects of DPA on durability properties, such as total absorption, capillary absorption, swelling, wetting and drying resistance, chemical durability, and accelerated carbonation, are analyzed. The results are interpreted in the context of existing literature, providing insights into the underlying mechanisms governing the observed behavior and highlighting the potential benefits and limitations of DPA as an additive in earth-based construction materials.

3.2. DPA addition effect on physical properties

3.2.1. Porosity accessible to water

The results illustrated in Fig.3. 1 unmistakably demonstrate the effectiveness of DPA addition on the porosity values accessible to water within the material structure. It is crucial to emphasize that the porosity notably decreases with the incremental addition of DPA at varying concentrations, precisely at 2%, 4%, and 6%. Our observations noted that the absolute lowest porosity value was achieved at a DPA concentration of 6%, resulting in a significant porosity measure of 28.38%. This specific measurement corresponds to the most considerable porosity reduction recorded, reflecting an impressive decline of 8.4% compared to the porosity observed in the reference specimen that was not treated with DPA. This important and significant result is thoroughly illustrated in Fig.3. 8, where we compare the scanning electron microscope (SEM) images across various experimental conditions ranging from 0% to 10% DPA addition. Interestingly, however, it was observed that when the content of DPA surpasses 6%, there was a surprising uptick in porosity levels; however, this increase did not escalate to levels that could be classified as uncontrollable or excessively high. According to extensive research conducted by Röhlen and Ziegert [171], the porosity values typically observed for earth mortars tend to fall within a range of approximately 20 to 30%. All the results we obtained fit within this specified range comfortably, thus confirming that our findings are consistent with the established norms within this field of study. Additionally, other scholarly studies conducted by researchers such as Santos et al. [172] and Ouedraogo et al. [173] have produced comparable results in their investigations of earth mortars and adobes, further solidifying and validating the trends observed in our systematic experimental results.

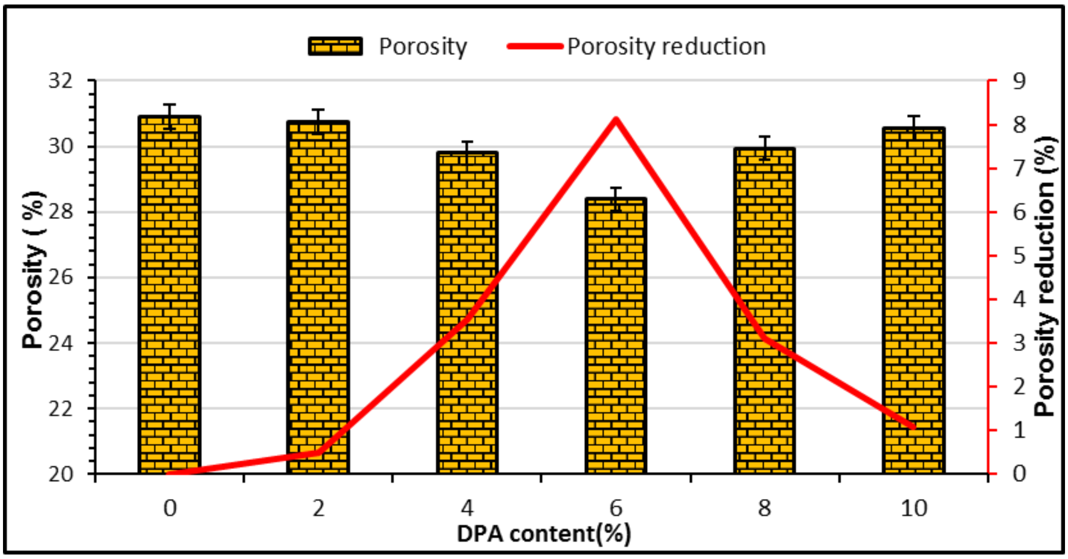


Fig.3. 1 Porosity of EMS as a function of DPA content

3.2.2. Linear shrinkage

Fig.3. 2 illustrates the notable outcomes of the linear shrinkage phenomenon observed in the earth mortar specimens, particularly when various percentages of DPA are incorporated into the mixtures. Among the samples meticulously tested and analyzed, the most minimal linear shrinkage was identified, where a DPA content of 6% was utilized, resulting in a shrinkage value of just 2.41%. This figure indicates a remarkable reduction in linear shrinkage of approximately 40%, particularly when compared to the mortar sample that contained no DPA, which exhibited a shrinkage value of 4.06%. The recorded linear shrinkages for the remaining cases that featured DPA content levels of 2%, 4%, 8%, and 10% were 3.73%, 2.77%,

3.14%, and 3.98%, respectively. While it is evident that there were notable variations in the shrinkage results displayed across different samples, it is particularly noteworthy that after the testing period, none of the samples exhibited any visible cracks, which is illustrated in Fig.3. 3. This complete absence of cracks can be attributed to the gradual yet effective lime reactions that take place within the mixtures during the curing process. The extensive research conducted by Meimaroglou and Mouzakis [174] highlights a significant finding; in their comprehensive study, which examined 49 unique types of various earth mortars, it was determined that the linear shrinkage of these materials does not surpass 2.5% when adhering to the stringent standards set by DIN 18946 a benchmark that could be considered quite rigorous in the field. They further assert that a linear shrinkage limit of 5% for earthen structures is considerably more achievable and realistic. In light of the compelling information presented, it becomes evident that the results obtained from our experiments align remarkably well with the ranges described by Meimaroglou and Mouzakis [174]. The linear shrinkage ratios observed in our study span between 2.4% and 4%, indicating that our findings are consistent with the established norms and expectations in earth mortars.

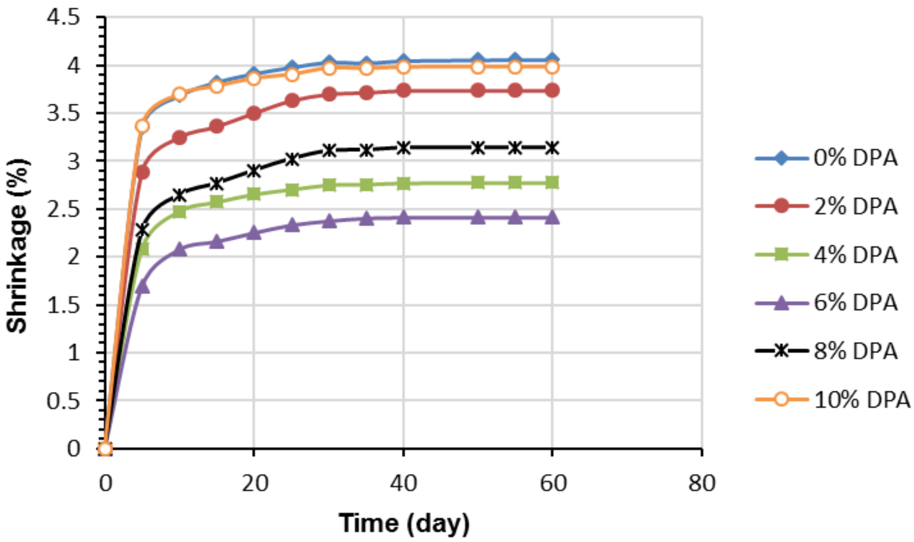


Fig.3. 2 Linear shrinkage of EMS as a function of DPA content.

Fig.3. 3 EMS after the end of the linear shrinkage test.

3.2.3. Bulk density

The bulk density of the material in question was thoroughly evaluated by calculating the precise ratio of its mass to the geometric volume of EMS, as graphically illustrated in Fig.3. 4. Upon a meticulous review of the results we obtained, it became increasingly clear that the values we measured span a range from 1505 to 1612 kg/m^3. Furthermore, our comprehensive investigation revealed that increasing the DPA content to 6% resulted in a noteworthy rise in dry bulk density. The strategic introduction of DPA substantially contributed to the formation of new hydration products, which in turn led to a significant reduction in the overall porosity of the material, as depicted in Fig.3. 8. This evident reduction in porosity provides a clear and logical explanation for the observed increase in density we documented. Based on extensive research conducted by Röhlen and Ziegert [171], we note that the dry bulk density of earth mortar is typically found to vary between the established range of 1400 and 1800 kg/m^3. Within this particular context, our findings indicate that the results obtained align with these established and recognized values. Additionally, the work done by Tânia Santos et al. [175] also reported similar findings, noting that the material density falls within these same limits, reinforcing our results' overall validity and credibility.

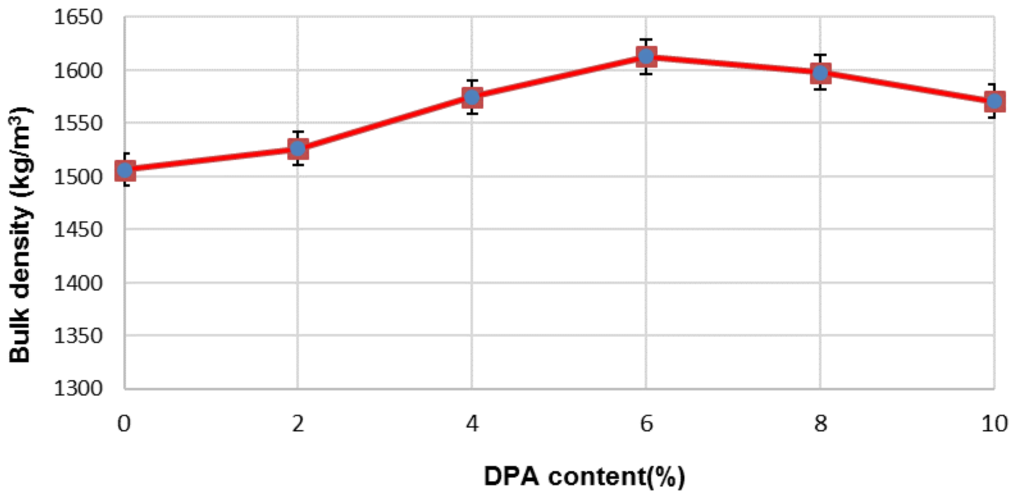


Fig.3. 4 Dry bulk density of EMS as a function of DPA content.

