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Title

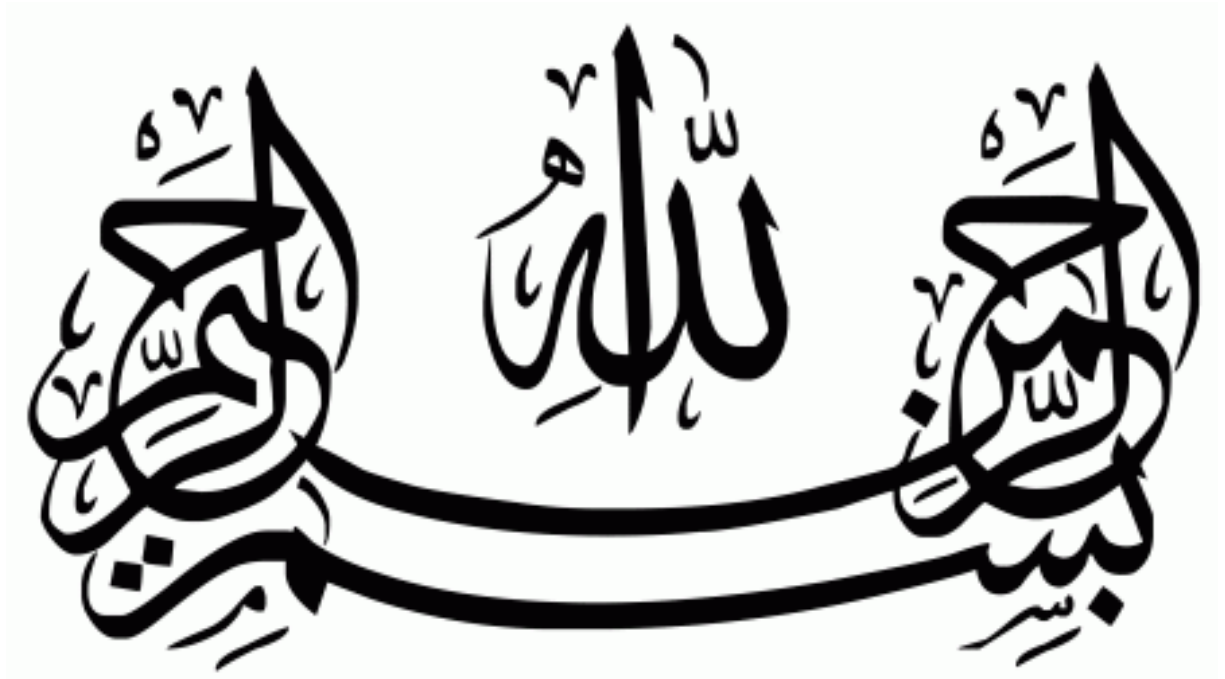
**Valorization of waste tire rubber and natural
fibers for the production of low
environmental impact mortars**

Defended on: 06/07/2026

Before the jury composed of:

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DEDICATION

*To my parents, my first foundation and constant strength
To the little ambitious girl I used to be, brave in ways she didn't yet understand
To my brothers and sisters, my silent pillars
To my small circle, rare souls I love quietly and admire endlessly
A rare constancy in a changing season
Laughter softened the weight of difficult moments
Steadfast hearts stood close, without needing to be named
To my nieces and nephews, small souls with a big place in my heart
To my supervisor, whose trust and insight gave direction to this path
To the laboratory team, whose steady hands and shared effort shaped this work
And to a presence that accompanied this journey in ways no page could contain,
defined not by words, but by impact.*

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To my brothers ... Mohamed, Abdelrazzek, and Bilal

thank you for your encouragement, your presence, and for being a constant source of strength

To my sisters, Amel and Saliha, and to my brother's wife, thank you for your love, your kindness, and for the warmth you bring into my life

To my little angels, from Maram to Sanad, the purest souls in my life, the light that softens my days and fills my heart with quiet joy.

To my tiny but precious circle, thank you for your loyalty, your quiet support, and for standing by me through every moment

To myself, thank you for holding on, for the countless sleepless nights, the quiet tears, and the silent victories. For showing up even when things were tough, for believing again after every doubt, and for turning pressure into progress.

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ABSTRACT

The main objective of this study is to demonstrate, both technically and experimentally, the feasibility of partially using waste tire rubber as a substitute for natural sand, with the incorporation of natural fibers, in the production of low environmental impact mortars. The experimental study involved comparing the performance of a reference mortar with that of mortars incorporating different substitution rates of waste tire rubber: 10%, 20%, and 30% with 1% of natural fibers. The properties were evaluated in both the fresh state (workability, consistency) and the hardened state (mechanical strength and durability performance). The results obtained indicate that the incorporation of waste tire rubber and natural fibers into the formulation of low-impact mortars is technically feasible. In fact, the performance of mortars with certain substitution rates remains comparable to that of the control mortar, both in terms of workability and mechanical strength. Beyond certain substitution rates, some deterioration in properties is observed, but they remain within acceptable limits depending on the intended applications. The partial use of waste tire rubber and natural fibers in the production of low environmental impact mortars represents a promising alternative for the valorization of waste materials, while also contributing to the reduction of natural resource exploitation and environmental pollution.

Keywords: valorization, waste tire rubber, natural sand, natural fibers, low-impact mortars, workability, mechanical strength, durability.

RESUME

L'objectif principal de cette étude est de démontrer, à la fois techniquement et expérimentalement, la faisabilité d'utiliser partiellement du caoutchouc de pneus usagés comme substitut du sable naturel, avec l'incorporation de fibres naturelles, dans la production de mortiers à faible impact environnemental. L'étude expérimentale a consisté à comparer les performances d'un mortier de référence avec celles de mortiers incorporant différents taux de substitution de caoutchouc de pneus usagés : 10 %, 20 % et 30 %, associés à 1 % de fibres naturelles. Les propriétés ont été évaluées à l'état frais (ouvrabilité, consistance) et à l'état durci (résistance mécanique et la durabilité). Les résultats obtenus indiquent que l'incorporation du caoutchouc de pneus usagés et des fibres naturelles dans la formulation de mortiers à faible impact est techniquement faisable. En effet, les performances des mortiers avec certains taux de substitution restent comparables à celles du mortier de contrôle, tant en termes d'ouvrabilité que de résistance mécanique. Au-delà de certains taux de substitution, une certaine détérioration des propriétés est observée, mais elles restent dans des limites acceptables selon les applications visées. L'utilisation partielle du caoutchouc de pneus usagés et des fibres naturelles dans la production de mortiers à faible impact environnemental représente une alternative prometteuse pour la valorisation des déchets, tout en contribuant à la réduction de l'exploitation des ressources naturelles et de la pollution environnementale.

Mots clés : Valorisation. Caoutchouc de pneus usagés, sable naturel, fibres naturelles, mortiers à faible impact environnemental, ouvrabilité, résistance mécanique, durabilité.

ملخص

الهدف الرئيسي من هذه الدراسة هو إثبات الجدوى، من الناحيتين التقنية والتجريبية، لاستخدام مطاط إطارات السيارات المستعملة جزئياً كبديل للرمال الطبيعي، مع دمج الألياف الطبيعية، في إنتاج المطاط ذو الأثر البيئي المنخفض. شملت الدراسة التجريبية مقارنة أداء المطاط مرجعي مع أداء مطاط يحتوي على معدلات استبدال مختلفة لمطاط الإطارات المستعملة 10 %، 20 %، و30 %، مرتبطة بـ 1 % من الألياف الطبيعية. تم تقييم الخصائص في الحالة الطازجة (القابلية للتشغيل، الاتساق) وفي الحالة المتصلبة (القوة الميكانيكية والمتانة). تشير النتائج المحصلة على أنها أن دمج مطاط الإطارات المستعملة والألياف الطبيعية في تركيبة المطاط ذو التأثير المنخفض ممكن فعلياً. في الواقع، يظل أداء المطاط مع بعض معدلات الاستبدال مقارناً لأداء المطاط المرجعي، سواء من حيث القابلية للتشغيل أو القوة الميكانيكية. خارج بعض معدلات الاستبدال، يُلاحظ بعض التدهور في الخصائص، لكنه تظل ضمن حدود مقبولة اعتماداً على التطبيقات المقصودة. يمثل الاستخدام الجزئي لمطاط الإطارات المستعملة والألياف الطبيعية في إنتاج المطاط ذات التأثير البيئي المنخفض بديلاً واعداً لتأمين النفايات، مع المساهمة في تقليل استغلال الموارد الطبيعية والتلوث البيئي.

الكلمات المفتاحية: تثمين، مطاط إطارات السيارات المستعملة، الرمال الطبيعي، الألياف الطبيعية، المطاط ذو الأثر البيئي المنخفض، القابلية للتشغيل، القوة الميكانيكية، المتانة.

Glossary

GLOSSARY

DPF: Date palm fibers
NF: Natural fibers
NA: Natural aggregates
WTR: Waste tire rubber
CR: Crumb rubber
RuC: Rubberized concrete
SCC: Self compacting concrete
EA: Early age
NS: Natural sand
MP: Mechanical performance
FM: Fineness modulus
RC: Reinforced concrete
ELT: End of life tires
RCM: Rubberized cementitious materials
RA: Rubber aggregates
RMC: Rubber modified concrete
CRC: Crumb rubber concrete
SP: Superplasticizer
FS: Flexural strength
CS: Compressive strength
P: Porosity
WA: Water absorption by immersion
S: Sorptivity
WL: Weight loss
C: Carbonation
RAP: Recycled aggregate pavement
RAA: Recycled asphalt aggregate

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GENERAL INTRODUCTION

General introduction

Problematic

The construction industry is currently facing a dual challenge: meeting the increasing global demand for infrastructure while significantly reducing its environmental impact. Cement-based materials, particularly mortar and concrete, remain the most widely used construction materials worldwide. However, their production is associated with substantial natural resource depletion and significant greenhouse gas emissions, notably due to cement manufacturing.

In parallel, the accumulation of industrial and agricultural waste such as end-of-life tires and date palm residues constitutes a serious environmental concern. Waste tires are non-biodegradable and difficult to dispose of, while date palm biomass residues are often burned, contributing to air pollution and inefficient resource management.

Within the framework of the circular economy, the valorization of these waste materials as secondary raw resources offers a promising pathway toward sustainable construction. The incorporation of crumb rubber (CR) derived from waste tires as a partial substitute for natural aggregates (NA), combined with natural fibers such as date palm fibers, may lead to innovative composite mortars with reduced environmental footprint and enhanced functional properties.

Objectives

This study aims to investigate the feasibility of producing eco-efficient mortars through the partial replacement of natural sand (NS) with CR and the incorporation of treated date palm fibers. The mechanical, physical, and durability performances of the developed composites are evaluated in both fresh and hardened states. Particular attention is given to the influence of rubber substitution rate and fiber content on workability, strength development, water absorption (WA), carbonation resistance, and overall sustainability.

Ultimately, this work aims to identify an optimal mortar formulation capable of achieving an acceptable balance between environmental sustainability, mechanical performance (MP), and durability. This is achieved by encouraging the use of locally sourced waste materials, aligning with the principles of sustainable construction and a circular economy.

General introduction

Structure of the dissertation

This dissertation is divided into two main parts. The first part is the theoretical one, which consists of two chapters; the first chapter, entitled "**Use of Fibers in Mortar and Concrete**," provides an overview of fiber-reinforced cementitious materials. It discusses the different types of fibers used in mortar and concrete, with particular emphasis on natural fibers and date palm fibers. This chapter also presents their physical, mechanical, and chemical characteristics, as well as their influence on the properties of cement-based materials.

The second chapter, entitled "**Effect of Waste Tire Rubber on the Properties of Mortar and Concrete**," focuses on the valorization of waste tires in the construction sector. It reviews the characteristics of CR and its incorporation into mortar and concrete mixtures. Furthermore, it examines the effects of rubber substitution on the mechanical strength, durability, and other relevant properties, based on findings reported in previous studies. The second part is committed to the experimental investigation, presented in two chapters; the third chapter presents the materials used in this study and describes the experimental methodology and testing procedures adopted. The fourth chapter is devoted to the presentation, analysis, and discussion of the experimental results. The influence of CR and date palm fibers on various properties is thoroughly investigated and interpreted. Finally, the dissertation concludes with a summary of the main findings, followed by recommendations and perspectives for future research.

CHAPTER I
***USE OF FIBERS IN MORTAR
AND CONCRETE***

I.1.Introduction

This chapter presents a bibliographic review on composite materials and the use of fibers in mortar and concrete. It begins with general concepts related to fiber-reinforced materials. The chapter then introduces the different types of fibers used in construction, with particular attention given to natural fibers and especially date palm fibers. Their characteristics, as well as their relevance as sustainable reinforcement materials, are discussed. Subsequently, the influence of date palm fibers incorporation on different properties of mortar and concrete is highlighted.

I.2.Generalities on Composite Materials

I.2.1. Definition

A composite material is typically regarded as a blend of two or more constituent materials at a macroscopically homogenous level. It can be described as a material comprising two or more chemically and physically distinct phases, separated by a clear interface. These different systems are combined judiciously to create a system with enhanced structural or functional properties that cannot be achieved by any of the individual components alone.

The main component of composites is a matrix in which reinforcements (fibers) are distributed, either regularly or randomly. The matrix keeps the reinforcements in place and guarantees load transfers, while the reinforcements primarily offer their excellent mechanical qualities (mechanical resistance, elastic limits, modules, etc.). [1] [3]

Essentially, a composite material consists of three phases:

- The matrix phase: also known as the continuous phase
- The filler or reinforcement phase: which is enveloped by the matrix material
- The interface phase: which has different structures and properties compared to the matrix and filler/reinforcement phases. [1]

The performance and characteristics of composite materials are directly influenced by the interaction and composition of these three phases.

Composite materials should exhibit the following features:

- They are microscopically nonhomogeneous with a distinct interface.
- There are significant differences in the performance of the component materials.

- The resulting composite materials should show a substantial improvement in performance.
- The volume fraction of the component materials should exceed 10% [1].

I.2.2. Composition

I.2.2.1. Matrix

The materials used for the matrix in composite design and manufacturing can be categorized into several types:

- Mineral matrices, which encompass cement, gypsum, and concrete mixtures, as well as silicon carbide and carbon.
- Organic polymeric matrices, including thermoplastic resins like polypropylene, polyphenylene sulfone, polyamide, and polyetheretherketone.
- Thermoset resins such as polyesters, phenolic, melamines, silicones, polyurethanes, and epoxies.
- Metallic matrices, which consist of aluminum alloys, titanium alloys, and oriented eutectics. [2]

I.2.2.2. Reinforcement

Reinforcements (See Figure I.1 and Figure I.2), which often involve fibers, are used in composite materials primarily to improve their mechanical qualities (rigidity, breaking strength, hardness, etc.) as well as to enhance physical attributes like electrical, thermal, and fire resistance. Low density, compatibility with the matrices, and simplicity of execution are the qualities that the reinforcements should have.