3.2.4. Ultrasonic pulse velocity

This test is a widely recognized method commonly employed to assess the consistency and quality of the mortar while also evaluating the presence of various imperfections, such as voids, cracks, or other defects that may negatively affect the structural integrity of the material being analyzed [176, 177]. The findings derived from the experimental data reveal that the longer the wave pulse was transmitted through the mortar sample, the more it indicated the existence of various defects. This tendency leads to a scenario where higher porosity and reduced density are observed, or perhaps a more heterogeneous structure is present overall. From the data presented in Fig.3. 5, it became abundantly evident that the wave propagation speed tends to increase significantly when the DPA content is raised to a level of 6%. Specifically, the optimum concentration of 6% DPA displayed an impressive increase of about 30% in propagation speed compared to a sample containing 0% DPA, showcasing the effects of the additive. Furthermore, incorporating DPA within the range of 2-6% notably reduced the presence of voids within the mortar matrix and contributed to an impressive increase in density. These factors played a crucial role in enhancing the speed at which the ultrasound pulse travels through the mortar sample. Conversely, it was observed that when the DPA content exceeded the level of 6%, this led to excessive quantities of ash, insufficient chemical reactions in the mix, and a matrix that was less resistant overall. As a result, this excessive content significantly diminished wave propagation speed within the mortar sample. Moreover, it is worth noting that

Prasanna et al. [178] identified that the ultrasonic pulse velocity could be remarkably improved in concrete mixtures, including ferrochrome ash and lime. This finding effectively showcases the potential benefits of certain additives in enhancing the properties and performance of concrete.

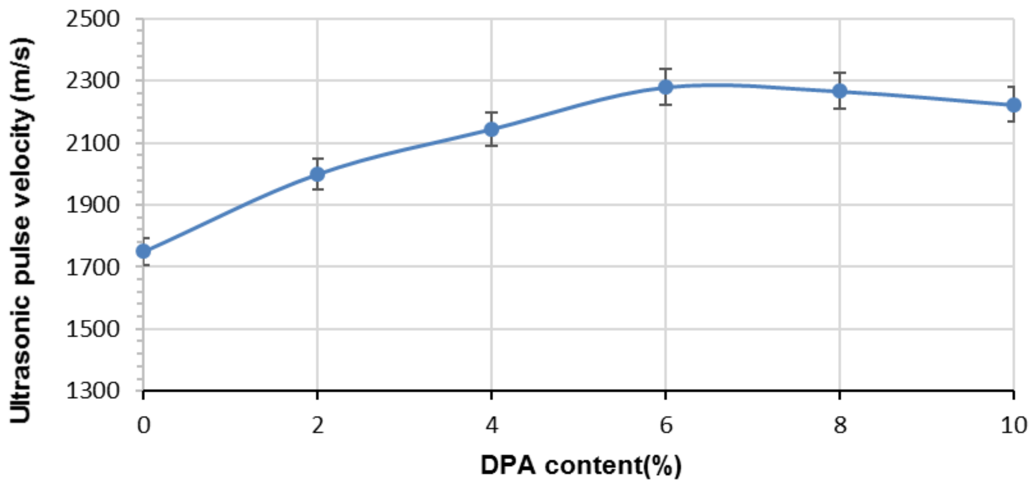


Fig.3. 5 Ultrasonic pulse velocity of EMS as a function of DPA content.

3.2.5. Abrasion resistance

Fig.3. 6 presents the detailed findings regarding the abrasion resistance of various thoroughly tested samples. This test is designed to simulate real-world conditions typically encountered in dry, arid regions, where different building materials are regularly exposed to significant abrasion caused by wind-borne sand particles' relentless and persistent influence. The results obtained from these rigorous tests illustrated a distinct and positive effect of DPA at concentrations of 2%, 4%, and 6%, with the resulting levels of abrasion resistance remarkably increasing to an impressive 166%, 235%, and a striking 471% respectively. However, it is noteworthy that when the DPA content was increased to 8% and 10%, a noticeable decrease in the abrasion resistance was observed, which raises important considerations for optimal usage. Additionally, it is worth mentioning that the strategic application of lime, combined with the inclusion of DPA into the samples, can significantly help reduce the existing gaps and greatly enhance the cohesion between the constituent particles of the mortar. Furthermore, the research conducted by Santos et al. [175] and Faria et al. [179] has involved several classifications of the abrasion resistance of earth mortars based on the well-established guidelines outlined by

DIN 18947 (DIN 2013). Through methodical and careful comparison, it becomes evident that the addition of DPA has led to a notable improvement in the overall performance and durability of the mortar, underscoring its effective role in enhancing the long-term integrity of building materials under harsh environmental conditions.

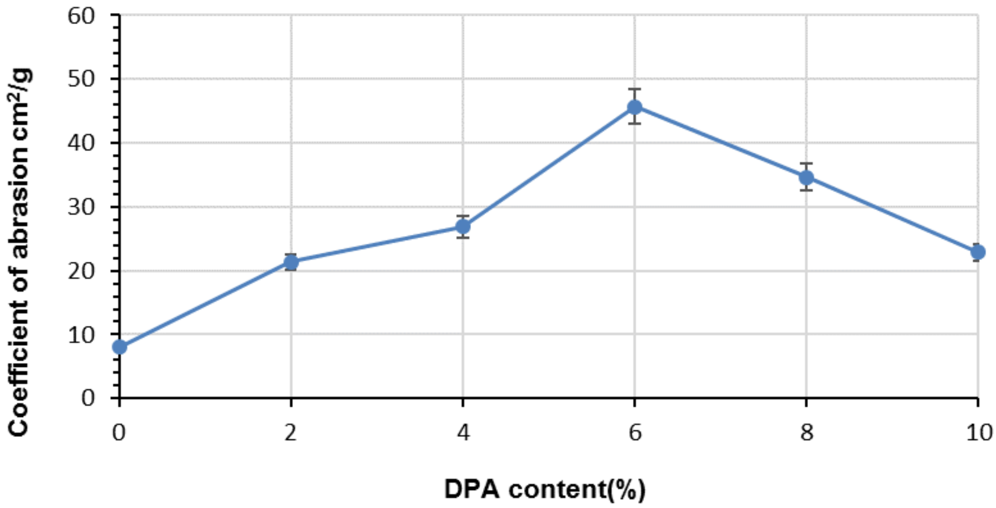


Fig.3. 6 Abrasion coefficient of EMS as a function of DPA content.

3.3. DPA addition effect on mechanical properties

3.3.1. Dry and wet compressive strength

It is of utmost importance and considerable significance to fully understand the compressive strength of earth mortar, as these strengths are some of the primary quality and performance indicators. This is particularly true when assessed in a wet state, which often reflects the more challenging and worst-case scenarios in real-world applications. Fig.3. 7 provides a clear and concise illustration showcasing the effectiveness of varying DPA content on both DCS (dry compressive strength) and WCS (wet compressive strength). Based on the comprehensive results from our detailed analysis, we observed a clear, notable, and substantial improvement in the strengths when a partial replacement of lime was executed, specifically utilizing different proportions of DPA. The strength consistently increases at 2%, 4%, and 6% DPA, while a decline is recorded at 8% and 10% higher percentages.

Furthermore, it is indicated that the optimum point or peak performance is achieved at the 6% mark, which serves as a crucial benchmark. In this particular case, the meticulously gathered measurements of DCS and WCS demonstrate an increase of 21.7% and 16.5%, respectively. These increases correspond to impressive resistances of 15.21 MPa and 9.27 MP, respectively, establishing significant milestones in enhancing strength. This notable increase can be reasonably explained through the formation of calcium silicate hydrate, which arises from the pozzolanic reactions between lime, soil mineral components, and the silica and alumina present in the DPA. This relationship is further demonstrated in Fig.2. 5, which showcases EDX images of the DPA [180, 181]. In addition to these crucial findings, it has been consistently shown that incorporating DPA contributes to a marked reduction in porosity. This reduction, in turn, leads to a corresponding increase in the overall resistance and stability of the mortar.

On the other hand, regarding the observed causes of deficiency in strength at higher DPA percentages, The issue with the deficiency stems from insufficient lime for the DPA to facilitate the pozzolanic reaction. This means that replacing more than 6% of the lime with DPA does not contribute to any increase in strength. This discrepancy implies that the partial replacement of lime with more than 6% DPA will likely result in no further strength gains [21, 182]. In Fig.3. 8, the SEM-EDX analysis that was performed on samples containing DPA at various levels of 0%, 6%, and 10% indicated that the Si/Ca content in the sample with 6% DPA was significantly more significant than the corresponding contents found in both the 0% and 10% samples. This indicates that as the Si/Ca content rises, the amount of formed calcium silicate hydrate (C-S-H) also increases proportionately, underscoring the importance of this balance . When comparing the ratio of the dry to the wet strengths, our findings reveal that these ratios do not exceed 2% at any point of measurement. This confirms that they consistently remain well within permissible limits [183].