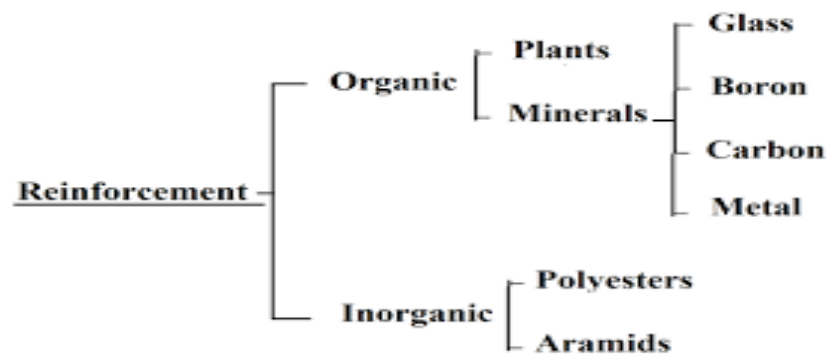


Figure I.1. Types of reinforcement [3].

I.2.2.2.1. Types of reinforcement

a. Aramid fibers

Aramids are aromatic polyamides with over 85% aromatic groups; Kevlar is a well-known example of an aramid fiber. They possess high mechanical properties like carbons, but they have low compressive strength. Poor fiber adherence to the composite material's matrix is typically the cause of the material's low mechanical strength under compression.

Fiber enzymes can be used to fix this. The weaknesses of aramid fiber composites can also be addressed by using hybrid fiber composites.

In the leisure sector, hybrid reinforcements like glass-Kevlar or carbon-Kevlar are commonly used (skiing, tennis rackets, etc.).

b. Ceramic fibers

Composite materials of the ceramic type often consist of a matrix and ceramic reinforcements. Chemical vapor deposition is used to create the fibers on a wire for support. These fibers are used in environments with extremely high temperatures, ranging from 500°C to 2000°C. These substances are specifically used in the heated components of aircraft engines.

c. Carbon fibers

Thin threads of elemental carbon with structures that switch from amorphous to crystalline graphite are known as carbon fibers.

These fibers are created from a fundamental polymer known as a precursor; the precursor's properties have a significant impact on the final carbon fibers' physical and chemical features.

d. Glass fibers

Glass fibers are by far the most advanced reinforcement material currently utilized in the creation of composite constructions due to their exceptional performance-price ratio. The latter have an interesting tensile strength but a low tensile modulus. They are considered as anisotropic materials and are composed of silicates.

e. Other types of fibers

This section gathers all of the remaining fibers, which are typically utilized for particular uses (high electrical conductivity, specific sheets). When compared to the previously listed fibers, they have comparatively low elastic moduli. The fibers in concern are :

- Plant-based fibers, such as cellulose, flax, hemp, etc.
- Mineral-based fibers (silica, asbestos);
- Synthetic fibers made of polyester, polypropylene, or polyethylene, such as nylon, polyester, etc. [3]

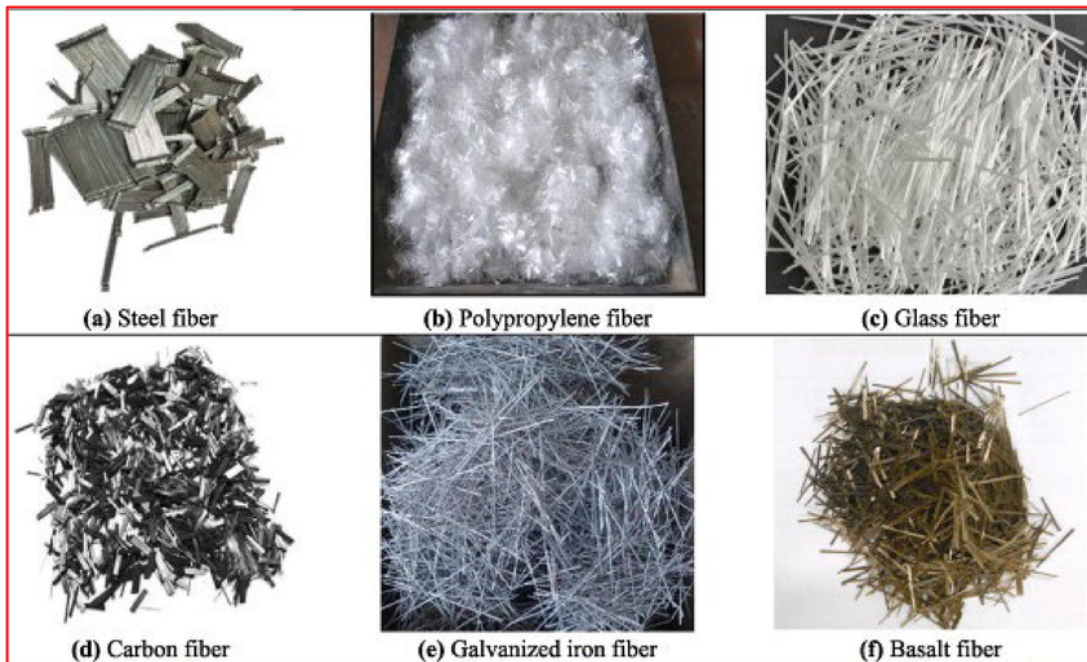


Figure I.2. Types of fibers commonly available and used in concrete reinforcement [4]

I.2.2.3. Additives and Fillers

The fillers, which usually consist of liquids, powders, or fragmented materials, are added to resin to strengthen the mechanical characteristics.

They are generally used in the same way as "conventional" plastics and can be mineral, organic (vegetable or synthetic), or metallic. Die costs can also be decreased by using non-reinforcing fillers. When developing constructions composed of composite materials, Additives like coloring or mold release agents are frequently used [3].

I.2.3. Properties

The following characteristics of a composite can be highlighted:

- A reduction in the thermal expansion coefficient,
- Increase in stiffness,
- Strong mechanical properties, fatigue behavior,
- High stability in dimensions,
- High wear resistance,
- Durability,
- Design adaptability,
- Low costs.

The primary drawbacks of composites are their challenging manufacturing and reproducibility, as well as the fact that some mechanical qualities are sacrificed in order to gain improved ones [5].

I.3.Mortar and Concrete

Mortar and concrete are essential examples of composite materials, where a cementitious matrix bind aggregates and reinforcements to create microscopically heterogeneous structures with enhanced properties. As particulate composites, they consist of a continuous binder phase (e.g., cement paste) and discontinuous fillers (e.g., sand, gravel, or waste-derived particles), often reinforced with fibers for improved performance.

I.3.1. Mortar

Mortar is a construction material created by combining cement, lime paste, sand, and water in specific proportions. Depending on the type of binding material used, building mortar can be categorized into cement mortar, cement lime mortar, lime mortar, and gypsum mortar, among others. Based on its primary uses, it is classified as either masonry mortar or finishing mortar. Masonry mortar is applied to structures like brickwork, stonework, and building blocks, serving the purpose of bonding and load transmission. Finishing mortar, also known as plastering mortar, is utilized for coating the surfaces of structures and their components, providing protection to the substrate, meeting functional needs, and enhancing aesthetic appeal [6].

I.3.2. Concrete

Concrete is one of the most frequently used construction materials today, serving as the foundation for modern structures worldwide. It is employed in the creation of foundations, pavements, building walls, architectural designs, highways, bridges, overpasses, and more. Its versatility ensures that concrete appears in nearly every construction project in some capacity. However, the varied nature of its ingredients, their combinations, and proportions lead to a broad spectrum of concrete types with different characteristics. Consequently, concrete remains a dynamic material that continues to be favored, particularly in the context of the circular economy [7]. Concrete is composed of a blend of aggregate, cement, water, supplementary cementitious materials (SCMs), and/or minerals, along with chemical additives. Once it sets, concrete exhibits the characteristics of solid rock, offering outstanding mechanical strength and strong resistance to chemical substances, freeze-thaw cycles, and fire. [8]

I.3.2.1. Composition of concrete

Concrete is composed of four primary elements: water, cement, fine aggregate, and coarse aggregate. The first two are the most commonly used substances globally and are responsible for concrete's binding properties. In contrast, fine and coarse aggregates are added to increase the material's strength, with differences in the granules' shape and size. [9]

I.3.2.1.1. Hydraulic binders (Portland cement)

The Romans and Greeks already knew how to make hydraulic binder by reacting slaked lime with volcanic ash. Research on the hydraulic properties of lime began at the end of the 18th century, eventually leading around 1840 to the production of modern cements. The Frenchman Louis Vicat discovered the principle of the hydraulicity of lime in 1817, relating to the proportion of clay and the firing temperature and published his findings without taking out a patent. In 1840, Louis Vicat discovered the principles of hydraulicity for Portland cements, through firing at a temperature of 1450°C, which produces clinker. [19]

I.3.2.1.1.1. Cement production

Portland cement is a mineral powder whose property is to harden under water, forming a paste that hardens through a reaction and hydration process. The manufacture of cement involves the following steps :

- Extraction and crushing
- Preparation of the raw mix
- Firing or calcination
- Grinding
- Shipping.

The main component of cement is clinker, obtained by firing a suitable mixture of 80% limestone and 20% clay [10]. The cement manufacturing process is shown in figure I.3.

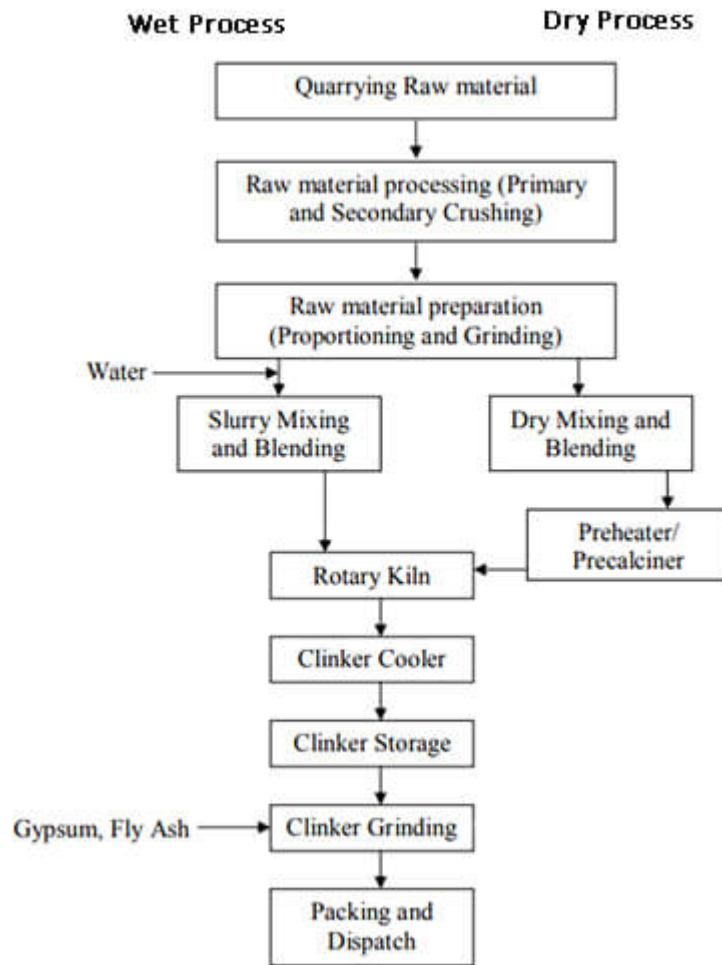


Figure I.3. Cement manufacturing process [11]

I.3.2.1.1.2. Properties of Cement

a. Physical Properties

1. Fineness of Grinding

Cement hydration begins on the surface of the grains, so it is the total surface area of the grains that represents the material available for hydration. The hydration rate therefore depends on the fineness of the cement grains. To achieve rapid hydration and high short-term strength, a high fineness of the cement is required. The method used to measure the specific surface area of cement is the Blaine method. [12]

2.Consistency

Consistency is the optimal percentage of water that allows obtaining a cement paste of normal consistency. The "Vicat" apparatus is generally used to measure this consistency, NF EN 196-3. [13]

3.Setting time

This is the term used to describe the stiffening of the cement paste. Setting is defined as the change from a fluid state to a solid state. The start of setting corresponds to the sudden increase in the viscosity of the cement paste and a rise in its temperature. The end of setting corresponds to the regular and progressive transformation of the cement paste into a rigid block. To measure the time of start and end of setting, the "Vicat" test is generally used; NF EN 196-3[13].

4.Shrinkage

Shrinkage is the reduction in the apparent volume of the material; it is measured on 4×4×16 cm specimens, stored in air at a temperature $T = 20^{\circ}\text{C}$ and relative humidity.

5.Expansion

The volumes of cement pastes, mortars, and concretes swell in the presence of moisture or water. The main causes of expansion are:

- excess gypsum.
- excess free lime CaO .
- excess free magnesium MgO .

Expansion is measured according to NF EN 196-3. [13]

b. Mechanical Properties

The mechanical strengths, both tensile and compressive, of cements are determined on standard mortar specimens measuring 4×4×16 cm at 28 days of age. The mortar strength is then considered representative of the cement's strength. Figure I.4 illustrates the development of the strengths of various cement hydration products.

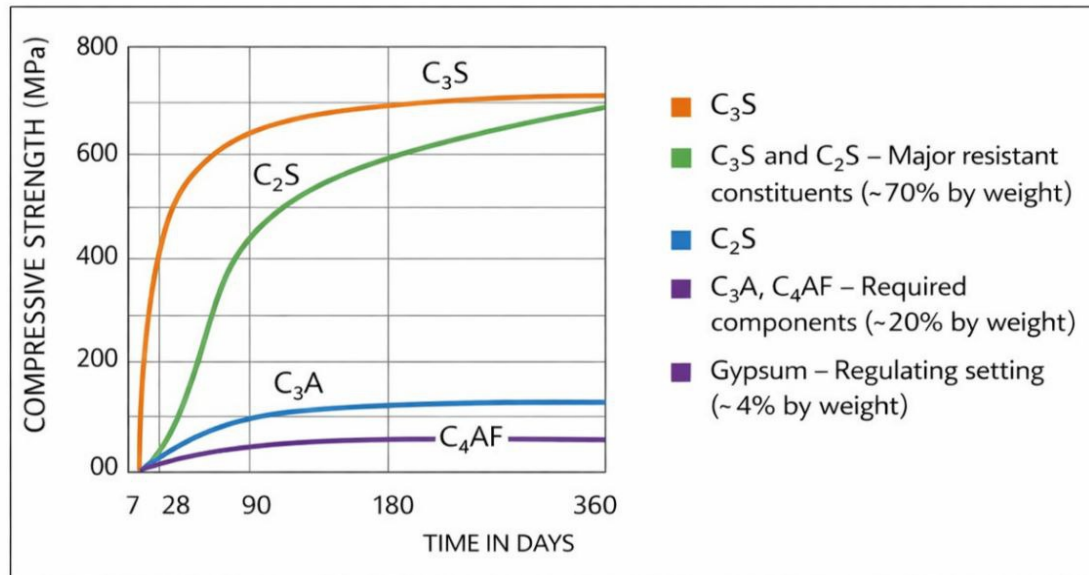


Figure I.4. Development of the strength of various cement hydration products [10]

c. Main Components of Cement

- C₃S: Constitutes approximately 55-65% of cement by volume. It affects early strength.
- C₂S: Constitutes approximately 15-20% of cement by volume. It affects late strength.
- Aluminates account for approximately 20% of cement. Alumina combines with CaO to form C₃A and with iron oxide to form C₄AF.
- C₃A: This is the most important phase affecting setting time. During the reaction, a high amount of heat is released.
- C₄AF: It affects the color of clinker and consequently of the cement.

d. Cement Specifications

d.1 Cement Classification

Standards NA 442 and EN 197-1[14] define five types of cements, classified according to their composition:

- CEM I : Ordinary Portland Cement
- CEM II : Portland Composite Cement

- CEM III : Blast Furnace Cement
- CEM IV : Pozzolanic Cement
- CEM V: Slag and Ash Cement

d.2. Physical and Mechanical Specifications

- Three normal strength classes are covered: 32.5, 42.5, and 52.5.
- For each normal strength class, a subclass for early-age strength (R) is defined.
- For all types of cement, the compressive strength determined according to EN 196-1 [15] must meet the specifications given in Table I.1.

Table I.1. Physical and Mechanical Specifications of Cements According to EN 197–1[14]

Strength class	Compressive strength MPa				Initial setting time	Soundness (expansion)
	Early strength		Standard strength			
	2 days	7 days	28 days		min	mm
32,5 N	–	≥ 16,0	≥ 32,5	≤ 52,5	≥ 75	≤ 10
32,5 R	≥ 10,0	–				
42,5 N	≥ 10,0	–	≥ 42,5	≤ 62,5	≥ 60	
42,5 R	≥ 20,0	–				
52,5 N	≥ 20,0	–	≥ 52,5	–	≥ 45	
52,5 R	≥ 30,0	–				

I.3.2.1.2. Aggregates

The term “aggregates” refers to inert materials like sand and gravel, or stones that are used in the composition of concrete. These are particles ranging from 0 to 125 mm in size, and may have a natural, artificial, or recycled origin.

a- Natural aggregates: Derived from loose or solid rocks extracted on-site, undergoing no treatment other than mechanical processing.

b- Artificial aggregates: Produced through the thermal transformation of rocks, ores, or industrial by-products (such as slag, etc.).

c- Recycled aggregates: These are materials that come from the demolition of existing concrete structures [10].

I.3.2.1.3. Mixing Water

Water, often referred to as the universal solvent, plays a crucial role in concrete as it interacts with cement to facilitate the hardening process. Its significance in concrete composition is underscored by the water/cement ratio, which is calculated by dividing the mass of water by the mass of cement used. A lower water content in the cement mixture can shorten the setting time of concrete, but it may not guarantee optimal stability or performance. Conversely, a higher water content can extend the setting time and potentially compromise the strength of the concrete [9].

I.3.2.1.4. Admixtures

Concrete's construction qualities, including workability, pumpability, setting properties, MP, durability (such as resistance to freeze-thaw cycles), and shrinkage properties, are enhanced by a variety of chemical admixtures. Chemical admixtures also make it possible to create and construct specific concretes, such as sprayed concrete, underwater concrete, high fluidity concrete, and high strength concrete [16].

I.4.Fiber-Reinforced Mortar and Concrete

In recent years, incorporating fibers into cement-based materials has become a popular method for enhancing the strength and longevity of construction materials. Traditionally, this reinforcement has been accomplished using synthetic fibers, such as steel, glass, or polymeric fibers, with polypropylene being one of the most widely used in the construction industry. It is well-regarded for its ability to enhance properties like tensile strength, flexural strength (FS), and crack resistance. However, the production of synthetic fibers requires significant energy and relies on non-renewable resources, leading to concerns about environmental sustainability [17].

Fiber-reinforced concrete (FRC) is a type of concrete that incorporates fibrous materials to enhance its structural strength. This concrete includes short, discrete fibers that are evenly spread and randomly aligned, such as steel, glass, synthetic, and natural fibers, each imparting different characteristics to the concrete. Moreover, the properties of FRC vary depending on the type of concrete, fiber materials, shapes, distribution, orientation, and densities. Fibers are typically added

to concrete to manage cracking caused by plastic and drying shrinkage. They also help decrease the permeability of concrete, thereby minimizing water bleeding. Generally, fibers do not enhance the FS of concrete, so they cannot substitute for moment-resisting or structural steel reinforcements [18].

Considering the economic perspective, fibrous reinforcement in concrete is often favored because it reduces costs compared to traditional concrete by eliminating the time needed for installing conventional reinforcement. Additionally, FRC offers benefits during the casting process since the fibers are pre-mixed into the concrete. This approach is particularly useful for creating secondary precast concrete components used in civil and industrial structures and infrastructure projects [19].

I.4.1. Fibers

We can say that the term "fibers" refers to a large family of materials that have been introduced to the market as new applications. They are classified according to their origin (natural, synthetic, and artificial), their shape (straight, wavy, needle-like, etc.), their size (macro or micro-fiber), and also by their mechanical properties. However, to choose which fibers to use for a given application, one must consider the compatibility of the fiber with the matrix, as well as the performance mode of the composite [20].

I.4.1.1. Natural fibers

Natural fibers (NF), especially those derived from plants, have gained attention as an eco-friendlier alternative. Their ability to biodegrade, renewability, and minimal environmental footprint make them a compelling choice to replace synthetic fibers. Additionally, numerous studies have demonstrated that these fibers can greatly improve the mechanical properties of mortars, enhancing compressive and FS, as well as attributes like workability and Ultrasonic Pulse Velocity (UPV) [17]. Nowadays, NF are increasingly viewed as a sustainable alternative to non-renewable synthetic fibers. They are also generally considered safer for both human health and the environment, as they typically do not require special handling and have a smaller carbon footprint compared to their synthetic counterparts [21].

I.4.1.1.1. Classification

NF can be categorized into three types based on their origins: animal, vegetal and mineral. The figure I.5 present the classification of NF.

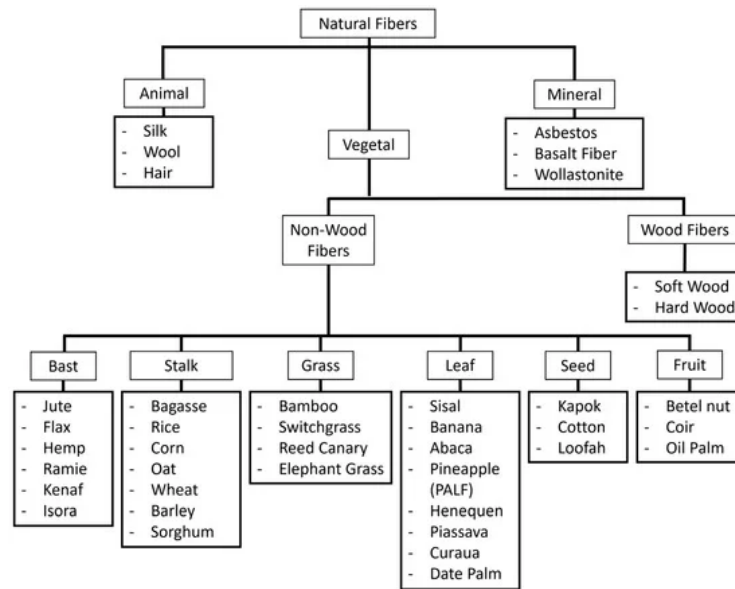


Figure I.5. Classification of natural fibers [21]

I.4.1.2. Vegetal fibers

In the past ten years, there has been an ongoing discussion about the potential benefits of incorporating plant fibers into innovative and eco-friendly composite materials. These vegetal fibers hold significant promise for replacing traditional materials that consume a lot of energy and are often blamed for contributing to global climate change. The environmental advantages of plant fibers lie in their cost-effectiveness (low cost and minimal resource use for production), renewability, low density, recyclability, biodegradability, and ability to sequester carbon [2]. Known as lignocellulosic fibers due to their high cellulose and lignin content, plant-based NF are the most plentiful type of NF [21].

I.5. Date palm fibers (DPF)

Algeria ranks among the top global producers of date palms, with a plantation area exceeding 160,000 hectares and housing 18.6 million date palms. This industry results in approximately

200,000 tons of waste annually, which includes bunches, petioles, trunks, and leaves of the palms. Typically, this waste is incinerated to prevent the accumulation of scorpions and snakes, but this process leads to environmental problems in nearby areas. Currently, date palm fibers are utilized in various sectors to enhance technical performance and reduce costs. In construction, they are incorporated into new building materials and used to improve thermal insulation units. A recent study highlights the application of DPF to lower the thermal conductivity of plaster concrete [22]. A significant quantity of NF remains unutilized worldwide. DPF present an innovative opportunity as an engineering material and could serve as a potential alternative to expensive and non-renewable manufactured fibers. Employing this fiber in engineering applications as a primary material would enhance the value of natural and sustainable resources [23].

I.5.1. Types of DPF

Date palm trees are primarily grown for their fruit rather than their fibers. Nevertheless, they are also recognized as a source of fibrous cellulose. Fibers can be obtained from different parts of the date palm, such as the midrib, spadix stems, leaflets, and mesh as illustrated in figure I.6. These various components of the date palm yield fibers are considered as byproducts. The midrib, located at the base of the date leaf, forms a fibrous covering around the palm and is connected to the trunk. The spadix stem represents the fruit-bearing branches of the date palm. The long, slender segments that make up the fronds of the date palm are known as leaflets, while the date palm mesh is a fibrous layer found on the tree's surface, such as around the trunk or near the base of the leaves. This layer is sometimes referred to as the tree surface, sheath, or coir.[24]

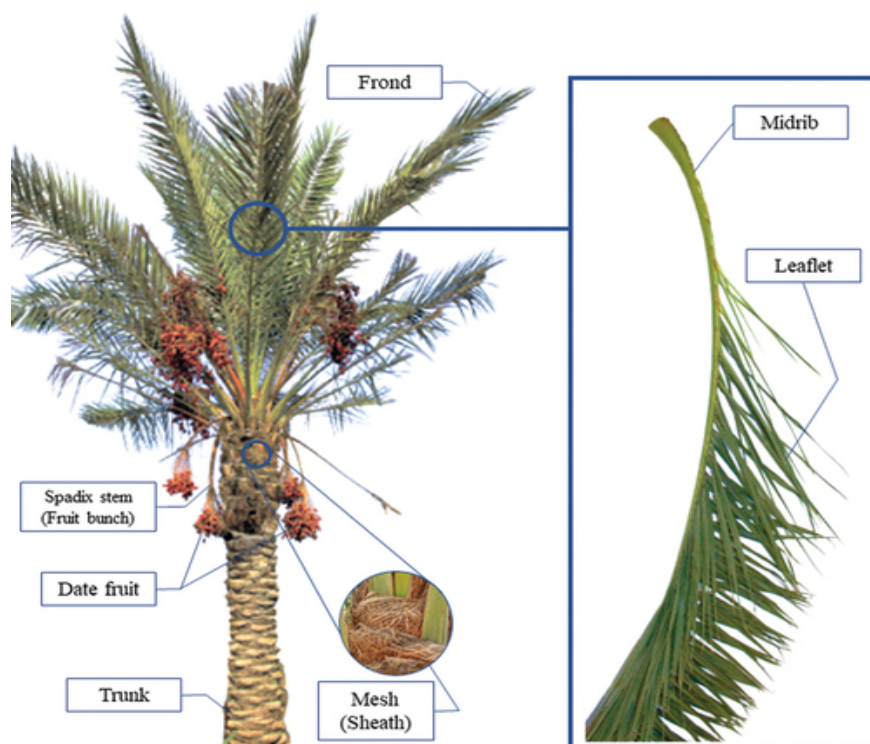


Figure I.6. *Date palm tree parts [25].*

I.5.1.1. Midrib

The midribs, often called the rachis or petiole, are surrounded by numerous leaflets. These midribs consist of strong fibers and vascular bundles, which have been studied for their potential use in various industrial applications, such as fiber extraction for textiles, composite materials, and other eco-friendly products. The extraction of midrib fibers can be done through mechanical or chemical methods, resulting in fibers with different diameters, lengths, densities, and aspect ratios. Mechanical processing yields midrib fibers with larger diameters, greater lengths, and higher aspect ratios. Research has shown that the outer layer of the midrib is stronger than the inner layer, as indicated by their ultimate tensile strength and specific ultimate tensile strength. This outer layer is utilized as reinforcement in the creation of bio-composites. Midrib fibers are available in three types: xylem, arch (central), and structural, and they are classified as leaf fibers. Besides supporting the leaf, these fibers contain vascular tissue that facilitates the transport of nutrients and water throughout the entire date palm tree. The midrib plays a vital role in maintaining the leaf's structure

and function through processes like transpiration and photosynthesis [25]. Various date palm tree parts are presented in figure I.7.



Figure I.7. Various date palm tree parts (a) midrib, (b) spadix stems, (c) leaflets, (d) mesh [25].

I.5.1.2. Spadix stem

The spadix stem (figure I.8) is a crucial part of the date palm's reproductive system, resembling a stalk that supports the flowers which later develop into fruits. Emerging from the base of the fronds, the spadix stalk can bear either male or female flowers, depending on the type of tree. While its primary role is in reproduction, spadix stems also hold potential for other applications, particularly in materials science and industry. The physical properties of spadix stem fibers vary based on the extraction method used. Compared to mechanical and biological methods, fibers obtained through chemical extraction are longer, wider, and possess a higher aspect ratio. These traits are indicative of the physical characteristics of the date palm tree's spadix stem. The fibrous nature of the spadix makes it an attractive choice for creating bio-composite materials. By combining spadix fibers with resins, lightweight and durable composites are produced, which are used in the furniture, automotive, and construction sectors [25].

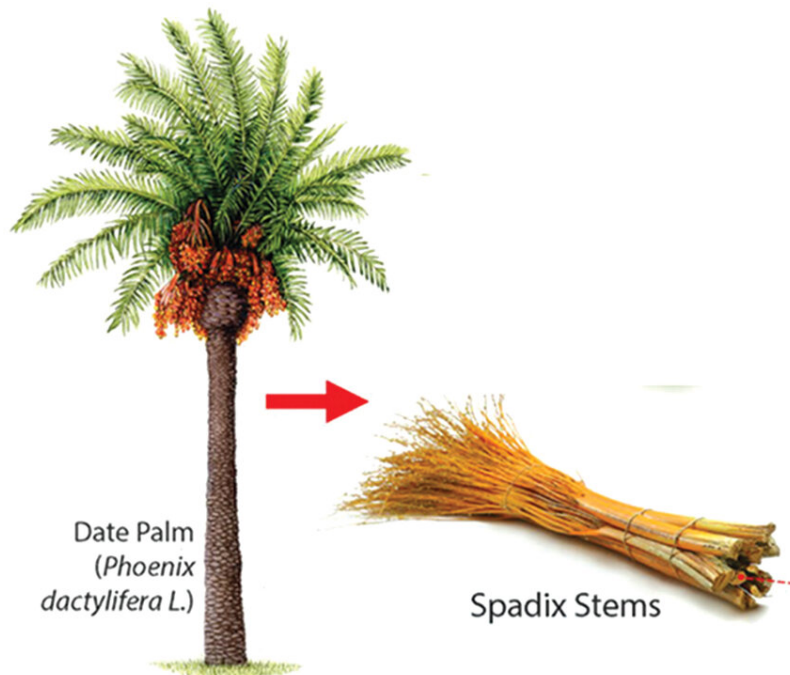


Figure I.8. Spadix stem from date palm tree [25].