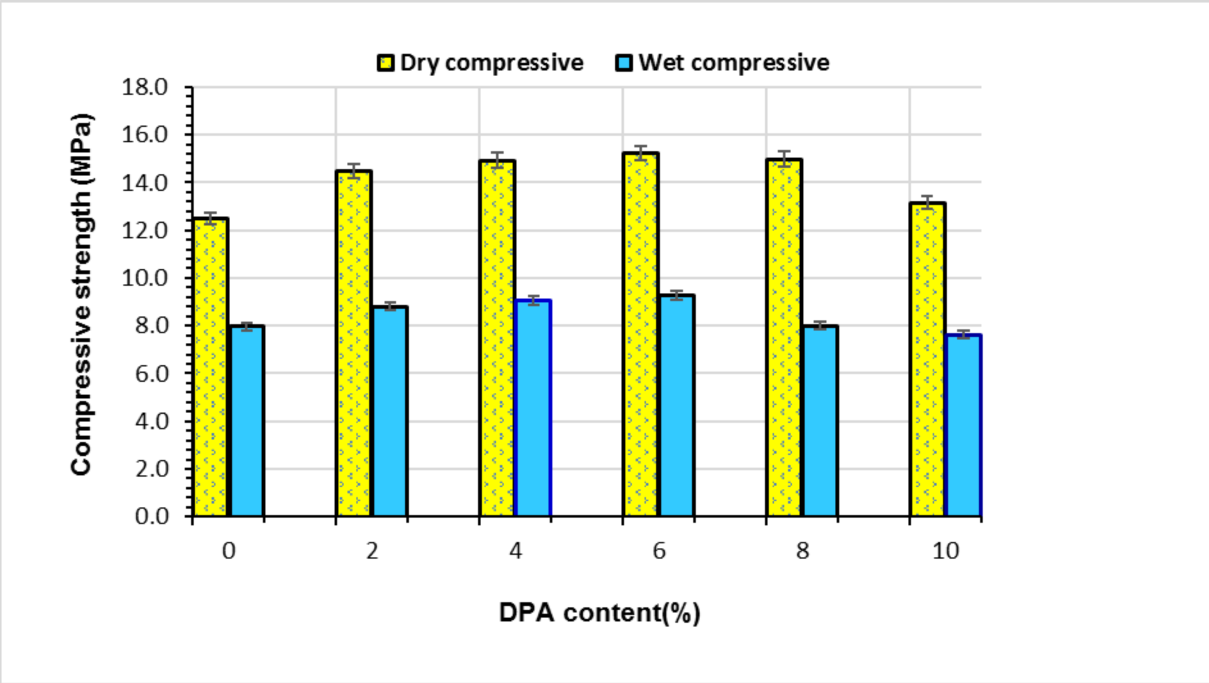
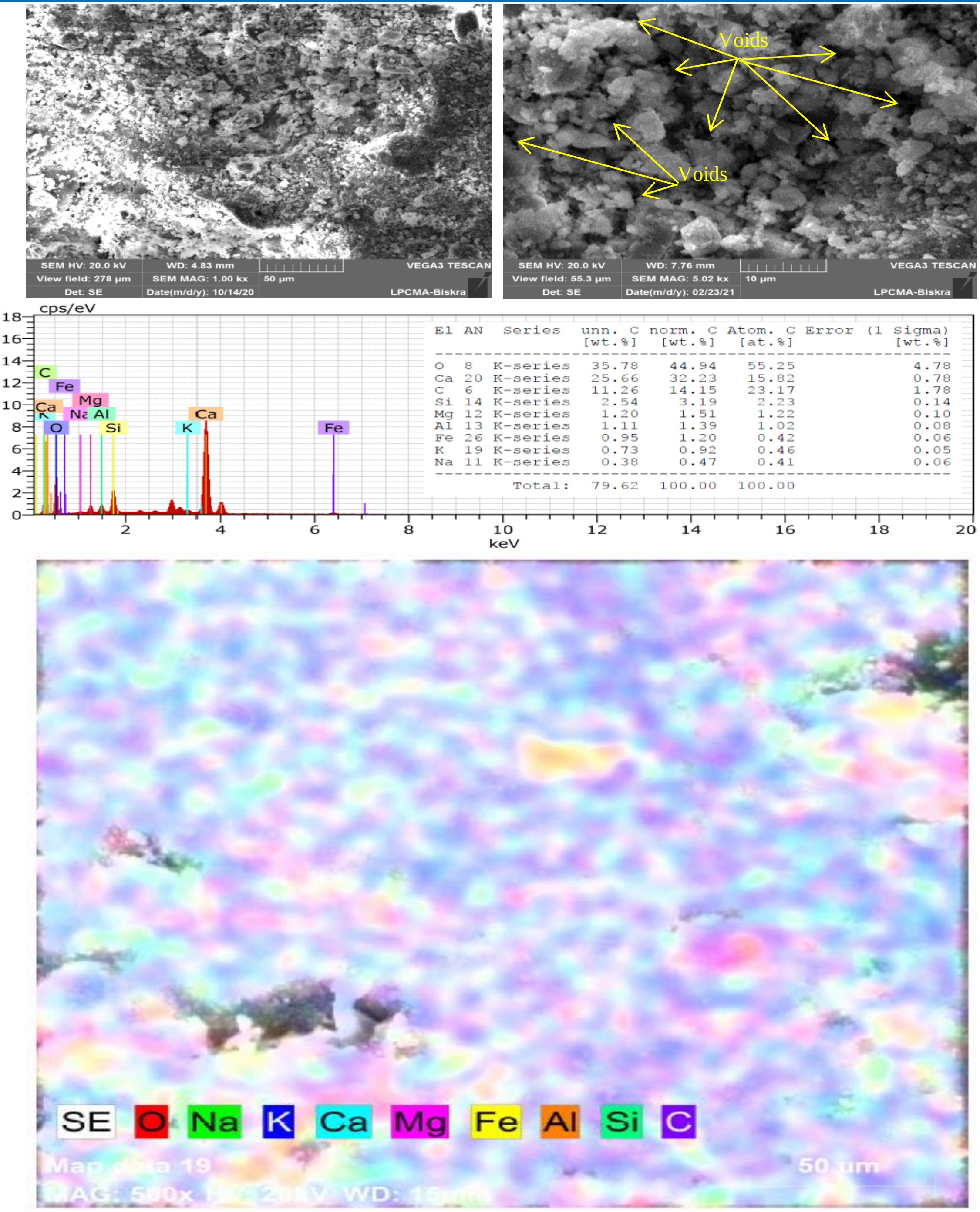
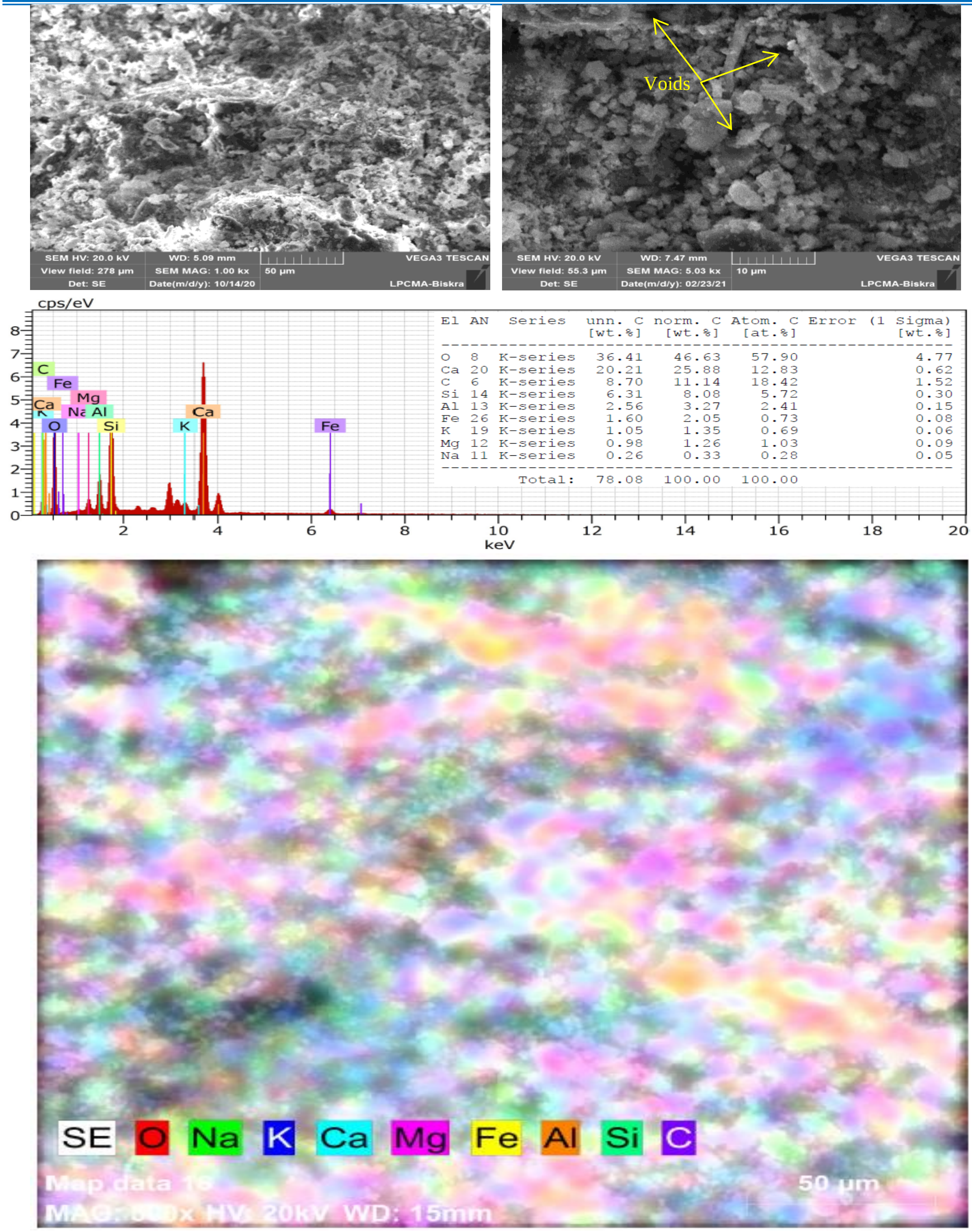


Fig.3. 7 Dry and wet compression strength of EMS as a function of DPA content.