I.5.1.3. Leaflets

The date palm tree is among the many plants that feature compound leaves, where each leaf consists of leaflets attached to a central stalk called the midrib. The leaflets of the date palm are arranged on the rachis or midrib in a feather-like pattern on both sides. These leaflets are the individual segments of a complex leaf, sometimes described as the date palm plant's slender, elongated leaf fibers [25].

I.5.1.4. Mesh

Another fiber, known as mesh, is obtained from the trunk of the date palm tree. When freshly harvested, the mesh fiber forms the outermost layer of the date palm trunk, resembling a woven mat of interlaced strands of varying sizes. Due to its sheet-like form, mesh fiber is easier to extract compared to other types of date palm fibers [25].

I.5.2. Processing and Treatment Methods of Date Palm Fibers

Integrating plant fibers into construction materials offers numerous benefits, including excellent wear resistance, effective thermal and electrical insulation, low density, and superior adhesion compared to industrial fibers. Nonetheless, a significant drawback of these fibers is their tendency to absorb water and moisture, a challenge that current research aims to address through pre-treatment methods. Various chemical and physical treatments are employed to enhance the durability of plant fibers, such as mercerization, acetylation, acylation, and silane treatment. These processes involve immersing the fibers in chemical solutions to alter their chemical bonds, reducing their sensitivity to stabilizers like cement or lime. In this study, three physical treatment techniques were selected from the literature, along with two additional treatments tailored to the raw earth material used in this research

I.5.2.1. Hornification Treatment

Hornification is an irreversible process that occurs when cellulosic fibers undergo drying-wetting cycles, preventing the formation of hydrogen bonds that can shear the fibers. This involves placing the fibers in an oven at 60°C for 7 hours, followed by immersion in water for 17 hours.

I.5.2.2. Water Immersion Treatment

This method entails fully submerging the fibers in water for 24 hours to prevent them from absorbing mixing water. Afterward, the fibers are left at ambient temperature for about fifteen minutes to remove excess surface water before being mixed with mortar. [26]

I.6. DPF in mortar and concrete matrix

I.6.1. Influence of DPF on Mortar and Concrete

I.6.1.1. Moisture buffer (Hygric properties)

Hygroscopic materials in building envelopes play a significant role in enhancing both the energy efficiency of buildings and the quality of indoor air. These materials have the capability to stabilize indoor relative humidity fluctuations, ensuring occupant comfort and reducing energy usage. Incorporating 15 wt.% of DPF into cement mortar results in composites that can meet both thermal and mechanical requirements, serving as either structural or insulating materials. Porosity (P) is a

crucial physical characteristic that can influence the hygric and thermal properties of building materials. Both total P and apparent open P are commonly employed to assess the void content in building materials. It can be inferred that utilizing DPF concrete in construction offers benefits for moisture management and helps minimize interstitial condensation in environments with high relative humidity [23].

I.6.1.2. Thermal properties

Thermal insulation materials are crucial for enhancing a building's energy efficiency. Residues from date palm trees are a noteworthy source of NF due to their renewable nature and abundant availability. Various other NF, such as hemp, rice husks, and other plant-based fibers, have also been explored by researchers. Mortars incorporating these fibers demonstrate good durability and are already employed in insulation and coating applications. Benmansour et al. investigated a novel composite material using DPF, discovering that adding an appropriate amount of DPF to mortar results in a composite with excellent mechanical and thermal properties, suitable for use as thermal insulation in buildings. The thermal conductivity findings indicate significant thermal insulation capability in a dry state. It is also important to mention that leaflets and mesh fibers are the most commonly used types in cement mortar. In conclusion, incorporating DPFs in cement mortars enhances thermal, acoustic, and mechanical properties, along with improved durability. Previous studies have shown that concrete with DPF effectively regulates heat, exhibits good moisture absorption behavior, and possesses high thermal insulation capacity. However, using DPF in cement composites involves a trade-off, as it leads to reduced workability and CS. [23]

I.6.1.3. Shrinkage

Drying shrinkage is a significant issue that contributes to the degradation of concrete structures. This problem arises from variations in relative humidity and ambient temperature, which alter the properties of hardened concrete, impacting its mechanical and drying shrinkage characteristics. The use of DPF has shown a comparable effect to shrinkage-reducing admixtures in minimizing early age (EA) drying shrinkage and the risk of cracking in self-compacting concrete (SCC). Even a small amount of DPF can effectively reduce cracking and EA drying shrinkage in hot and dry conditions. Specifically, a 0.1% addition of DPF with a length of 2 cm can reduce EA drying shrinkage by 50% compared to plain SCC specimens. However, there is a slight reduction in the

CS of SCC when DPF is added. The inclusion of small quantities of DPF does not significantly impact the physical, mechanical, and hardened properties of self-compacting concrete [23].

I.6.1.4. Sound Absorption

Similar to other physical characteristics, sound absorption is a focus of various researchers. Belakroum et al. discovered that incorporating DPF enhances absorption capacity, as indicated by the measured sound absorption coefficient, which is 0.65 for medium frequencies and 0.55 for high frequencies with 50% fiber content. [23]

Table 3 presents a comparison of the elongation at break, modulus of elasticity, and tensile strength values for various parts of DPF against some NF. The mechanical properties of DPF vary slightly depending on the part; for instance, surface fibers exhibit the highest tensile strength at 170 MPa and a modulus of elasticity of 4.74 GPa, while pedicel fibers show the lowest tensile strength at 86 MPa and a modulus of elasticity of 3.00 GPa. The petiole DPF has the smallest elongation value at 0.95%, whereas the surface DPF has the largest at 16%. These differences in properties are attributed to their physical and chemical characteristics [23].

Table I.2. Mechanical characteristics of date palm fiber compared with some NF [23].

Fiber Type	Tensile Strength (MPa)	Elongation (%)	Modulus of Elasticity(GPa)
Surface DPF	170 ± 40	16 ± 3	4.74 ± 2
Petiole DPF	90 ± 8.87	0.95 ± 0.42	7.00 ± 2.00
Leaflets DPF	100.12 ± 43.87	2.68 ± 0.49	4.00 ± 1.33
Pedicels DPF	86 ± 5.00	2.37 ± 0.15	3.00 ± 1.00
Fibrillium DPF	90 ± 30.70	4.59 ± 0.90	3.66 ± 2.33
Cotton	287 – 597	3.0 – 10.0	5.5 – 12.6
Sisal	137 – 577	—	15.2 – 34.0
Flax	345 – 1500	1.2 – 3.2	27.6 – 80
Jute	393 – 800	1.5 – 1.8	10 – 30

I.6.1.5. Mechanical Properties and Cracking

To enhance FS after cracking during water curing and hot dry curing, using a fiber percentage of 2-3% and a length of 15–60 mm is effective. The addition of fiber also improved toughness coefficients. However, this fiber percentage results in a decrease in CS and initial cracking. For each type of concrete, the initial cracking strength diminishes in hot-dry climates. Cracks and the overall degree of voids decrease in water curing but increase in hot-dry conditions. The use of DPF boosts the material's ability to absorb water vapor due to its porous structure. This behavior is effective in high relative humidity environments, allowing the materials to release the absorbed water in dry conditions, thus acting as a hygric regulator. The CS tests showed that incorporating DPF led to a reduction in resistance limit [23]

Lahouioui et al. examined the effects of incorporating both treated and untreated DPF into concrete at levels of 2.5%, 10%, and 20%. They found that adding these fibers, whether treated or untreated, led to a decrease in both CS and thermal conductivity of the composite materials. Specifically, the CS dropped from 20.69 MPa in the reference material to 14.85 MPa with treated DPF and to 4.79% with DPF materials after a 2.5% inclusion. Additionally, WA increased as the fiber content rose. Despite this, treated DPFs were still considered suitable for enhancing strength and thermal insulation in concrete compared to untreated fibers. Alward et al. further explored the impact of treated and nitrated DPF on concrete properties, using fibers 30 mm in length and replacing them in concrete at levels of 0.4%, 0.8%, and 1%. Their study showed a 9.37% and 13.65% increase in CS with 1% untreated leaf (UL) and 0.8% treated leaf (TL), respectively. Although the tensile strength of the material decreased, the FS improved by 3.3% and 7% with a 1% replacement using both treated and untreated materials. The durability of concrete was also enhanced in terms of reducing crack and void formation due to the fibers. The presence of more DPF in the mortar increased its ductility, primarily due to the interconnectivity of DPF and the transmission of stresses between fracture surfaces (Benaimeche et al.). Numerous studies have reported the use of DPF as a natural fiber in cement-based products like mortar and concrete. Ozerkan et al. added DPF to mortar at dosages of 0%, 0.5%, 1%, and 2%. When up to 1% DPF was added, the initial and final setting times of the mortar, as well as its drying shrinkage, increased. However, these factors decreased when 2% fiber was added. Moreover, DPF increased the mortar's WA rate, with 0.5% being the optimal addition. They observed improvements in mechanical properties and

resistance to sulfate attack with up to 1% DPF, attributing these enhancements to better compaction between the fibers and cement matrix, resulting in a homogeneous mix. It was found that a low DPF content of up to 0.5% could be the most effective amount for enhancing the mechanical properties and durability of concrete. Excessive fiber content can reduce the feasibility or efficiency of concrete due to poor dispersion and increased P, leading to higher WA because of the fragile nature of fibers. However, the ductility of DPF-reinforced concrete appeared to be significantly enhanced even with high fiber content. Additionally, the length of fibers played a crucial role in maintaining concrete efficacy. Short DPF, up to 50 mm, were more beneficial for sustaining the compressive, tensile, and FS of concrete, while longer fibers resulted in poor durability [27].

I.6.2. Sustainability Index

The sustainability of different types of concrete is assessed using a sustainability index. To this end, research has been conducted to evaluate the carbon footprint, energy usage, renewability, durability, and water consumption of various concretes, including those reinforced with date palm and other biobased materials. The quantitative data on carbon emissions and energy consumption is derived from life cycle assessments of different concrete mixtures. On the sustainability index, a score of 1 represents the least sustainable material, while a score of 10 indicates the most sustainable. DPF stands out with an index score of 8, the highest among all concretes, due to its low energy consumption, minimal carbon emissions, and excellent durability and recyclability. However, it also has some performance drawbacks, such as increased P and WA, which can reduce the overall durability of the concrete (See Figure I.9) [27].

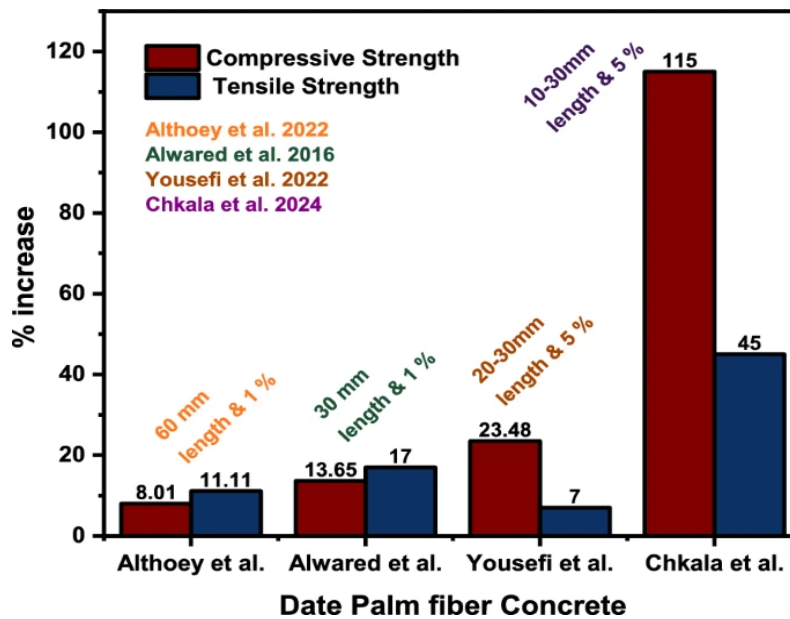


Figure I.9. Effect of DPF content and length on CS and TS [27]

I.7. Conclusion

This chapter has summarized fiber-reinforced cementitious materials, focusing on the role of natural fibers in promoting sustainable construction. DPF, in particular, have gained significant interest because due to their abundance, low cost, renewable nature and can enhance the toughness, crack resistance, and ductility of cement-based materials. Utilizing these fibers also provides an eco-friendly approach to managing agricultural waste and lessening the environmental impact of construction materials. DPF has shown encouraging potential as a reinforcement in both mortar and concrete. Nevertheless, this study is centered on mortar with the aim of assessing its suitability for the development of more sustainable cementitious materials.

CHAPTER II:
EFFECT OF WASTE TIRE
RUBBER ON THE PROPERTIES
OF MORTAR AND CONCRETE

Chapter II: Effect of waste tire rubber on the properties of mortar and concrete

II.1. Introduction

As the automotive and transportation sectors continue to advance, the volume of discarded tires is rising each year. It is estimated that worldwide tire production surpasses 3 billion units annually. By 2030, the number of waste tires is expected to reach 5 billion units. This type of solid waste (See figure II.1), often referred to as "black pollution," is challenging to break down in the natural environment. Presently, primary disposal methods like landfilling and incineration produce significant amounts of toxic and hazardous substances, posing a grave threat to the ecological environment [28]. Additionally, the incineration process demands a considerable amount of fossil fuels, further intensifying the depletion of energy resources. In this scenario, the recovery and reuse of waste tires present a promising solution to these issues. By transforming waste tires into high-value materials (such as reclaimed rubber and CR) or energy products (like pyrolysis oil and pyrolysis gas), it is possible to reduce reliance on virgin resources and significantly decrease carbon emissions, thereby supporting global carbon neutrality objectives [29].



Figure II.1.waste tire rubber in the environment [30]

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II.2. Waste tire rubber

Used tires can be classified as bulky special industrial waste due to their large volume, which prevents them from being collected under the same conditions as household and similar waste [31,32]. In 2002, according to the national inventory of special industrial waste, the production of such waste was 325,000 tons per year, with a stockpile amounting to 2,008,500 tons.

Used tires represent the largest share of rubber waste (more than 85%) [33], with approximately 26,000 tons of used tires generated annually in Algeria [34]. Moreover, it is estimated that nearly 1.2 billion new tires are produced worldwide each year and discarded at the end of their service life [35], of which more than 50% are disposed of without further use [36,37]. In general, the main sources of rubber waste are tire management and usage. For example, the United Kingdom produces about 4.3×10^8 kg of used tires annually [38,39], while Japan generates 2.1×10^8 kg of tire wear debris [40].