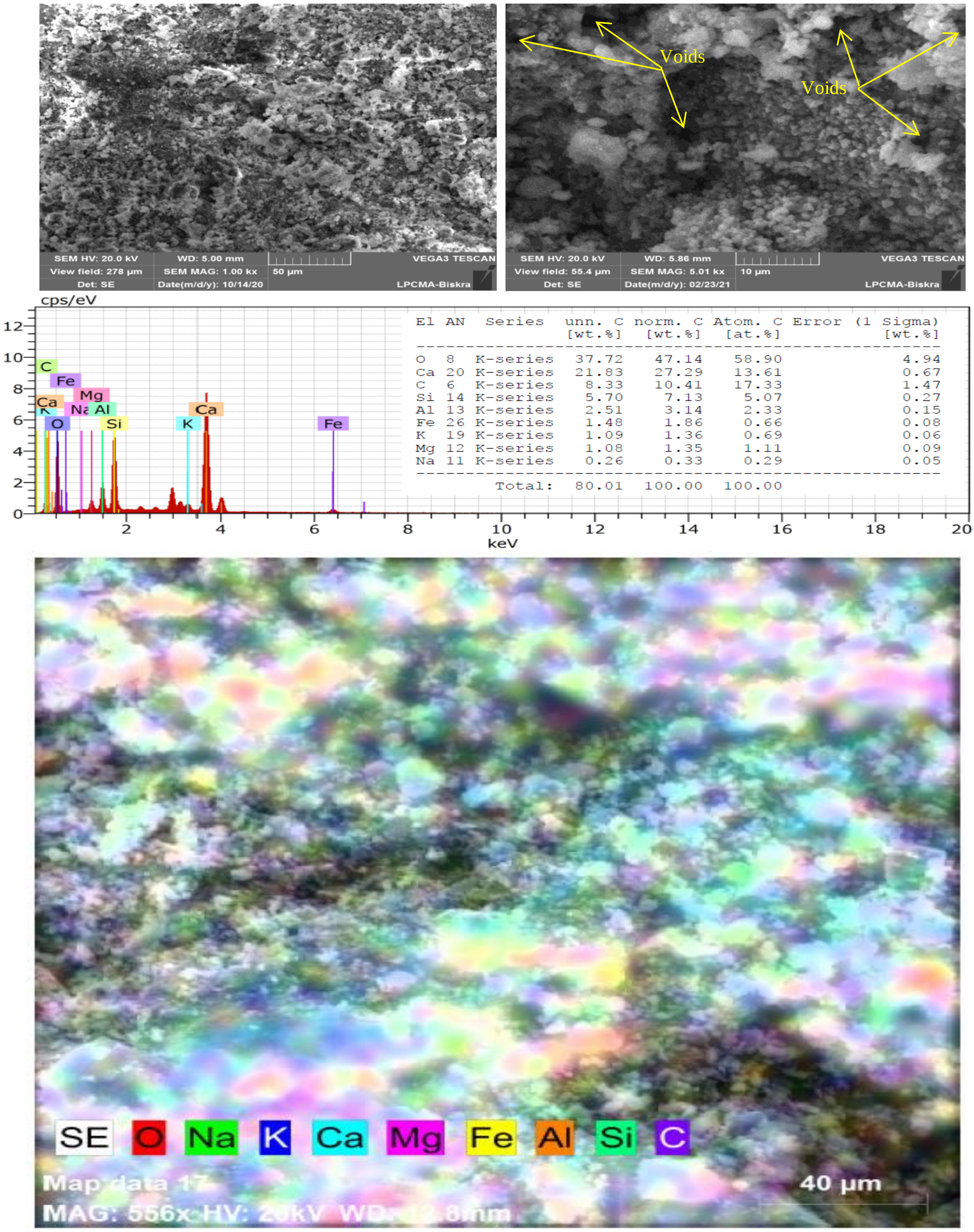


El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	35.78	44.94	55.25	4.78
Ca	20	K-series	25.66	32.23	15.82	0.78
C	6	K-series	11.26	14.15	23.17	1.78
Si	14	K-series	2.54	3.19	2.23	0.14
Mg	12	K-series	1.20	1.51	1.22	0.10
Al	13	K-series	1.11	1.39	1.02	0.08
Fe	26	K-series	0.95	1.20	0.42	0.06
K	19	K-series	0.73	0.92	0.46	0.05
Na	11	K-series	0.38	0.47	0.41	0.06
		Total:	79.62	100.00	100.00	

a: SEM and EDX of EMS 0% DPA



b: SEM and EDX of EMS 6% DPA



El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	37.72	47.14	58.90	4.94
Ca	20	K-series	21.83	27.29	13.61	0.67
C	6	K-series	8.33	10.41	17.33	1.47
Si	14	K-series	5.70	7.13	5.07	0.27
Al	13	K-series	2.51	3.14	2.33	0.15
Fe	26	K-series	1.48	1.86	0.66	0.08
K	19	K-series	1.09	1.36	0.69	0.06
Mg	12	K-series	1.08	1.35	1.11	0.09
Na	11	K-series	0.26	0.33	0.29	0.05
		Total:	80.01	100.00	100.00	

c: SEM and EDX of EMS 10% DPA

Fig.3. 8 SEM and EDX of EMS: (a) 0% DPA, (b) 6% DPA and (c) 10% DPA

3.3.2. Flexural strength and mechanical behavior

A three-point bending test was meticulously conducted to gain a comprehensive and detailed understanding of the mechanical behavior exhibited by the samples, which were incorporated with DPA. Fig.3. 9 and Fig.3. 10 showcase the intricate load-deflection curves alongside the corresponding flexural stress-strain curves obtained from this extensive testing process. Analyzing these significant Figures reveals that all samples consistently exhibit an elastic linear behavior that subsequently leads to brittle failure upon reaching their specified limits. Notably, the flexural strength values demonstrate an initial increase with the gradual rise in DPA content, peaking at an optimal 6%, followed by a subsequent decrease as the ratio of DPA continues to increase further, as illustrated in Fig.3. 11 and thoroughly detailed in Table.3. 1. Interestingly, while the ultimate strain of the samples indicates a decline with an increase in the DPA percentage, this observation elucidates the increase in brittleness of the samples that correlate with the higher DPA content used in the mixture. Moreover, both Fig.3. 11 and Table.3. 1 provide valuable insights into the elastic modulus results observed for the samples under testing. The modulus of elasticity adheres to the trend seen in the compressive and flexural strengths, reflecting a consistent and established relationship between these critical mechanical properties. Notably, the values for the modulus of elasticity range between 217.55 MPa and 399.05 MPa. The percentage of DPA that proved ideal based on the optimal performance across all mechanical properties tested was precisely 6%. The incorporation of this 6% DPA into the earth mortar mixture yielded sigsignificanthancements in the mechanical properties, precisely marked improvements of 18.06% in flexural strength and an impressive 83.42% in the modulus of elasticity when compared to the reference sample used for baseline measurements. As a result, the values attained were 2.81 MPa for flexural strength and 399.05 MPa for the modulus of elasticity, respectively. The corresponding and significant rise in C-S-H content can explain this notable and considerable increase in mechanical properties. This critical factor is substantiated by the findings of the microstructure analysis conducted via SEM-EDX techniques, which were presented extensively in Fig.3. 8.

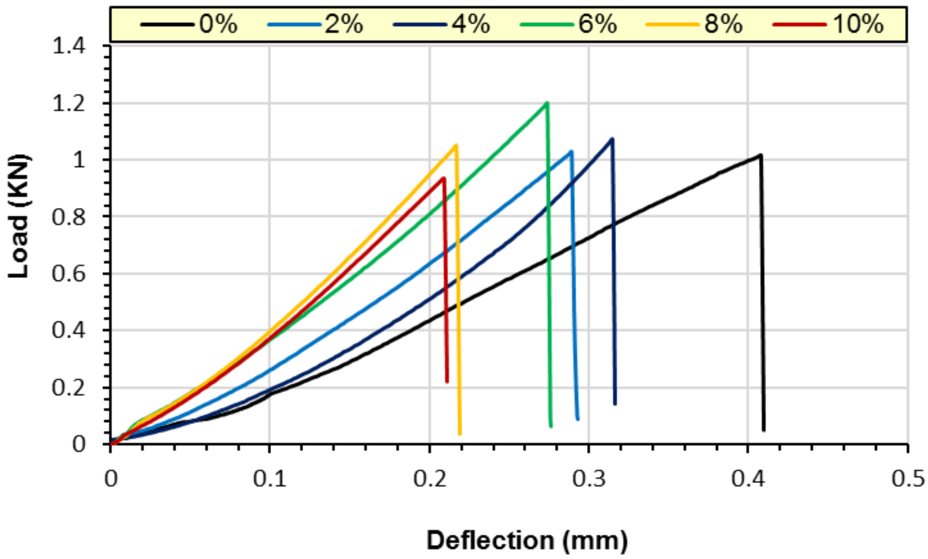


Fig.3. 9 Flexural load-deflection curves of EMS.

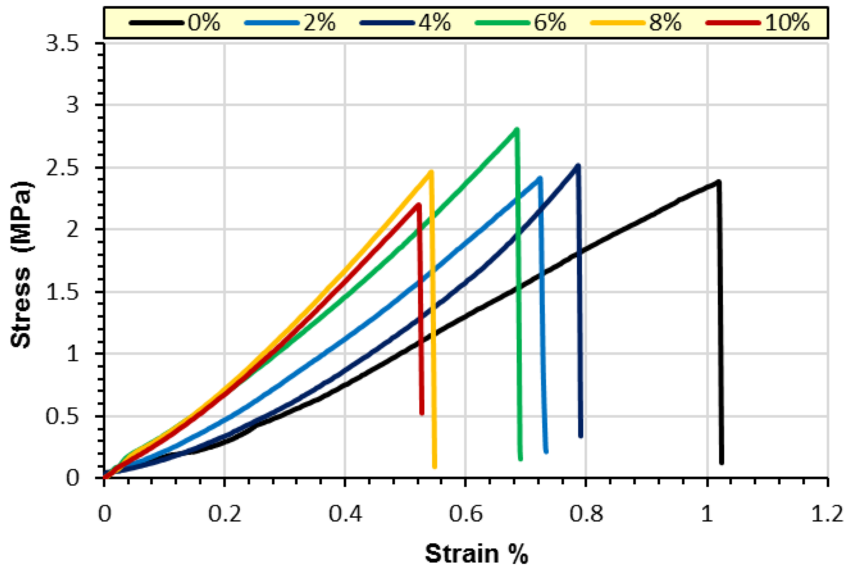


Fig.3. 10 Flexural stress–strain curves of EMS.

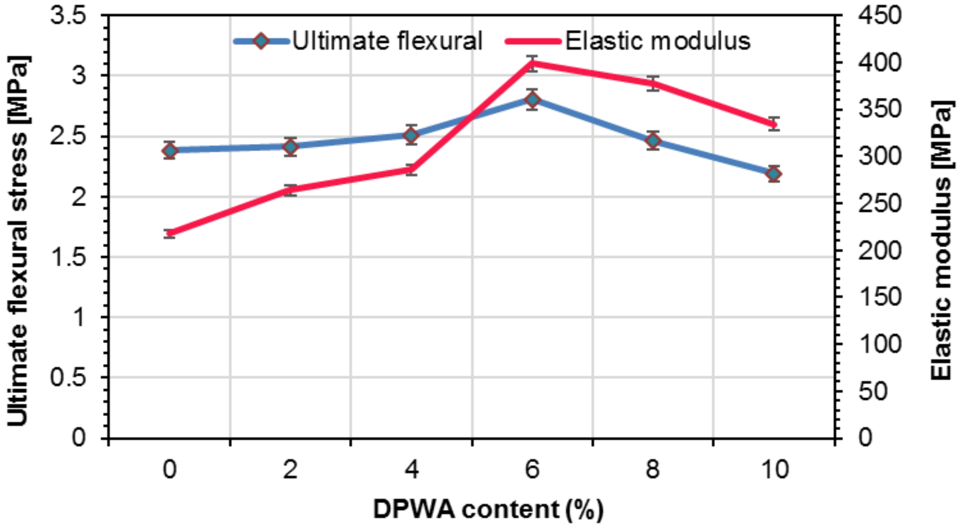


Fig.3. 11 Maximum flexural strength and Flexural elastic modulus of EMS as a function of DPA content.

Table.3. 1 Flexural mechanical parameters.