A tire is defined as a continuous pneumatic covering made of natural rubber, synthetic rubber, or a combination of both, surrounding a wheel, whether new, used, or retreaded [41]. Due to their non-biodegradable nature, waste tires are associated with negative impacts on public health and the environment when landfilled or improperly disposed of without treatment. They occupy large areas and cause pollution problems, including groundwater contamination, air pollution, and soil contamination. However, one positive aspect of used tires is that they retain many valuable properties, making them a potential raw material [42], such as:

- ✓ Elasticity
- ✓ Structural strength
- ✓ Drainage capacity
- ✓ Calorific value
- ✓ High carbon content
- ✓ Durability
- ✓ Damping capacity

The different management strategies for waste tires are highlighted in Figure II.2.

Chapter II: Effect of waste tire rubber on the properties of mortar and concrete

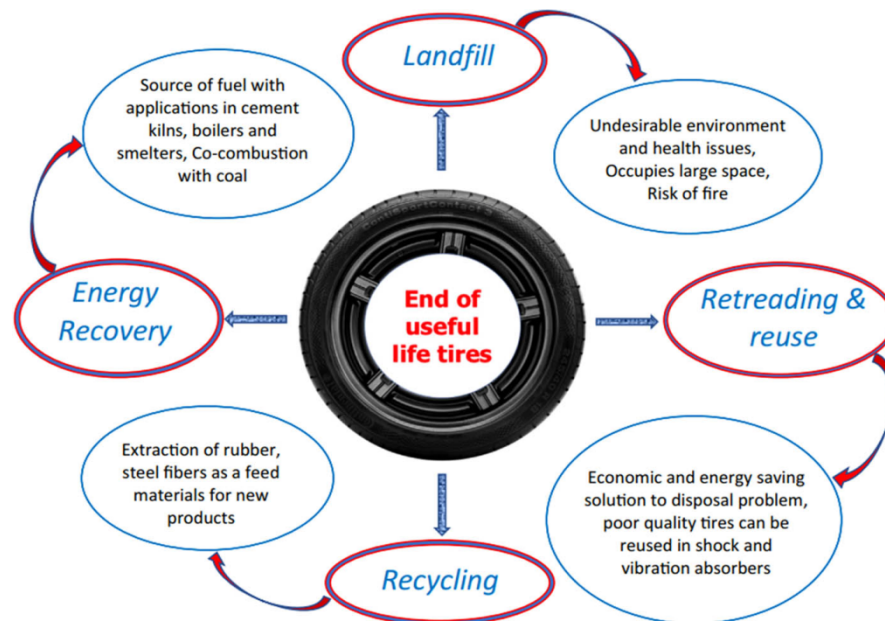


Figure II.2. Management strategies for waste tires at the end of useful life [30]

In recent years, the construction sector has been tackling the challenge of integrating sustainability into its production processes by seeking more eco-friendly raw materials or utilizing solid waste materials as concrete aggregates. One potential solution for waste tire rubber (WTR) is to incorporate it into cement concrete, substituting some of the NA. This approach could be environmentally beneficial as it aids in disposing of waste tires and curbing environmental pollution. Additionally, it helps lower carbon dioxide emissions by preventing tire fires. Economically, this method is viable since it allows for the conservation of some expensive NA [43].

II.3. Main Components of a Tire

Polymers can be divided into two main groups: thermoplastics and thermosetting materials. Rubber derived from waste tires is classified as a thermosetting material, which cannot be softened or remolded by heating due to the cross-linked structure of rubber and the presence of stabilizers and other additives. This makes it difficult to reuse waste tires in the manufacturing of new tires. [44]

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WTR is primarily sourced from car and truck tires. While the rubber content in car and truck tires varies across different countries, the proportion of natural and synthetic rubber, which is around 40–45%, remains consistent. Car tires are manufactured in greater numbers than truck tires, so research on WTR recycling predominantly uses rubber from car tires. Previous studies have utilized six types of rubber for recycling WTR into building materials, with the term name, particle size range, and type of natural aggregate replacement specified in. The production of rubber particles is mainly accomplished through two methods: mechanical grinding and cryogenic grinding, followed by screening of rubber particles derived from waste tires to substitute fine or coarse natural aggregate. Tire rubber is processed into rubber powder or rubber ash to replace a portion of cement, while rubber fiber and CR are used to partially replace fine aggregate [45]. The basic material composition tires are presented in Table II.1. [44]

Table II.1. *Typical Constituent Materials of Tires [44]*

Composition	Car Tire (%)	Truck Tire (%)
Natural rubber	14	27
Synthetic rubber	27	14
Fillers (Carbon black, silica, Carbon, chalk, etc.)	30	30
Reinforcing materials (steel, polyester, rayon, nylon)	15	15
Vulcanization chemicals (sulfur, zinc oxide, etc.)	6	6
Plasticizers (oils and resins)	16	16
Anti-aging agents and other chemicals	2	2

II.4. Methods and Potential Uses of Waste Tires Worldwide and in Algeria

At the end of the 1950s, only about one-fifth of the rubber hydrocarbons used in the United States and Europe were recovered. By the mid-1980s, less than 1% of the global polymer consumption was in the form of recovered material. At the beginning of the 21st century, half of the consumed rubber was in the form of recovered material. It is expected that in the 21st century, most rubber waste will be recycled as recovered material due to the daily increase in environmental awareness [46]. Waste tires are recycled or managed through landfilling, incineration, or other methods

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depending on how each country adopts these practices, particularly in developed countries that are highly concerned with tire production. Unfortunately, in Algeria, there is a lack of awareness regarding the principles of the 3Rs (Reduce, Reuse, Recycle) [47].

There are two types of tire waste:

- **Used tires that are non-reusable** and must be disposed of or redirected for another use.
- **Used tires that are reusable**, which must be retreaded or sold as second-hand tires [48].

II.4.1. Landfilling Method

Landfilling is one of the earliest methods for disposing of used tires. In 1977, approximately 70% of recovered rubber, mainly in the form of tires, was sent to landfills [49]. Moreover, in 1994, 75 to 80% of used tires in the United States were landfilled. However, landfilling whole tires has been banned in most operations due to their large volume and tendency to float to the surface over time [50].

Particularly in Algeria, the accumulation of used tires in open dumps is still common. To address this, since 2001, the Algerian government has opted to manage municipal solid waste (including used tires) through landfilling, which involves underground storage of waste. It launched an ambitious program of landfill sites across the country, with 65 sites identified: 16 completed, 28 under construction, and 21 in the planning phase [51]. By the end of 2007, this number had increased following the results of pilot projects, notably the Ouled Fayet project, reaching 80 projects: 20 completed, 34 under construction, 26 in planning, and 15 new projects. The concerned wilayas are Skikda, El-Taref, Annaba, Guelma, Souk Ahras, Batna, Tébessa, Médéa, Tizi-Ouzou, Sétif, Biskra, Algiers, M'Sila, Ouargla, Blida, Djelfa, Jijel, Béjaïa, and Chlef [52].

The main advantages of this technology are:

- ✓ It provides a universal solution that ensures ultimate waste disposal
- ✓ It has relatively low costs and is easier to implement compared to other waste management technologies.

However, this method also has several drawbacks, including the need for large land areas for landfills and pollution issues, such as contamination of groundwater, air, and soil. Therefore, we

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can conclude that this method is the least desirable due to environmental problems and has no future potential.

II.4.2. Energy Recovery Method

Energy recovery primarily involves using waste tires as an alternative fuel for energy production; this energy can provide heat or electricity [53]. This technology was introduced in the United Kingdom in 1865 with the British Destructor and later exported to the United States and Europe [54]. Additionally, incineration has been widely applied in many developed countries, particularly those with limited landfill space, such as Japan and several European countries. For example, China has multiple municipal waste incineration plants, including the one in Shanghai, which is considered the largest in the country. This plant is designed to process 3,000 tons of waste daily and generate approximately 270 million kilowatt-hours of electricity per year. Moreover, this includes the use of waste tires as fuel in cement kilns [55,56]. This process is still in use, but it creates a new air pollution problem and represents a low-value recovery process for waste rubber.

In Algeria, incineration is applied only for hospital waste, while it is not used for waste tires, as natural gas is preferred in the cement industry.

II.4.3. Material Recovery Method

Material recovery is the concept of reducing the number of waste tires accumulating in landfills by using them as raw materials. According to ASTM D6270-08, 2014 [57], WTR can be classified as particulate rubber (bale rubber, granulated rubber, ground rubber, and powdered rubber), coarse shredding, tire-derived aggregate, tire shredding, and whole tires. Tables II.2 and II.3 summarize the established nomenclature and sizes of WTR according to ASTM D6270-08, 2014 [57]. The various sizes of tire particles used in several applications are illustrated in Figure II.3.

Table II.2. ASTM D-6270 Classification of Recycled Waste Tires by Size [57].

Classification	Lower Limit (mm)	Upper Limit (mm)
Shredded Tire	Not specified	–
Coarse Shredding	50 × 50 × 50	762 × 50 × 100

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Tire-Derived Aggregate	12	305
Tire Flakes	50	305
Tire Chips	12	50
Granulated Rubber	0.425	12
Ground Rubber	–	< 0.425
Bale Rubber	–	< 0.425

Table II.3. Nomenclature and Dimensions of Waste Tire Rubber According to ASTM D6270 [57]

Granulated Rubber	Non-spherical	Below 425 μm (40 mesh) to 12 mm
Ground Rubber	Non-spherical	Below 425 μm (40 mesh) to 2 mm
Powdered Rubber	Non-spherical	Below 425 μm (40 mesh)
Coarse Flakes	–	Over 50 mm $\times$ 50 mm $\times$ 50 mm but less than 762 mm $\times$ 50 mm $\times$ 100 mm
Tire Chips	Basic geometric shape	Between 12 and 50 mm
Tire-Derived Aggregate	Basic geometric shape	Between 12 and 305 mm
Tire Flakes	Basic geometric shape	Between 50 and 305 mm

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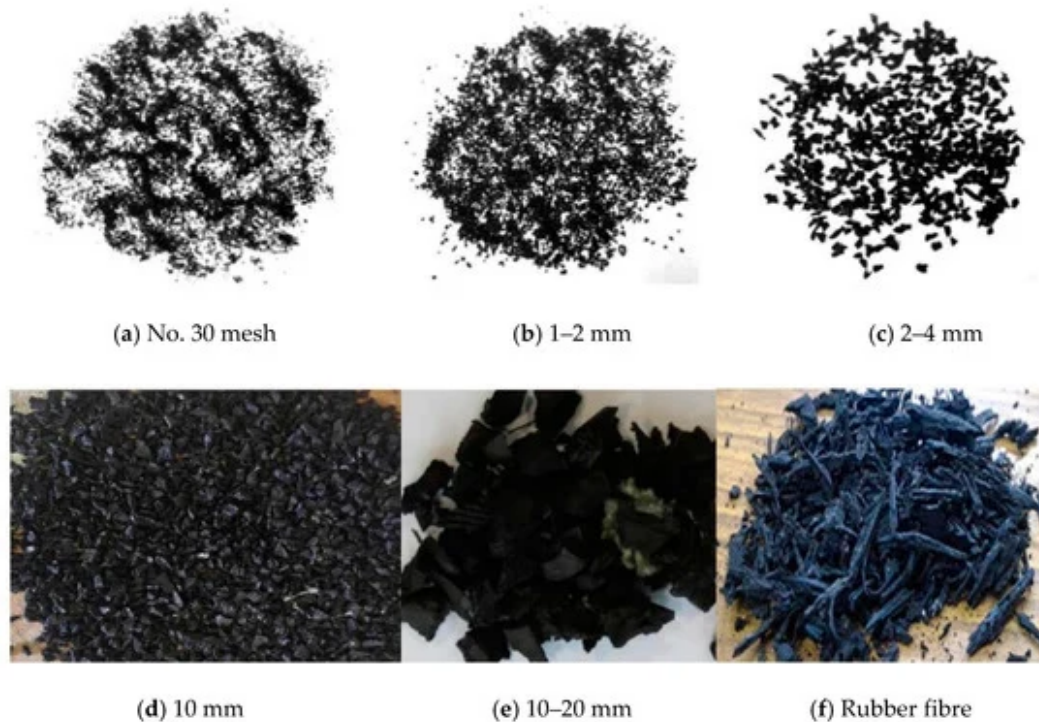


Figure II.3. Various sizes of tire particles [45].

II.5. Recovery of end-of-life tires (ELTs) as Tire-Derived Aggregate for Mortars/Concrete Applications

Disposing of tires is challenging due to their slow degradation, substantial weight, and resistance to biodegradation. Utilizing CR is crucial for safeguarding the environment. This material finds applications in the automotive sector, sports venues, road construction using asphalt, shock absorption, and building structures such as industrial and bathroom flooring, floor tiles, and foundation waterproofing. Recent research indicates that the process of repurposing tires, from collection to disposal, presents minimal health and environmental hazards, though further investigation is advised. In civil engineering, there is a growing trend of using CR as a replacement for aggregates in cement-based mortar and concrete. Experiments have shown that substituting fine aggregates with rubber particles of varying sizes impacts workability and water permeability more than it does fresh density and concrete strength. Another study found that rubberized concrete (RuC) exhibited greater flexibility but reduced compressive and tensile splitting strength, which

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was seen as a beneficial outcome. Similarly, low levels of rubber substitution (2–12%) led to notable decreases in CS and elastic modulus. Nonetheless, experimental findings demonstrated that replacing aggregates with rubber particles enhanced the deformability of concrete. The size and content of the rubber positively influenced its resistance to abrasion and freeze-thaw cycles. Studies on the use of CR in concrete are more prevalent than those focusing on its application in mortar. For example, da Silva et al. discovered that recycled tire rubber particles increased the micropore volume in cementitious paste [58].

Incorporating fine CR into composite materials helps mitigate the environmental issues associated with discarded tires and reduces the overall weight of structural mortar, which can otherwise be quite heavy. Utilizing rubber and decreasing the NS content in mortar offer ecological advantages. Opting for CR as a substitute for NS represents a sustainable design breakthrough. Achieving densities around 1500 kg/m^3 is possible with a complete replacement of NS by CR. While some research has explored the fracture energy of CR concrete, further investigation is necessary for mortars containing significant amounts of rubber. In summary, based on numerous studies, the use of RuC is often driven by its low density, excellent sound absorption, strong resistance to chemical degradation, freeze-thaw cycles, and chloride ion penetration, as well as its improved damping capacity, impact resistance, and toughness [58].

Researchers have explored the impact of incorporating waste rubber into cement-based materials. When compared to conventional cementitious materials, those enhanced with rubber offer several benefits, including reduced density, excellent freeze-thaw durability, enhanced ductility and impact resistance, superior chloride resistance, and effective thermal insulation. Additionally, rubberized cementitious materials (RCM) seem to be more appropriate for use in pavement engineering than their traditional counterparts. In 2003, the Arizona Department of Transportation constructed the first RuC pavement, which remains operational to this day. Furthermore, researchers have combined waste rubber powder with sand and cement to create railway sleepers. These sleepers provide advantages such as being lightweight and having good impact resistance, while also minimizing train noise and vibration. Consequently, RCM hold significant potential in the realm of civil engineering [59].