DPA content (%)	Flexural strength (MPa)	Modulus of elasticity (MPa)	Ultimate strain
0	2.38 ± 0.11	217.55 ± 24.29	1.02 ± 0.09
2	2.41 ± 0.09	263.87 ± 33.23	0.72 ± 0.04
4	2.51 ± 0.09	285.69 ± 33.44	0.79 ± 0.05
6	2.81 ± 0.12	399.05 ± 24.84	0.68 ± 0.04
8	2.46 ± 0.10	377.51 ± 26.23	0.54 ± 0.02
10	2.19 ± 0.08	334.44 ± 20.20	0.52 ± 0.02

3.4. DPA addition effect on durability properties

3.4.1. Total absorption

In this test, we aimed to gain deeper insights into the samples' carrying capacity and performance characteristics while also investigating how they maintain their stability despite being submerged in water for an extended duration of approximately four days. For this comprehensive experimental endeavor, we employed two specific methods to assess the outcomes accurately: the first method involved immersion in plain, unaltered water. In contrast, the second method utilized immersion in boiled water that had reached a high temperature. The findings of the first method are effectively illustrated in Fig.3. 12, which showcases the results obtained from the immersion in plain water. Upon conducting the test, we observed that the maximum water absorption occurred after a careful duration of five hours; interestingly, following this peak, there was a relative increase in absorption until a clear point of stabilization was ultimately reached. The results from the second method, which are represented in Fig.3. 13, depict the outcomes of immersion in boiling water. It became unmistakably evident that the absorption rate in this alternative method was significantly greater, leading us to conclude that the sample to boiling water reached complete saturation much more quickly.

Additionally, an overall examination of total absorption results indicated that incorporating DPA had a notable and statistically significant effect in reducing the absorption rates observed. This reduction can be attributed to the diminished number of voids in the samples, particularly at the 6% DPA concentration, where the lowest water absorption level was reliably recorded. The scanning electron microscopy (SEM) images presented in Fig.3. 8 further support this observation, revealing that the sample containing 6% DPA exhibits noticeably fewer voids than the other tested samples. It is critical to note that all the values obtained through both immersion methods remained comfortably within the acceptable limits for clay bricks, which typically range from 0% to 30%, as documented in reference [184].

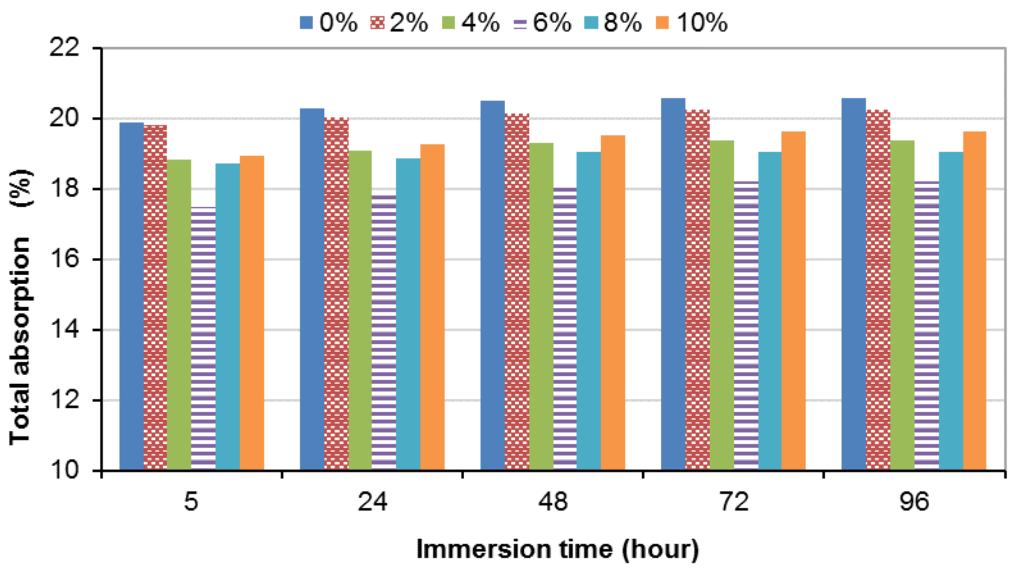


Fig.3. 12 Total absorption of EMS as a function of DPA content, after immersion in water.

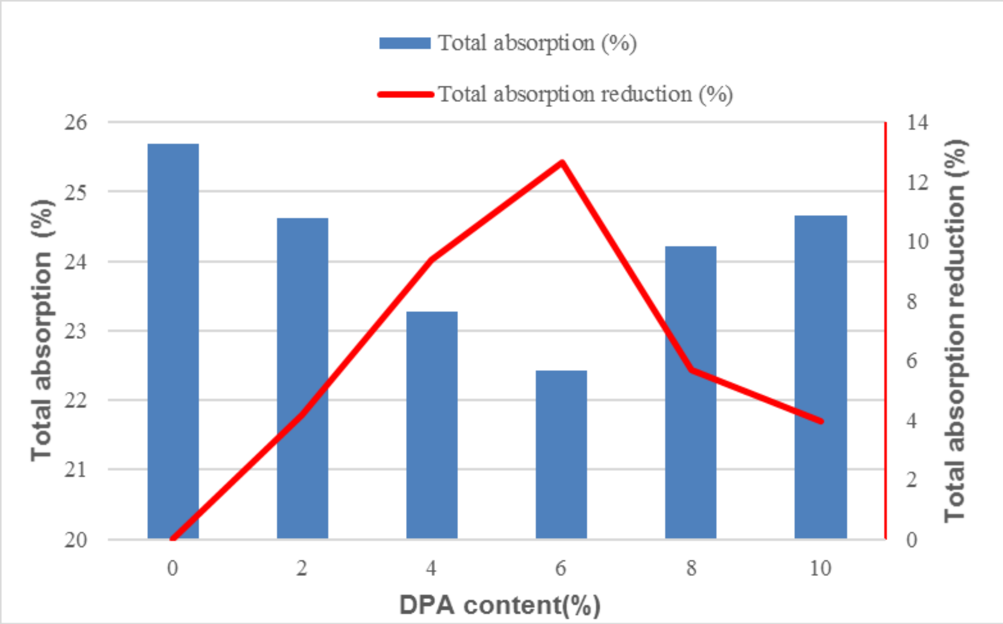


Fig.3. 13 Total absorption of EMS as a function of DPA content, after boiling test.

3.4.2. Capillary absorption

Any brick component can effectively absorb water through the natural process known as capillarity [185]. The volume of water absorbed serves as an essential criterion for evaluating our assessment of the quality of the bulk material; generally, the lower the absorption that occurs, the better the overall performance and efficacy of the brick are perceived to be [186]. In Fig.3. 14, we can observe the capillary water absorption coefficient test outcomes, denoted by Cb. Our comprehensive findings revealed that the lowest absorption level was recorded at 6% of DPA, which exhibited a remarkable decrease of 36% when we compared this to the level of 0% of DPA. It is essential to highlight that DPA, in conjunction with lime, effectively filled the pore spaces within the soil and concurrently worked to cement them together. This process ultimately resulted in a significant reduction in capillary absorption. By thoroughly analyzing the results obtained from this specific and critical test, we can systematically categorize the bricks as exhibiting weak capillary characteristics by the limit values explicitly stipulated in the standard NF XP 13-901, wherein Cb is noted to be less than 20.

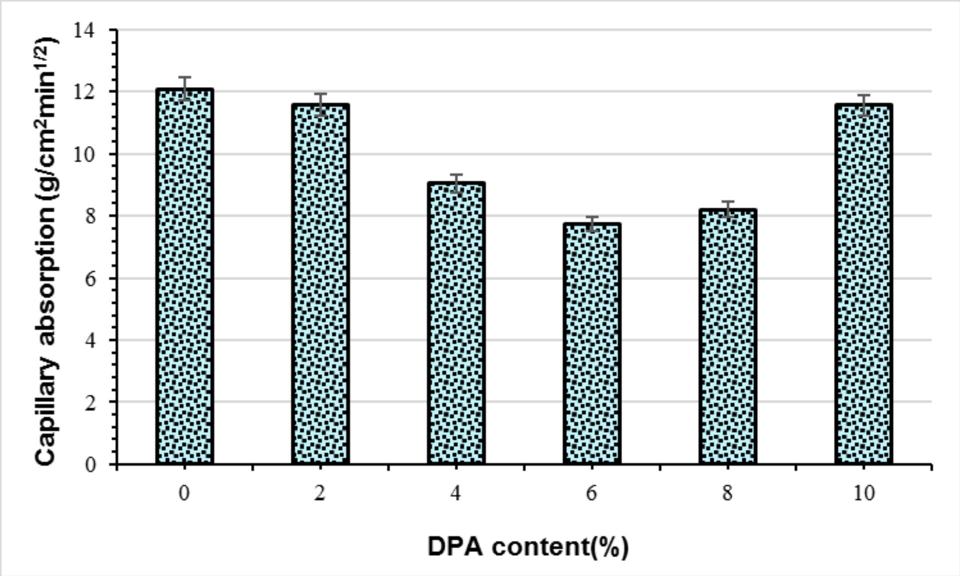


Fig.3. 14 Capillary absorption of EMS as a function of DPA content

3.4.3. Swelling

Swelling is one of the most prominent and telling signs of building degradation, particularly in semi-arid climate regions that share similarities to our specific study area referenced earlier [187]. In Fig.3. 15, we illustrate the remarkable effectiveness of the swelling results observed upon carefully adding DPA to the soil mixture. It has been remarkably noted that the swelling phenomenon experienced a significant decrease as the content of DPA increased, especially notable at specific ratios of 2%, 4%, and 6% DPA. Our extensive findings indicate that the lowest recorded swelling values occurred at a DPA content of 6%, displaying an impressive and substantial decrease of approximately 105%. Adding both lime and DPA plays a vital and significant role in effectively minimizing soil swelling, as these materials facilitate essential particle flocculation processes and promote vital pozzolanic reactions within the soil matrix. Numerous studies conducted in this field have confirmed that several effective solutions exist to reduce soil swelling, mainly through the strategic incorporation of lime (as profoundly highlighted by El Shinawi A; Guidobaldi et al.) [188, 189].

Additionally, the combined use of lime and fly ash has been extensively discussed in the insightful work of Cheshomi et al. [190]. These informative findings suggest a more profound and comprehensive understanding of how these crucial materials interact with various soil properties. Moreover, they greatly emphasize the importance of meticulously selecting the proper additives to mitigate swelling, which can ultimately lead to significantly enhanced structural stability in vulnerable areas, thereby contributing to the overall integrity and longevity of the structures within these regions.