RuC is also beneficial for applications such as reducing vibrations in structures, industrial flooring, road surfaces, retaining walls, and bridge walkways and decks. Additionally, it can be employed

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in hydraulic structures like tunnels and dam spillways, where high resistance to abrasion is crucial. It is also suitable for thermal and sound insulation systems, running tracks, roadside barriers that require high impact energy absorption, parking lots, and areas in cold climates that experience significant freeze-thaw cycles [60].

CR plays a vital role in promoting sustainable construction by serving as a low-carbon substitute that can greatly diminish the carbon footprint linked to conventional building materials. Utilizing CR in road infrastructure materials supports environmental objectives by recycling waste, minimizing landfill usage, and decreasing greenhouse gas emissions tied to the extraction and production of raw aggregates. This environmentally conscious method aligns with circular economy principles and fosters sustainable growth in the infrastructure sector. Employing CR as a stabilizer in various road infrastructure materials offers notable benefits not only from economic and environmental viewpoints but also regarding the thermo-hydro-mechanical properties of the stabilized materials. Previous research suggests that, with accurate dosing, CR can shift from being a challenge to a solution in numerous applications [61]. The different stages involved in processing waste tires for concrete production are shown in Figure II.4.

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FigureII.4. Flow chart of waste rubber tires from waste into concrete [62].

II.5.1. CR in Asphalt Mixtures

Asphalt mixtures are created by combining asphalt binder with coarse aggregates like gravel, fine aggregates such as sand, mineral fillers like Portland cement or hydrated lime, and sometimes additives, all at high temperatures, reaching approximately 160~180 °C. The temperature at which mixing occurs is a crucial factor in designing Rubberized Asphalt Mixtures (RAMs). RAMs typically require higher temperatures than conventional asphalt mixtures, by about 30~50 °C, which influences their mix design as well as their mechanical properties and sustainability. It's noteworthy that CR can be added to various asphalt mixture types, including hot, warm, half-warm, and cold mixtures. Regardless of the method used to incorporate CR, it enhances the asphalt binder's ability to endure recoverable strains, thereby decreasing the brittleness of the asphalt mixtures over time [61].

II.5.2. CR in Railways

Traditional railway pavement materials, like granular ballast and standard asphalt layers, often fall short in meeting the durability and vibration-damping needs of modern railway systems. To address this, CR-modified asphalt (CRMA) has emerged as a sustainable and high-performance alternative. CRMA is created by integrating WTR into asphalt mixtures, resulting in increased

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flexibility, enhanced fatigue resistance, and superior vibration reduction, making it a compelling option for railway use. Recent research suggests that CRMA significantly extends the fatigue life of railway pavements, especially in high-speed railway systems where conventional asphalt layers degrade under repeated loading. Moreover, railway infrastructure is challenged by rising axle loads, traffic volumes, and train speeds, prompting the increased adoption of asphalt stabilized ballast (ASB), asphalt underlayment (AUL), and asphalt slab track bed (ASTB) for maintenance and repair purposes. Rubber-modified asphalt (RMA) offers a promising solution for high-speed rail by providing greater stiffness and damping properties compared to traditional materials, thereby significantly minimizing vibrations. Additionally, innovative uses of WTR in railway substructures are being investigated to lower energy demands and enhance the life cycle performance of track systems. Incorporating rubber into asphalt improves its dynamic performance, fatigue resistance, and crack resistance under heavy cyclic loads, aligning with sustainability goals [63].

The different processing stages used in the transformation of waste tires into CR for asphalt applications are illustrated in Figure II.5.



Figure II.5. Processing of waste tires into CR for asphalt application[63].

Although RCM offers several benefits, its use in civil engineering remains limited. This is mainly because of two factors: firstly, civil engineering professionals lack a thorough understanding of

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the benefits of RCM due to insufficient knowledge; secondly, incorporating waste rubber diminishes the compressive and flexural properties of rubberized mortar, hindering its progress. However, for engineering applications with minimal compressive and FS requirements, such as plastering mortar, rubberized mortar presents a significant advantage [58].

II.6. Environmental Benefits of incorporating CR in concrete

The environmental consequences of incorporating waste automobile tires into concrete extend well beyond the emission of greenhouse gases. This practice can decrease the reliance on NS and mitigate the risk of natural disasters. Historically, the unchecked extraction of NS and gravel on a large scale has been linked to landslides, riverbed alterations, and the degradation of the surrounding ecological environment. By substituting NA with CR derived from waste automobile tires, the amount of NS and gravel used in concrete can be reduced, which in turn lowers the risks of geological disasters and floods in river areas, thereby postponing the depletion of NS and gravel resources. Additionally, it reduces the air pollution risk associated with burning tires. Incineration, although the cheapest and earliest method for disposing of waste automobile rubber tires, is also the most environmentally damaging. The burning process releases significant amounts of CO₂ and SO₂ into the atmosphere, greatly increasing the concentration of PM₁₀ in the air. These toxic gases are known to be carcinogenic or mutagenic, posing serious health risks to humans and contributing to air pollution.

Enhancing the natural environment and minimizing the consumption of land resources due to waste tire disposal: Besides incineration, other methods for disposing of waste automobile tires include piling and landfill dumping. Accumulating waste tires can lead to mosquito breeding and heighten fire risks. Given that rubber is notoriously resistant to degradation, even over a century, rubber tires contribute to soil and groundwater pollution near landfill areas. As a result, the practices of piling and landfill dumping of rubber tires consume substantial land areas, thus depleting land resources [64].

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II.7. Effect of Rubber Aggregates on the Characteristics of Fresh and Hardened Concrete

The addition of CR to concrete has a notable impact on its fresh properties, particularly influencing workability, water requirements, and air content. These effects are closely linked to the particle size, levels of replacement, and the intrinsic characteristics of rubber, such as its water-repellent nature and uneven surface texture. Although RuC provides environmental and functional advantages, the challenges related to its fresh properties require strategies for enhancement, including the incorporation of supplementary cementitious materials (SCMs) and chemical additives [30].

II.7.1. Fresh state

II.7.1.1. Workability

One of the primary impacts of CR on fresh concrete is a decrease in workability. Rubber particles, being hydrophobic, struggle to absorb water, leading to uneven distribution within the cement matrix and disrupting the mix's smooth flow. As the CR content rises, the decline in workability becomes more evident. Increasing the rubber proportion in concrete significantly reduces its unit weight. This reduction is due to rubber's naturally low specific gravity compared to traditional aggregates. This phenomenon is linked to the formation of voids and capillaries by rubber particles, which affect the mix's packing density and consistency. Additionally, larger rubber particles worsen this problem because their greater surface area and irregular shapes require more water to maintain flowability [30].

II.7.1.2. Setting time

The penetration resistance test conducted at room temperature (20 °C) was used to determine the total setting time. The experimental findings reveal that as the replacement ratio increases, the setting time decreases. Although the results do not specify an exact threshold for acceptable performance, they provide designers with a range of variations in air content and setting time relative to the replacement ratio, which can be beneficial for practical applications. The air content fluctuates by about 200%, and according to homogenization principles, this level has a significant effect on P, mechanical properties, and thermal properties, as some authors have noted. The

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increase in air content with a higher fraction of CR aggregates is attributed to their non-polar nature and rough surface, which trap air bubbles. Figure II.6 presents the slump test results for various specimens obtained in accordance with NF EN 12350-2 and NF EN 1015-3 standards, showing that workability significantly diminishes when the CR content surpasses 60% [58].

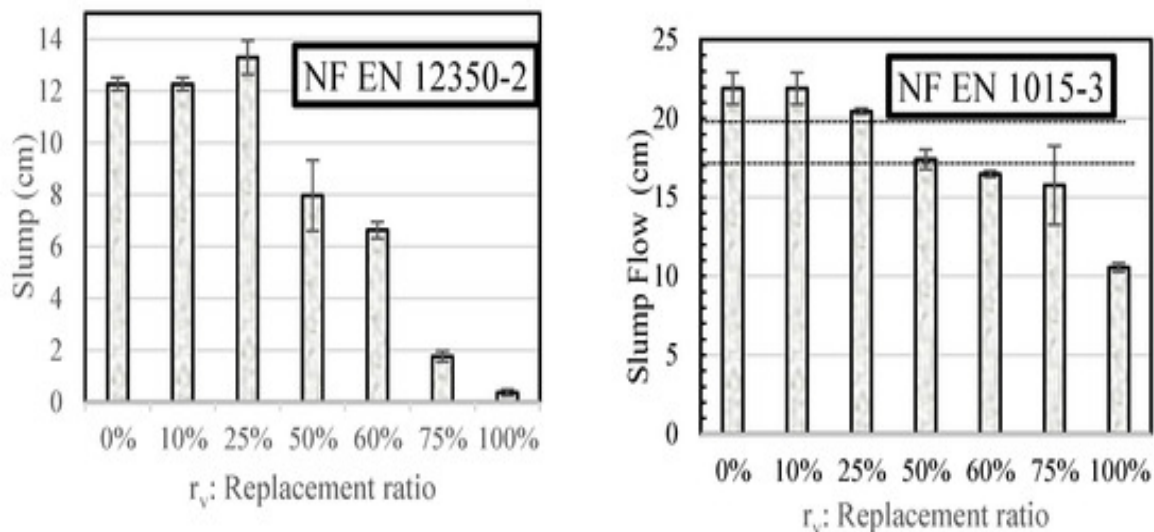


Figure II.6. Workability of the different mixes as a function of CR rates [58]

To sum up, the properties of mortar in its fresh state, including density, workability, air content, and setting time, are essential for its use in construction projects. The observation that air content increases linearly with the amount of CR substitution suggests that CR particles, due to their non-polar characteristics and rough texture, capture air bubbles. This rise in air content can greatly affect the P and mechanical characteristics of the mortar once it hardens. Additionally, the reduction in setting time with increased CR content implies that the mortar may harden more quickly, which could be advantageous for fast-paced construction but might also reduce the available time for application [58].

II.7.2. Hardened state

II.7.2.1. Density and porosity

The density of various hardened mortars was determined by placing the samples under reduced pressure conditions for 4 h and then immersing them in water for 44 h.

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The figure II.7 shows that the mortar density decreased as the ratio of CR to NS increased because rubber has a lower density compared to sand. Likewise, the mortar's P rises as the CR content increases [58].

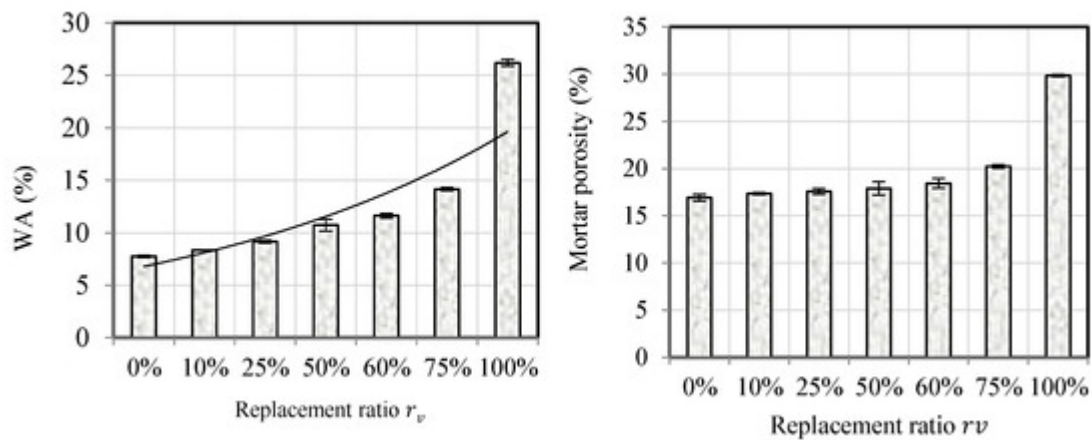


Figure II.7. Water absorption coefficient and P evolution of mortars versus replacement ratio of NS by CR [58].

II.7.2.2. Compressive strength

The figure II.8 shows that the CS consistently increases over time during the curing process, regardless of the replacement ratio. At a 10% CR content, the CS rose from 28.85 MPa at 7 days to 43.68 MPa at 90 days. At a constant age, the CS decreases as the CR replacement ratio increases, as indicated by the figure. Specimens aged 28 days showed strengths of 38.52 MPa, 32.38 MPa, 19.28 MPa, 9.7 MPa, 7.7 MPa, 4.27 MPa, and 2.41 MPa at CR contents of 0%, 10%, 25%, 50%, 60%, 75%, and 100%, respectively. There is a notable decrease in resistance after surpassing 25% [58].

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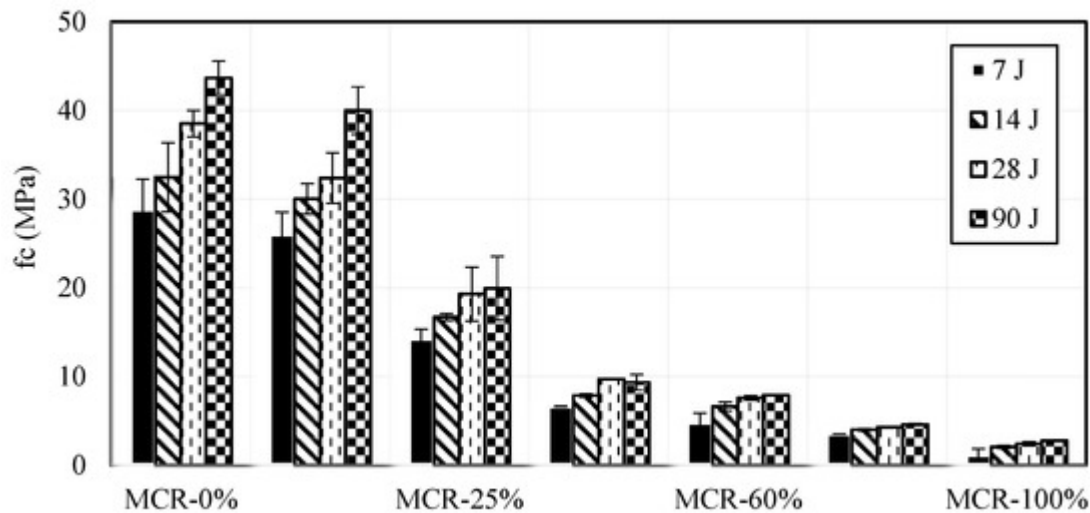


Figure II.8. Effect of CR content on CS[58]

The decrease in CS with increasing CR content is expected due to the lower stiffness and strength of rubber compared to NA. However, mortars with up to 50% CR content still meet the requirements for non-structural repair applications, indicating their potential for use in less demanding structural elements. The established relationships between CS and CR content or density provide a useful guideline for designing mortars with specific strength requirements [58].

II.7.2.3. Flexural and Tensile strength

As for CS, it can be seen that it increases with time irrespective of the replacement ratio. For example, at 25% CR content, FS increased from 3.8 MPa at 7 days to 4.71 MPa at 90 days. However, the figure shows that at a fixed age, FS reduced with the CR replacement ratio. For example, specimens of age 90 days have strengths of 9.3 MPa, 7.45 MPa, 4.71 MPa, 3.5 MPa, 2.75 MPa, 2.15 MPa, and 1.35 MPa when the CR content is 0%, 10%, 25%, 50%, 60%, 75%, and 100%, respectively (See figure II.9). In coherence with the CS tests, the results show a significant drop in resistance, as can be seen beyond 25% [58].