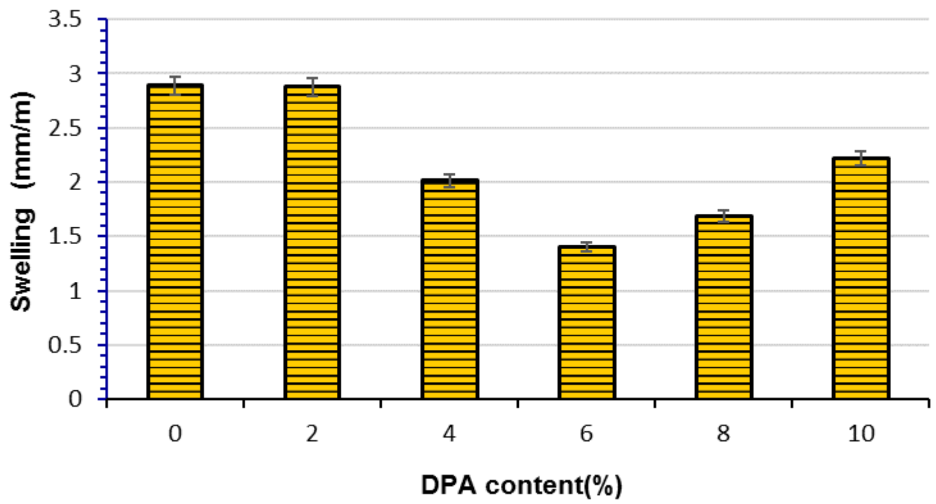


Fig.3. 15 Swelling by immersion of EMS as a function of DPA content.

3.4.4. Drying and wetting

The drying / wetting test is one of the tests recommended to assess the durability of earth mortars according to the standard ASTM D559. According to Gresillon [191], the bricks that are not protected from rain and wind will collapse. Fitzmaurice set severe weight loss limits according to ASTM D559: 5% and 10% for regions with annual rainfall >500 mm and < 500 mm, respectively[192]. Fig.3. 16 shows the results of this test with variation in DPA content at 0% of DPA there is a slight loss of mass and when adding DPA in different proportions, nothing is lost but on the contrary, there is an increase in mass. During the hydration and drying process, lime and DPA complete the reactions and produce portlandite (Ca (OH) 2), ettringite and C-S-H, which explains the slight loss and increase of mass. In Fig.3. 17 shows the samples before and after the test and it note the absence of any deterioration. This indicates that fixation with lime and addition of DPA was very effective, to the point that samples were unaffected and it volume altered by the test intentionally.

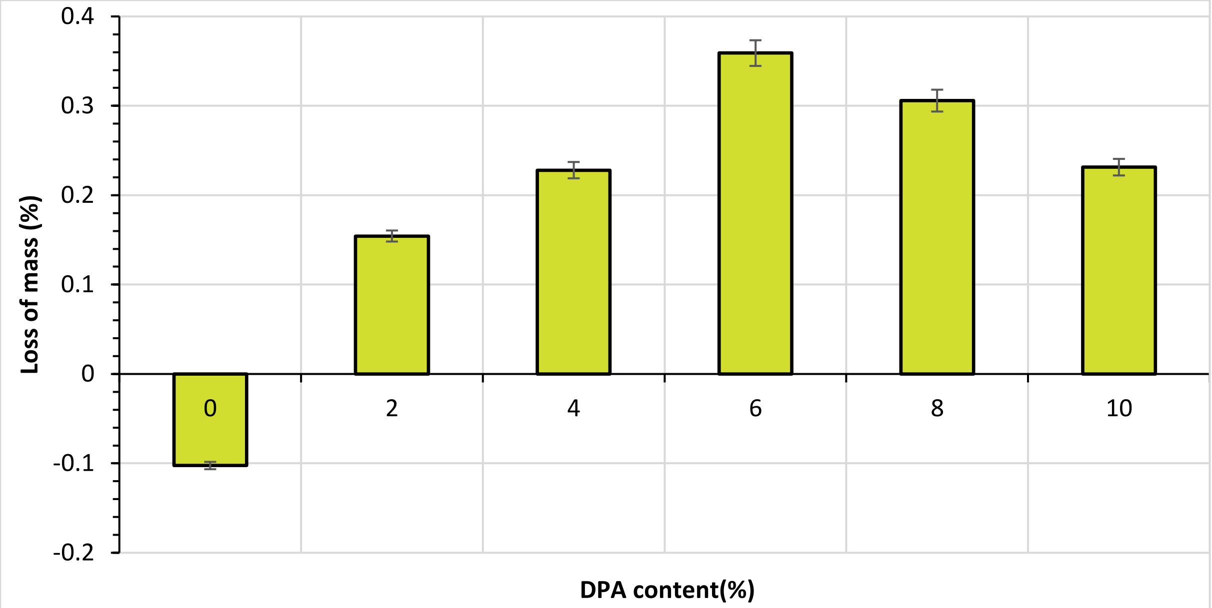


Fig.3. 16 Loss of mass (drying/wetting test) of ESM as a function of DPA content.

Fig.3. 17 (a) ESM before the test, (b) ESM after the test

3.4.5. Chemical durability

Fig.3. 18 illustrates the significant variation in the compressive strength, flexural strength and mass Loss (%) of EMS subjected to sodium sulfate (Na_2SO_4), hydrochloric acid (HCl), and sulfuric acid (H_2SO_4). These samples were systematically compared against control reference samples that remained untreated. The impact of aggressive chemical solutions on EMS is evident through the significant reductions in compressive strength, flexural strength, and mass loss (%), aligning with findings from previous studies.

3.4.5.1. Effect of Sulfuric Acid (H_2SO_4)

Exposure to sulfuric acid induced the most severe deterioration in EMS across all DPA levels. For compressive strength, reference samples showed values of 12.51 MPa at 0% DPA, 14.93 MPa at 4% DPA, 15.21 MPa at 6% DPA, and 13.16 MPa at 10% DPA, while H_2SO_4-exposed samples exhibited reductions to 8.73 MPa, 12.51 MPa, 13.07 MPa, and 9.64 MPa, respectively. Similarly, flexural strength in reference samples ranged from 3.23 MPa at 0% DPA to 2.93 MPa at 10% DPA, decreasing to 2.26 MPa at 0% DPA, 2.34 MPa at 2% DPA, 2.67 MPa at 6% DPA, and 2.05 MPa at 10% DPA after H_2SO_4 exposure. Mass loss was highest under H_2SO_4 exposure, reaching 20% at 0% DPA, 18% at 2% DPA, 13% at 6% DPA, and 19% at 10% DPA. These results align with Bezerra et al. [168], who noted that sulfuric acid causes the dissolution of calcium-based compounds, forming gypsum and ettringite and expanding microcracks, leading to substantial structural weakness. Kasinikota and Tripura [193] also reported comparable mass loss trends in earth-based materials, underscoring their high susceptibility to sulfuric acid attacks.

3.4.5.2. Effect of Hydrochloric Acid (HCl)

Hydrochloric acid exposure caused substantial degradation in EMS mechanical properties compared to reference samples. Compressive strength in reference samples was 12.51 MPa, 14.93 MPa, 15.21 MPa, and 13.16 MPa at 0%, 4%, 6%, and 10% DPA, respectively, dropping to 9.38 MPa, 12.59 MPa, 13.37 MPa, and 10.55 MPa after HCl exposure. Flexural strength declined, with reference values of 3.23 MPa, 3.30 MPa, 3.43 MPa, and 2.93 MPa, reducing to 2.43 MPa, 2.54 MPa, 2.76 MPa, and 2.16 MPa, respectively. Mass loss under HCl exposure was 15% at 0% DPA, 11% at 4% DPA, 10% at 6% DPA, and 14% at 10% DPA. These findings corroborate Sharifi and Visrudi [194], who demonstrated that HCl dissolves calcium hydroxide and destabilizes the binding phases within the mortar matrix, resulting in significant mechanical degradation. Belghit et al. [195] also observed similar reductions in strength and durability in mortars exposed to acidic environments, highlighting the vulnerability of cementitious materials to prolonged acid exposure.

3.4.5.3. Effect of Sodium Sulfate (Na_2SO_4)

Sodium sulfate exposure led to a more moderate reduction in EMS mechanical properties compared to acid exposures, yet still significant compared to reference samples. Compressive strength in reference samples was 12.51 MPa, 14.93 MPa, 15.21 MPa, and 13.16 MPa at 0%, 4%, 6%, and 10% DPA, respectively, decreasing to 10.77 MPa, 12.97 MPa, 13.42 MPa, and 11.00 MPa after Na_2SO_4 exposure. Flexural strength in reference samples ranged from 3.23 MPa to 2.93 MPa, reducing to 2.59 MPa, 2.64 MPa, 2.92 MPa, and 2.25 MPa, respectively. Mass loss under Na_2SO_4 exposure was the lowest among the chemicals, at 12% at 0% DPA, 10% at 2% DPA, 7% at 6% DPA, and 11% at 10% DPA. This degradation is attributed to the formation of sulfate-based expansive compounds, causing internal micro cracking, as documented in sulfate-exposed mortars [193]. Bezerra and Azeredo [168] reported similar compressive and flexural strength reductions under sulfate attack, confirming that sulfate solutions induce gradual but consistent weakening of cementitious materials.

The strength retention ratios across chemical exposures clearly illustrate the varying degrees of degradation in EMS. Samples exposed to Na_2SO_4 consistently demonstrated high retention, maintaining approximately 83.5% to 88.2% of their reference compressive strength and 76.8% to 85.1% of their reference flexural strength, depending on the DPA content. Exposure to HCl resulted in retention of about 75.0% to 87.9% of compressive strength and 73.7% to 80.5% of flexural strength. In contrast, H_2SO_4 exposure consistently yielded the lowest retention, ranging from 69.8% to 85.9% for compressive strength and 70.0% to 77.8% for flexural strength. These findings align with previous research, which reported that sulfuric acid causes the most severe deterioration, followed by hydrochloric acid and sodium sulfate [196]. Mass loss trends further support these observations, with sulfuric acid exposure causing the highest material loss, reinforcing its destructive impact.

This study's findings confirm that chemical exposure significantly compromises the mechanical and durability properties of earth mortars. Sulfuric acid caused the most severe degradation, followed by hydrochloric acid and sodium sulfate. The incorporation of DPA up to 6% improved resistance to chemical attacks, but higher dosages (8%-10%) resulted in noticeable reductions in both compressive and flexural strength.