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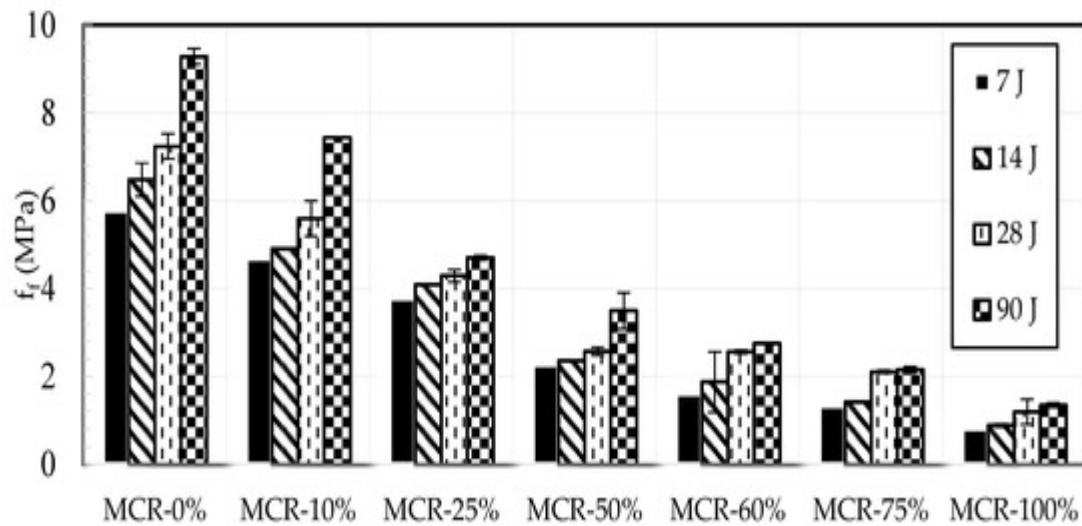


Figure II.9. Effect of CR content on FS during the curing [58].

Similar to CS, flexural and tensile strengths decrease with higher CR content. The significant drop in strength beyond 25% CR suggests a threshold for maintaining adequate MP.

II.7.2.4. Elastic modulus

The correlation between the elastic modulus and CR content mirrors the pattern seen in CS. The modulus follows a trend akin to the resonance frequency, diminishing as CR content rises for a specific curing period. This indicates that a higher CR content in mortars results in greater creep potential, as the material becomes more pliable and less rigid. There are no particular elastic modulus standards for non-structural repair categories. According to these criteria, mortars with up to 25% CR substitution can be utilized as structural repair materials in civil engineering projects. On the other hand, mortars with higher CR substitution levels are better suited for non-structural uses due to their reduced elastic modulus. The decline in the dynamic modulus of elasticity with increased CR content signifies the mortar's decreased stiffness. Although this may restrict its application in structural contexts that demand high stiffness, it also suggests enhanced damping properties, which can be beneficial for minimizing vibrations and noise. The link between the elastic modulus and CR content offers valuable insights for designing mortars with specific stiffness and damping features [58].

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II.7.2.5. Drying and Shrinkage

Considering the effect of CR on P and WA, it is crucial to examine its influence on drying and shrinkage. These factors are significant contributors to damage in concrete structures, particularly when contraction is restricted, leading to tensile stresses. Shrinkage is known to increase with the volume fraction of paste in concrete and decrease with relative humidity. The findings also indicate that shrinkage rose notably when CR content exceeded 60%. This is attributed to the inferior structural performance of rubber particles compared to sand particles; the former being considerably more flexible, providing minimal confinement for mortar, resulting in greater shrinkage. Shrinkage occurs during cement hydration as the hardening phases continue to cure. It significantly affects the overall strength of cement mortar, as it is linked to microcracks that concentrate stresses and may cause failure. The increased drying rate and shrinkage with higher CR content underscore the necessity for careful management of these properties. Although faster drying can be beneficial for reducing construction time, increased shrinkage can lead to cracking and compromised structural integrity [58].

II.7.2.6. Thermal properties

The thermal conductivity of tire CR particles, is consistently at least ten times lower than that of NS. Consequently, as the proportion of "CR" increases, the thermal conductivity of rubberized mortars decreases. This decrease becomes more pronounced as the relative particle size increases [58].

II.7.2.7. Acoustic Properties

Increasing the rubber content in concrete improves sound absorption, particularly in the mid to high-frequency range (500-2000 Hz). Rubber particles enhance the P of the concrete, creating voids that trap and dissipate sound energy, making the material more effective at absorbing sound in this frequency range. Additionally, the elasticity of rubber compared to traditional concrete aggregates contribute to better sound absorption, as it allows the material to more efficiently dissipate the energy of sound waves. However, the data also shows that as the curing time increases, the sound absorption coefficient (SAC) tends to decrease. This is because, as the concrete cures, the matrix hardens and the rubber particles become more integrated into the structure. As the material stabilizes and the concrete continues to hydrate and set, its P decreases

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slightly, leading to a reduction in sound absorption. While the SAC may still remain relatively high, the decreased P and increased stiffness of the material contribute to a slight decline in sound absorption performance over time. The findings of this study are consistent with previous research, such as the work by (Flores Medina et al., 2016), which also found that the sound absorption coefficient of RuC increases with higher rubber content, particularly in the medium and high-frequency ranges. This improvement is likely due to the formation of pores in the concrete by the rubber particles, which increase its P and enhance its sound absorption performance. Additionally, the lower elastic modulus of rubber compared to traditional aggregates results in reduced stiffness, making the concrete more capable of absorbing mid to high-frequency sound waves [65].

II.8. Durability parameters

II.8.1. Water Absorption and Permeability

Permeability plays a crucial role in determining the longevity of concrete, as it has a direct impact on its resistance to freeze-thaw cycles, chloride intrusion, and acid and sulfate attacks. It measures how easily fluids can pass through concrete, with the depth of water penetration serving as the standard metric for assessing permeability.

The factors influencing the permeability of reinforced concrete (RC) are similar to those affecting WA, primarily due to increased P in the internal structure and the presence of macro- and micro-cracks. Rubber particles incorporated into concrete tend to rise within the cement matrix because of their lightweight and elastic nature, leading to poor compaction of RC and consequently higher P and water permeability as seen in figure II.10. The weak bonding between the cement matrix and rubber aggregates (RA) results in cracks around the rubber particles. These cracks, both around the rubber particles and within the cement, facilitate the penetration and movement of external water throughout the concrete. According to Su et al., using rubber particles with smaller sizes (0–0.5 mm) as a substitute for fine aggregate can more effectively reduce permeability compared to larger rubber particles (0.5–3 mm). The superior filling capability of smaller rubber particles contributes to a denser internal structure in RC. When RA is well-graded and consists of various sizes of rubber particles, it becomes more compact, as fine rubber particles can efficiently fill the voids created by larger particles [45].

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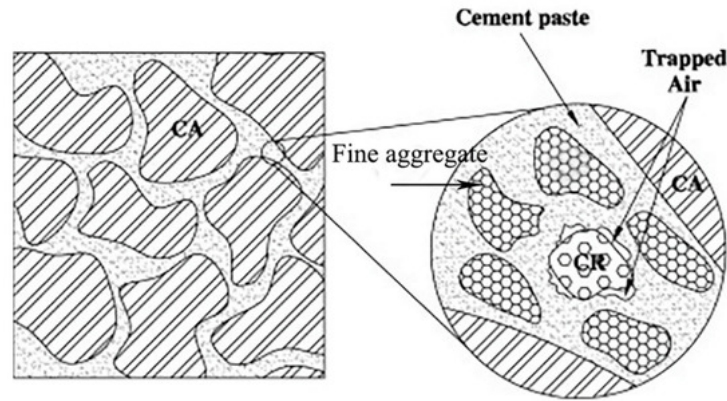


Figure II.10. Microstructure of CR concrete [45].

II.8.2. Chloride Ion Penetration

One major overlooked threat to building safety is the longevity of concrete in marine environments. The cycle of drying and wetting greatly accelerates the spread of corrosive ions, with the tidal range area becoming the most critical zone for concrete structure corrosion. Chloride ion diffusion is a key factor affecting concrete durability. Figure II.11 illustrates that incorporating rubber into concrete significantly reduces the absorption of free chloride ions at various depths, with the resistance to chloride ion erosion ranked as RC-2 group > RC-1 group > RC-3 group > OC group. This suggests that adding an appropriate amount of rubber to concrete can improve its resistance to chloride ion damage and minimize chloride ion penetration. It is advised to use a 10% rubber content to effectively reduce free chloride ion absorption at different concrete depths [62].

Chapter II: Effect of waste tire rubber on the properties of mortar and concrete

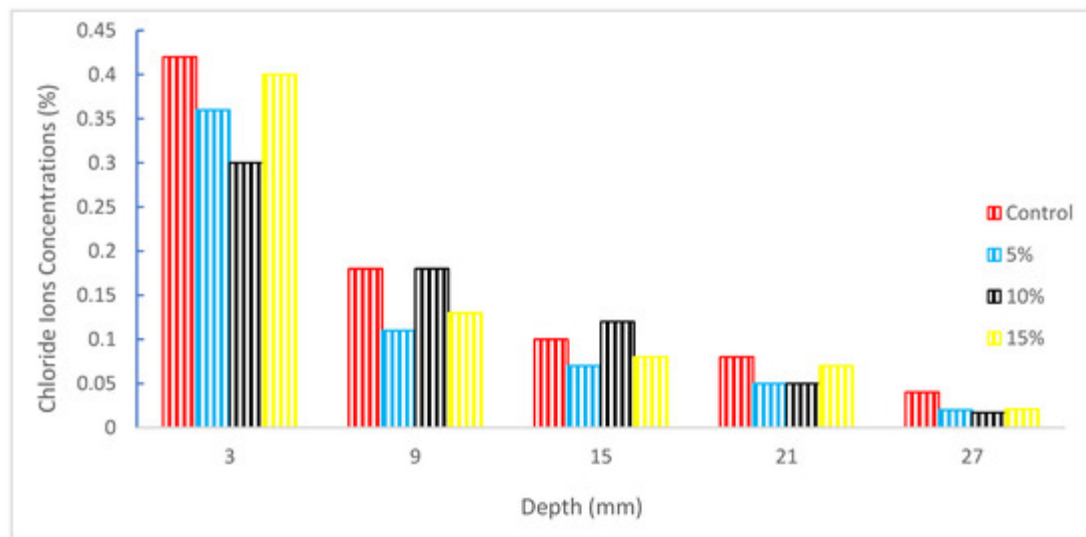


Figure II.11. Chloride ion penetration[62]

RuC is more temperature, pressure, and impact resistant than conventional concrete. Additionally, it is more economical and economical. Rubber-modified concrete (RMC) has been observed to have relatively low tensile and CSs. On the other hand, they have excellent sound and thermal insulation, minimal shrinkage, strong impact resistance, appropriate water resistance with low absorption, and increased acid resistance. Studies show that CRC (CR concrete) specimens did not shatter after failure when compared to a typical concrete mix. This behavior may be useful for construction that needs to have strong impact resistance qualities. RuC with enhanced impact resistance was clearly visible in concrete samples that were aggregated with thick rubber.

Additionally, RuC's unique qualities will find new uses in building structures as sound barriers, shock absorbers for highway construction, and earthquake shock-wave absorbers. It reduces concrete's vulnerability to catastrophic collapse, plastic shrinkage, and cracking [62].

Concrete that has had scrap tires added to it is currently used for precast sidewalk panels, non-load-bearing building walls, and precast roofs for green buildings. It is frequently used for construction-related tasks like building skid-resistant ramps and recreational walkways and courts. Thanks to this new property, these concretes are expected to be used in architectural applications like nailing concrete, where high strength is not necessary, wall panels that require low unit weight, construction elements, impact-prone Jersey barriers, and railroads to secure rails to the ground

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[62]. Since RuC can also be used in non-load-bearing components like lightweight concrete walls, building facades, or other light architectural units, waste tire-modified concrete mixes may offer a practical substitute for standard weight concrete. Rubberized mixes can be used anywhere cement-stabilized aggregate bases are needed, particularly beneath flexible pavements. The other useful applications are perfect for use in areas that frequently freeze and thaw because they can be poured in larger sheets than ordinary concrete [62].

II.8.3. Acid and Sulphate Resistance

The incorporation of rubber into concrete enhances resistance to sulphate attack primarily due to improved deformation capacity, which alleviates internal expansion stress caused by sulphates. Rubber particles absorb expansion stress, limit crack development, and reduce crack width, thereby restricting sulphate ion penetration. Fine rubber particles increase micro-voids that act as buffers against volume expansion from gypsum and ettringite. Studies show that RuC with 10% silica fume exhibits greater sulphate resistance, attributed to a denser matrix and reduced aluminate/ $\text{Ca}(\text{OH})_2$ content, hindering sulphate intrusion. Pretreatment of CR with NaOH or coating with modifiers, especially synthetic resin, improves adhesion with cement and enhances sulphate resistance, CS, and freeze–thaw durability. However, replacing NA with rubber aggregate in high-strength concrete may reduce sulphate resistance due to increased WA, facilitating aggressive solution ingress. Figure II.12 illustrates the resistance of RuC to H_2SO_4 corrosion as influenced by different rubber contents [45].

Chapter II: Effect of waste tire rubber on the properties of mortar and concrete

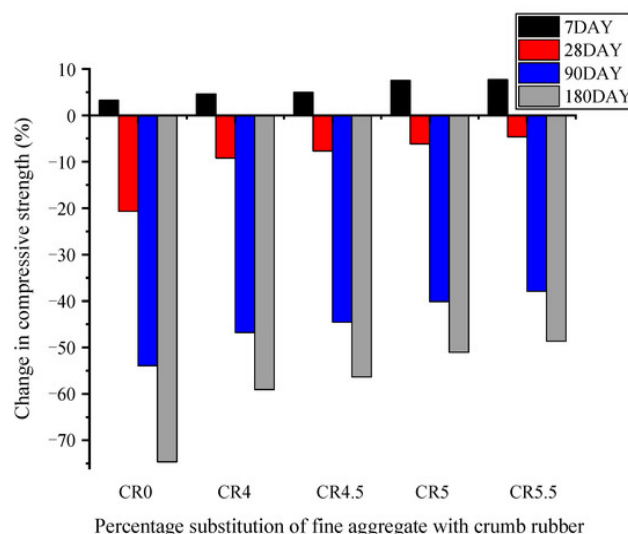


Figure II.12. Change in CS after soaking in 3% H_2SO_4 solution [45].