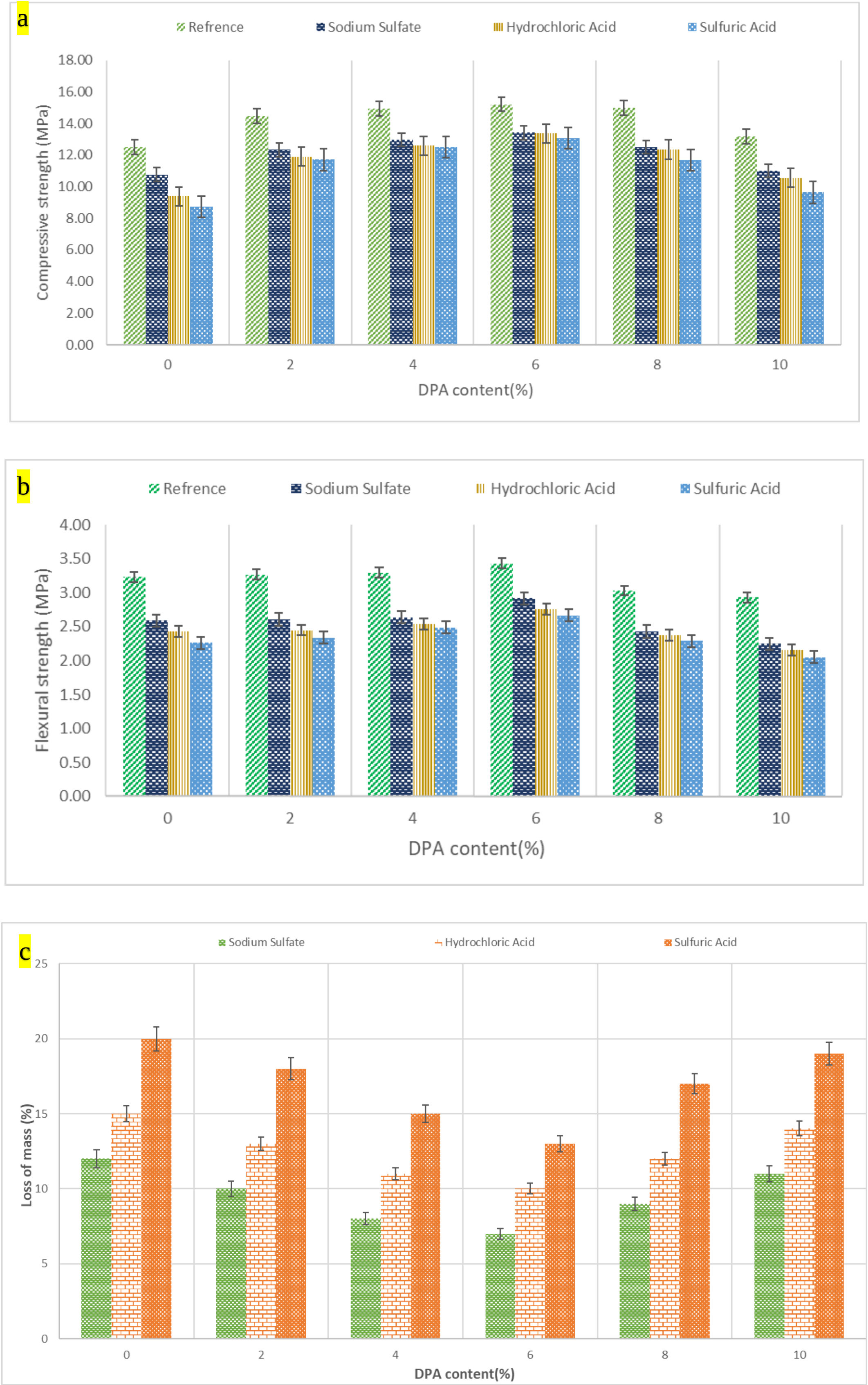


Fig.3. 18 Effect of DPA Content of EMS under Chemical Exposure: (a) Compressive Strength, (b) Flexural Strength, (c) Loss of Mass

3.4.6. Accelerated Carbonation

The mechanical properties (compressive strength and flexural strength) and mass gain of EMS incorporating DPA at concentrations of 0%, 2%, 4%, 6%, 8%, and 10% were evaluated post-accelerated carbonation to assess their performance under simulated carbon-rich conditions, as shown in Fig.3. 19. Before accelerated carbonation, the compressive and flexural strengths exhibited a non-linear response to DPA content: samples without DPA (0%) displayed baseline values of 12.51 MPa (compressive) and 3.23 MPa (flexural), while strengths peaked at 15.21 MPa and 3.43 MPa, respectively, at 6% DPA, indicative of optimal pozzolanic enhancement and reduced porosity, but declined to 14.98 MPa and 3.03 MPa at 8% DPA, and further to 13.16 MPa and 2.93 MPa at 10% DPA, likely due to increased porosity or weakened bonding at higher ash levels. After accelerated carbonation, mass gain over 42 days under ambient conditions also revealed distinct trends: samples at 0% DPA increased from 101 g to 109 g (7.9% gain), attributed to moisture sorption or incipient carbonation; at 2% DPA, mass surged from 115 g to 124 g (7.83% gain), indicating pronounced hygroscopicity or carbonation-driven reactions; at 4% DPA, mass rose from 114 g to 122 g (7 % gain), stabilizing post-14 days; at 6% DPA, mass modestly increased from 103 g to 109 g (5.8% gain), suggesting stability; whereas at 8% and 10% DPA, mass remained constant at 106 g and 110 g, respectively (0% gain), potentially reflecting saturation, porosity limitations, or diminished reactivity, with the preparation process at 65 ± 2°C possibly contributing to early carbonation, especially at 8-10% DPA, accelerating carbonation levels within days. The samples with 0% DPA showed a compressive strength of 13.48 MPa (7.8% increase) and a flexural strength of 3.49 MPa (8.0% increase), correlating with their pre-carbonation mass gain of 7.9%, likely due to $CaCO_3$ formation but constrained by limited pozzolanic activity and potential porosity changes, with mass gain potentially increasing or stabilizing further, reflecting carbonation enhancement tempered by initial limitations. For 2% DPA, the compressive strength rose to 15.08 MPa (4.22% increase) and flexural strength to 3.85 MPa (17.74% increase), alongside a pre-carbonation mass gain of 7.83%, indicating robust pozzolanic and carbonation-driven enhancement through C-S-H or $CaCO_3$ formation, which mitigates porosity and bolsters cohesion, though the significant mass gain warrants scrutiny for potential instability, with post-carbonation mass gain likely augmented due to intensified CO_2 reactivity. At 4% DPA, the compressive strength reached 16.01 MPa (7.2% increase) and flexural strength 3.54 MPa (7.3% increase), aligning with a pre-carbonation mass gain of 7 %, suggesting stable improvement from pozzolanic activity and carbonation, enhancing mechanical properties without excessive hygroscopic effects, with post-carbonation mass gain potentially sustained or slightly increased. For 6% DPA, the compressive strength increased to 16.08 MPa (5.7% increase) and flexural strength to 3.63 MPa (5.83% increase), corresponding to a pre-carbonation mass gain of 5.8%, confirming this as the optimal ratio for maximizing mechanical strength and mass stability through balanced pozzolanic and carbonation effects, with post-carbonation mass gain likely remaining stable or modestly enhanced due to optimal cohesion and porosity reduction. However, at 8% DPA, the compressive strength decreased to 14.98 MPa (0% change) and flexural strength to 3.03 MPa (0% change), with no pre-carbonation mass gain, likely due to elevated porosity or impaired bonding limiting carbonation benefits, despite the high CO_2 exposure, with post-carbonation mass gain remaining unchanged or minimally increased, reflecting early carbonation during preparation and porosity constraints. Similarly, at 10% DPA, the compressive strength remained at 13.16 MPa (0% change) and flexural strength at 2.93 MPa (0% change), with no pre-carbonation mass gain, indicating porosity-driven constraints and minimal carbonation enhancement, with post-carbonation mass gain likely unchanged or slightly increased, constrained by preparation-induced carbonation and bonding limitations. These findings are consistent with prior research studies on Palm Oil Fuel Ash (POFA), such as Awal and Abubakar [197], which report strength and mass gain enhancements at moderate ash levels due to pozzolanic or carbonation reactions but declines or stability at higher concentrations due to porosity, aligning with the limited response at 8% and 10% DPA.

Additionally, the studies conducted by Blaisi [198] and Kareche et al.[199] support the enhancement of mechanical properties and mass stability or gain at low to moderate DPA or fiber contents. However, their findings indicate a reduction in efficacy at higher replacement levels due to increased porosity and weakened interfacial bonding. These results align with the observed post-carbonation trends in the present study, highlighting the importance of an optimal DPA concentration (approximately 6%) in achieving maximum compressive strength, flexural strength, and mass gain under accelerated carbonation conditions. Furthermore, the influence of early carbonation effects during material preparation must be considered, as it can impact the hydration reactions and contribute to variations in mechanical performance and long-term durability.

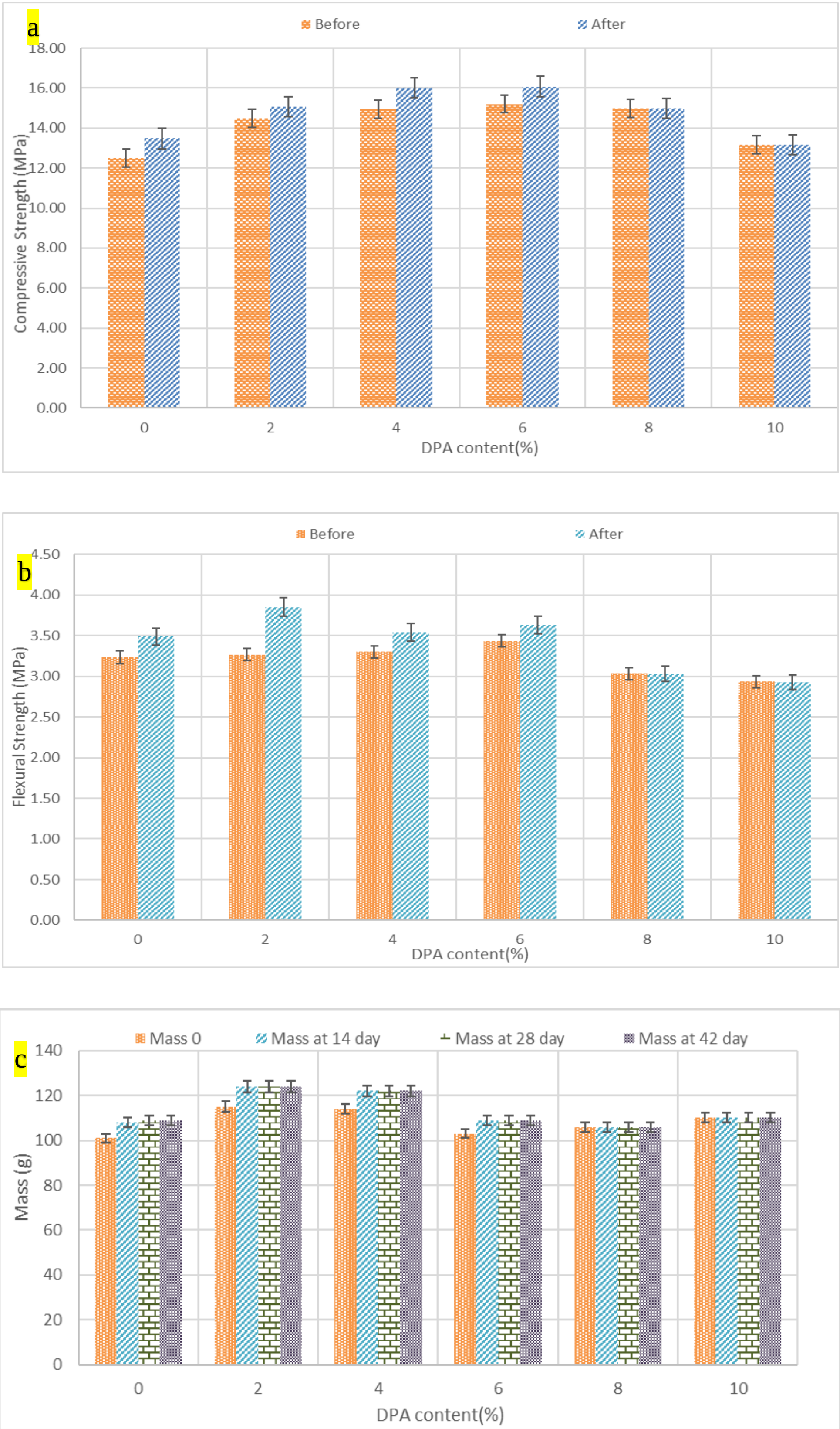


Fig.3. 19 Effect of DPA Content of EMS after accelerated carbonation: (a) Compressive Strength, (b) Flexural Strength, (c) Mass of EMS

3.5. Conclusion

This chapter has presented a comprehensive analysis of the impact of Date Palm Ash (DPA) addition on the properties of earth mortar, revealing its potential as a sustainable and effective stabilizer. The results demonstrate that an optimized DPA content of 6% significantly enhances the physical, mechanical, and durability characteristics of earth mortar. Specifically, this concentration led to improved porosity, bulk density, ultrasonic pulse velocity, abrasion resistance, linear shrinkage, and compressive strength, aligning with the findings that DPA improves the mortar's matrix and strengthens molecular bonding.

Furthermore, the chapter highlighted the complex interplay between DPA content and durability, particularly in chemically aggressive environments. While 6% DPA enhanced chemical resistance, higher concentrations (8%-10%) were found to reduce durability, especially in the presence of sulfuric acid. Accelerated carbonation tests revealed that 6% DPA strikes an optimal balance, promoting both strength and stability under carbon dioxide-rich conditions, with the added benefit of pozzolanic and carbonation-driven reinforcement. These effects were less pronounced at higher DPA concentrations due to increased porosity or premature carbonation.

These findings support the viability of DPA as a sustainable alternative to traditional stabilizers in earth construction. However, the limitations identified, such as the reduced durability at higher DPA levels and the vulnerability to specific chemical exposures, emphasize the need for careful consideration of DPA content and environmental conditions in practical applications. Future research should focus on optimizing DPA content in conjunction with other stabilizers and exploring protective measures to mitigate chemical deterioration. Overall, the results presented in this chapter contribute valuable insights into the potential of DPA-stabilized earth mortar as a promising, eco-friendly solution for resilient and low-impact construction practices.

GENERAL CONCLUSION AND PERSPECTIVES

GENERAL CONCLUSION AND PERSPECTIVES

1. General conclusion

Earth-based construction materials offer a range of benefits, including affordability, energy efficiency, reduced environmental footprint, and improved thermal comfort. This research focused on optimizing earth mortar by determining an ideal lime content of 12% by weight of soil and evaluating the partial replacement of lime with date palm ash (DPA) to enhance its physical, mechanical, hygroscopic, chemical durability, and carbonation resistance properties. Through extensive experimental analysis, this study yielded significant insights into the performance of DPA-stabilized earth mortar (EMS) under diverse conditions.

Incorporating DPA at an optimal level of 6% markedly improved the mortar's matrix. It reduced porosity by 8%, increased dry bulk density, ultrasonic pulse velocity, and abrasion resistance by 7.1%, 30%, and 471%, respectively, and enhanced linear shrinkage, dry compressive strength (DCS), and wet compressive strength (WCS) by 40%, 21.7%, and 16.5%, respectively, compared to lime-only samples. Elastic modulus also rose by 83%, while hygroscopic properties improved with reductions in total water absorption, capillary water absorption, and swelling. Microstructural analysis via SEM and EDX confirmed these enhancements, showing fewer voids and increased calcium-silicate-hydrate (C-S-H) content, strengthening molecular bonding.

Chemical durability tests have unequivocally confirmed the significant influence of DPA on the performance of EMS at a concentration of 6% DPA. The samples were systematically subjected to exposure to various aggressive chemical solutions, including potent substances such as sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and sodium sulfate (Na_2SO_4), each of which resulted in varying degrees of material degradation. Among these tested chemicals, H_2SO_4 was found to cause the most severe deterioration of the material properties. Specifically, exposure to sulfuric acid led to compressive strength diminishing to 13.07 MPa, while flexural strength was reduced drastically to 2.67 MPa. Furthermore, mass loss during this exposure reached a troubling 13%. In contrast, exposure to HCl resulted in a somewhat less severe impact, with compressive strength decreasing to 13.37 MPa and flexural strength also experiencing a reduction, falling to 2.76 MPa. The mass loss associated with hydrochloric acid exposure was recorded at 10%. On the other hand, sodium sulfate showed a milder effect compared to the aforementioned acids. Its application lowered compressive strength to 13.42

MPa, while flexural strength was reduced to 2.92 MPa, and mass loss was limited to 7%. The tests reveal a consistent trend in strength retention when exposed to different chemical agents. Sulfuric acid (H_2SO_4) proved to be the most destructive, consistently resulting in the lowest retention of strength. Hydrochloric acid (HCl) was less detrimental than H_2SO_4 but more so than sodium sulfate (Na_2SO_4). Sodium sulfate (Na_2SO_4) consistently yielded the highest strength retention, indicating that it was the least destructive among the chemical agents tested. Specifically, when considering the average flexural strength retention across all ash percentages, H_2SO_4 exhibited an average retention rate of approximately 73.40%. HCl showed an average retention of around 76.64%, while Na_2SO_4 demonstrated the highest average retention at approximately 80.36%. This hierarchy of destructive effects is further corroborated by the mass loss data, which show that H_2SO_4 consistently caused the highest mass loss and Na_2SO_4 the least These observations underscore the critical finding that a 6% DPA incorporation significantly enhances chemical resistance in materials, whereas higher dosages of DPA, specifically in the ranges of 8% to 10%, can lead to a paradoxical reduction in overall durability and chemical stability.

Accelerated carbonation tests have revealed an important role played by DPA in the enhancement of mechanical properties and overall stability of the materials tested. Before carbonation took place, the compressive and flexural strengths reached their highest values at 15.21 MPa and 3.43 MPa with the inclusion of 6% DPA; however, as the concentration was increased to 10% DPA, there was a decline in strength values to 13.16 MPa for compressive strength and 2.93 MPa for flexural strength. This decrease is attributed to an increase in porosity that occurs at higher DPA percentages. After the carbonation process, the samples containing 6% DPA exhibited a modest increase in strength, resulting in values of 16.08 MPa, which corresponds to a 5.7% increase, as well as 3.63 MPa for flexural strength, equating to a 5.8% increase. Additionally, these samples experienced a 5.8% gain in mass, demonstrating the dual effect of pozzolanic and carbonation-driven reinforcement mechanisms through the formation of calcium silicate hydrate (C-S-H) and calcium carbonate ($CaCO_3$). On the other hand, lower levels of DPA, specifically at 2%, demonstrated even higher gains, such as an 4.22% increase in compressive strength and a 7.83% gain in mass. Conversely, DPA concentrations between 8% and 10% showed no significant changes in strength, which could be attributed to constraints imposed by increased porosity or the occurrence of early carbonation during the preparation phase of the samples. These findings collectively highlight that 6% DPA presents the optimal balance for achieving desirable strength and maintaining stability when conditions rich in carbon dioxide are present.

These findings demonstrate that DPA at 6% significantly enhances the performance of earth mortar across multiple dimensions of mechanical strength, chemical resistance, and carbonation behavior, making it a viable, sustainable alternative to traditional stabilizers. This research advances the utilization of bio-waste in construction, aligning with global sustainability goals by improving the viability of earth-based materials for modern applications. However, limitations emerged: higher DPA levels (8%-10%) reduced durability and carbonation benefits, and chemical exposure highlighted vulnerabilities, particularly sulfuric acid. Finally, this work establishes DPA-stabilized earth mortar as a promising, eco-friendly solution, laying the groundwork for resilient, low-impact construction practices.

2. Perspectives

This study utilized unconventional raw materials, namely date palm ash (DPA) and earth-based constituents, to enhance the properties of earth mortar. It represents an initial effort to characterize these components, explore their interactions, and assess the feasibility of developing sustainable construction composites from agro-resources. The promising results open up multiple avenues for future research, each building on the axes investigated in this work. The following perspectives could guide subsequent studies:

1. Evaluation of the real-scale mechanical properties of earth mortar structures stabilized with optimal DPA content (6%) in applications such as walls or panels.
2. Investigation of the dynamic behavior of DPA-stabilized earth mortar under real-scale conditions, including thermal and moisture cycling, to assess practical performance.
3. Development of numerical and analytical models to simulate the behavior of composite structures made from DPA-stabilized earth mortar, incorporating chemical durability and carbonation effects.
4. Analysis of the microstructure of DPA-stabilized earth mortar using advanced techniques like X-ray micro-tomography or XRD to elucidate void reduction and C-S-H formation mechanisms further.

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