II.8.4. Carbonation Resistance

Cement and concrete rank as the most commonly utilized construction materials globally, second only to water. Their production process is responsible for approximately one-third of greenhouse gas emissions. The demand for these materials is anticipated to rise in the upcoming decades. Notably, concrete's ability to absorb CO_2 is significant. It possesses a substantial capacity to capture carbon, transforming CO_2 into chemically stable carbonates, thus facilitating long-term CO_2 sequestration. This feature helps counteract part of the calcination reaction, considerably lowering CO_2 emissions throughout concrete's lifecycle. Ignoring concrete's CO_2 absorption would lead to an overestimation of the cement and concrete industry's carbon emissions, which could impact policy development and the enforcement of environmental standards. Consequently, CO_2 curing of concrete is seen as a promising carbon neutralization technology. A crucial factor in accurately assessing its storage potential is evaluating the CO_2 absorption capacity, which is reflected in the degree of carbonation (C) of concrete or mortar.

In theory, the depth of C can be assessed using the phenolphthalein spray technique. To prepare the phenolphthalein ethanol solution as an indicator, dissolve 1 g of phenolphthalein in 95% ethanol and then dilute the mixture to 100 mL. Apply this phenolphthalein indicator to the freshly cut surface of the mortar specimen. The section of the mortar that is fully carbonated will maintain its original hue because it lacks alkaline substances, whereas the uncarbonated section will turn purplish-red due to its high alkalinity. After allowing 30 seconds for the color to develop, use a

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steel ruler to measure the distance from the marked points, spaced every 10 mm on both sides, to the point where the color change occurs as shown in figure II.13 [66].

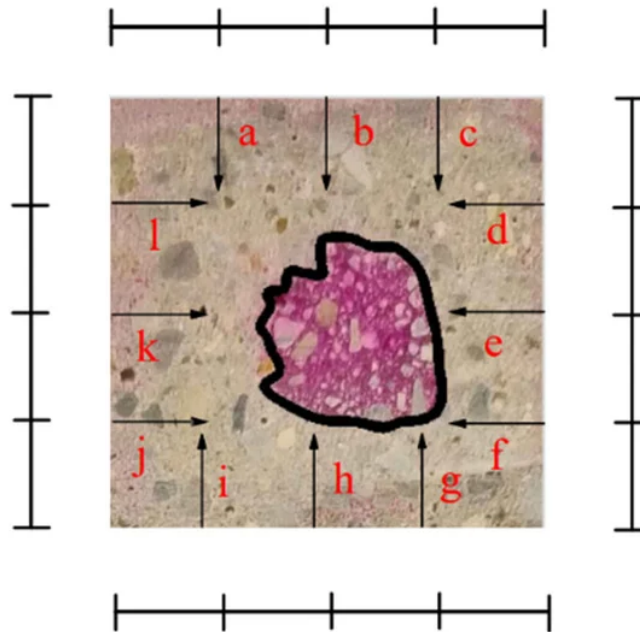


Figure II.13. Measurement method of *C* depth [66]

The process of *C* presents a significant threat to concrete and mortar, as it reduces alkalinity and leads to increased spalling. *C* is a crucial factor in assessing the lifespan of structures, often necessitating frequent maintenance for those with lower durability. This process can impact the functionality, stability, and visual appeal of structures. Mortars are widely used in construction for various purposes, such as binding, padding, and providing protection in mortar structures. Due to their high strength, affordability, ease of handling, and molding capabilities, mortars have numerous applications. Additionally, the various properties of mortars significantly influence mortar structures, as mortar makes up a substantial portion of concrete's volume. Moreover, different environmental and climatic conditions can affect the longevity of mortar. Carbon dioxide in the air can penetrate mortar and alter the hydrated cement paste, leading to a decrease in the pH of the pore solution [67].

RA improves the extent of *C* by substituting NA in concrete. According to Gheni et al., the depth of *C* is significantly influenced by the permeability of concrete, which is determined by the size

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and distribution of internal pores. As the duration of exposure to a CO₂ environment increases, so does the C depth. The incorporation of rubber into concrete negatively impacts its workability; during mixing, the concrete's interior becomes difficult to compact, leading to the formation of internal pores and cracks that facilitate CO₂ penetration. Replacing coarse aggregate with rubber results in a greater C depth than substituting fine aggregate. Thomas and Gupta discovered that the reduction in C resistance is akin to the decrease in permeability resistance. Both are due to the hydrophobic properties of RA, which make it challenging for the cement matrix to adhere securely to the rubber particle surface. Consequently, internal pores and cracks proliferate, creating a broad pathway for CO₂ to infiltrate the concrete. Pham et al. observed that pretreating CR with NaOH improved adhesion and hindered CO₂ penetration. Additionally, the residual NaOH on the rubber reacted with CO₂, aiding in the reduction of C depth [45].

II.9. Conclusion

Tire waste represents a significant environmental and public health challenge due to its non-biodegradable composition, large volume, and complex chemical makeup. The accumulation of discarded tires in landfills not only occupies valuable space but also creates breeding grounds for disease-carrying mosquitoes, contributing to the spread of illnesses such as malaria and dengue. Furthermore, tire fires are difficult to control and release harmful gases that degrade air quality. The slow decomposition of tires can lead to the leaching of toxic substances into soil and groundwater, posing risks to ecosystems and agriculture. Given these concerns, recycling tire rubber, particularly through its incorporation into fiber-reinforced mortar and concrete, offers a promising sustainable solution. This approach helps reduce environmental pollution, conserves natural resources, and supports public health by diverting waste from landfills and minimizing hazardous impacts. Emphasizing such innovative recycling methods is essential for advancing environmental protection and sustainable construction practices.

CHAPTER III:
MATERIALS AND
EXPERIMENTAL PROCEDURE

III.1. Introduction

The objective of this chapter is to describe the materials used in the formulation of the various mortar mixtures investigated and to detail the experimental methodology adopted in this research work. Particular attention is given to the characterization of WTR and DPF, as well as the conventional constituents employed in the production of the mortars.

This study aims to assess the feasibility of partially substituting conventional aggregates with waste tire rubber in mortar formulations, combined with NF, in order to produce mortars with reduced environmental impact. Replacement WTR of 0%, 10 %, 20%, and 30% were examined, together with 1% of DPF content. The behavior of the resulting mortars was evaluated in both the fresh and hardened states and systematically compared with that of a reference mortar made exclusively with 1% of DPF content.

The materials used in this study are as follows:

- Composite Portland cement (CEM II/A 42.5) supplied by the Oued Isly cement plant (Chlef, Algeria).
- Natural sand (NS)
- Recycled sand: Crumb rubber derived from waste tires (CR)
- Palm fibers (DPF) : leaflets
- Admixtures: superplasticizer (SP)

III.2. Experimental Program

This experimental program was carried out at the laboratory of Ahmed Ben Yahia El Wancharissi University in Tissemsilt. Its objective is to determine selected properties of plastic mortar by incorporating WTR as partial replacements with a fixed percentage of palm fibers. NS was partially replaced by CR at substitution rates by volume of 0%, 10%, 20%, and 30%, while palm fibers were fixed at 1%. The cement used in this study is a composite Portland cement CEM II/A-L 42.5 N. In order to obtain a plastic mortar, the sand content and the water-to-cement ratio (W/C) were kept constant, and a superplasticizer was added. For a given mixture, the flow spread was measured using the flow table test until the desired workability was achieved. Prior to conducting mortar tests, an initial stage of the experimental program, referred to as material characterization

tests, was performed. During this stage, all relevant physical and mechanical properties of the constituent materials were determined in accordance with the applicable standards.

Finally, the prepared mortars were subjected to a series of physical and mechanical tests to evaluate their behavior. This study aims to investigate the physical and MP of DPF-based mortar containing different proportions of CR through the following laboratory tests in the fresh and hardened state

Fresh state

- Workability
- Consistency
- Spread test

Hardened state

- Flexural strength (FS)
- Compressive strength (CS)
- Porosity (P)
- Water absorption by immersion (WA)
- Water absorption by capillarity: sorptivity (S)
- Weight loss (WL)
- Carbonation (C)

III.3. Materials Used

III.3.1. Cement

For this study, a Portland composite cement CEM II A-L 42.5 N produced by the Oued Sly cement plant (Chlef) containing 12% limestone was used (Figure III.1). Its average CS at 28 days is greater than 42.5 MPa, and its specific Blaine surface area is 3200 cm²/g. Its chemical and mineralogical characteristics are presented in Table III.1.

Table III.1. Chemical and mineralogical composition of Portland cement (CEM/ A 42.5)

Chemical composition%								Mineralogical composition %			
SiO ₂	Al ₂ O ₃	FeO ₃	CaO	SO ₃	MgO	K ₂ O	LOI	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
22.6	4.2	3.55	62.17	2.19	0.63	0.42	1.84	41.8	33.3	5.1	10.7

**FigureIII.1.** Portland cement (CEM II/A 42.5)

III.3.2. Sand

In this work, two types of sand were used: NS (0/4 mm) sourced from the river (Tissemsilt) and crumb obtained from grinding used rubber tires (Figure III.2). Several characterization tests were conducted on these two types of sand to optimize their use in mortar production.



NS



CR

Figure III.2. View of the different sands used (natural sand / recycled sand).

III.3.3. Mixing Water

For this research, water from the potable water supply network of the city of Tissemsilt was used, which is clean drinking water. It meets all the requirements set by standard P18-303[68]. Its chemical composition is given in Table III.2.

Table III.2. The chemical composition analysis of the mixing water.

Sample	Ca	Mg	Na	K	Cl	SO ₄	HCO ₃	NO ₃	Dry residue	pH
Concentration (mg/l)	53	6.6	5.8	0.5	11	9	168	2	160	7.3

III.3.4. Superplasticizer

Superplasticizers (Figure III.3) are polymeric substances that reduce the water requirements of a cementitious material (mortar or concrete) and increase its flowability without impacting the mechanical strength. The superplasticizers are chemically different from normal water reducers and are capable of reducing water content up to about 30%.

Chapter III:**Materials and experimental procedure****Figure III.3.** Sika superplasticizer**Table III.3.** Physicochemical Characteristics of the Superplasticizer

Sample	Chloride ion content	Appearance	color	PH	Density	Recom mended dosage	Chemical nature
Concentration (mg/l)	$\geq 0,1\%$	Liquid	Light to dark brown	$4,5 \pm 1$	$1,055 \pm 0,015$	0,7% to 2,5%	Polycarboxylate ethers(PCE)

III.3.5. Fibers

The fibers used in this work are those of the date palm leaves, specifically the "leaflet" part (See Figure III.4). These fibers have a light green color and a cross-section that can be considered rectangular. The preparation of the leaflet fibers begins with cleaning them with water to remove impurities. They are then cut to a length of 20 mm and an average width of approximately 1 mm before being dried at ambient temperature.



Figure III.4. DPF used after treatment and cutting

III.4. Characteristics of Sands

III.4.1. Grain Size Distribution

A particle size analysis was carried out in accordance with standard NF P-94-056[69]. This test provides an overview of the variation in grain sizes. It involves dividing a material into several particle size classes using a series of sieves stacked on top of each other. The material under study is placed at the top of the sieves, and the grains are sorted by vibrating the sieve column. The masses of the different residues and passing fractions are related to the initial mass of the material. The resulting percentages are then presented graphically. Each test uses 2000 g of sand, and the percentages of the passing fractions are determined from the measured residue values. The sieve shaker used is shown in Figure III.5.



Figure III.5. Sieve used to determine the granulometry of sands.

A particle size distribution curve makes it possible to anticipate potential issues in mortar formulation. When the curve is regular and continuous, the skeleton of the formulated mortar will be compact (better compactness of the mortar skeleton), in addition, the continuity of the curve

allows for mortars with better fluidity and no segregation problems. The particle size distribution curves for ordinary aggregates and recycled aggregates are shown in Figure III.6.

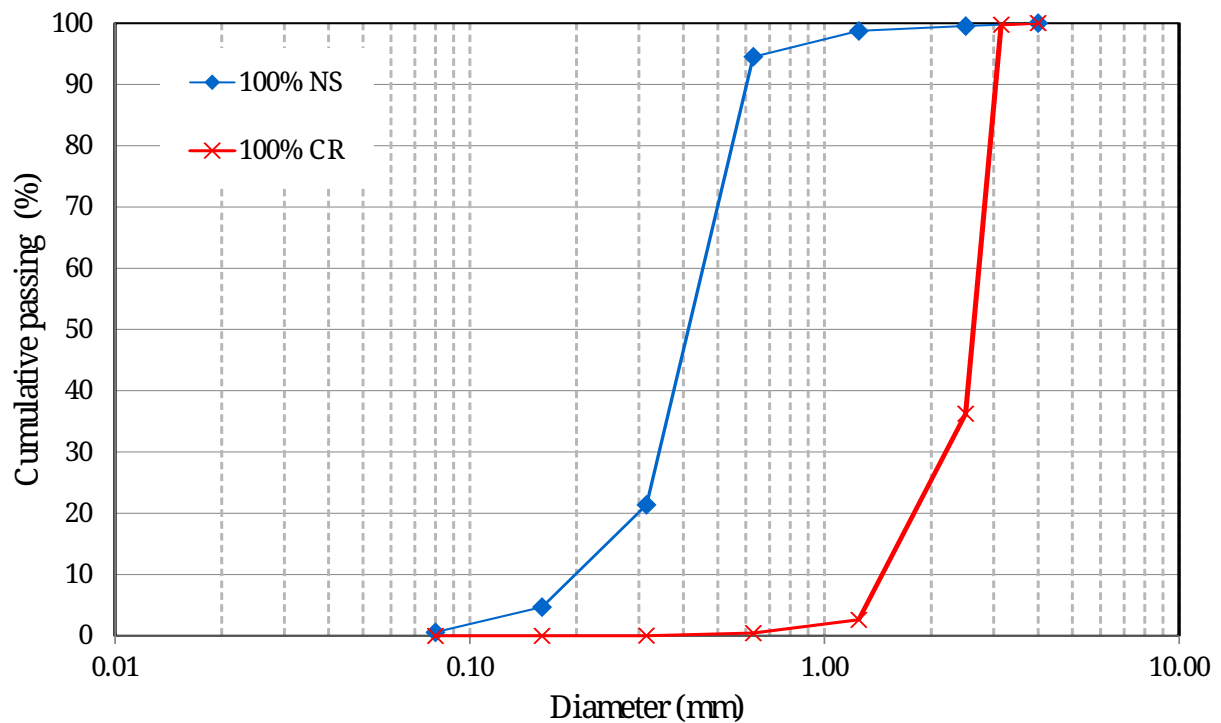


Figure III.6. Particle-size distribution curves of the two sands used.

III.5.2. Fineness Modulus

Determining the fineness modulus (FM) is one of the most important steps in preparing and formulating a mortar, since knowing the FM will indicate the amount of fine and coarse elements that make up the sand. The proportion of these elements greatly affects the behavior of the mortar (workability, risk of segregation, loss/increase in strength). According to standard NF P94-056[69], the fineness modulus of an aggregate is equal to 1/100th of the sum of the cumulative residues, expressed in %, of the different sieves as shown by the formula below:

$$M_f = \frac{R_5 + R_{2,5} + R_{1,25} + R_{0,315} + R_{0,16} + R_{0,08}}{100} \quad (\text{III.1})$$

Depending on the value of the fineness modulus, sands can be classified as follows

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- $1.8 < FM < 2.2$: Fine sand, mainly used for its positive effect on workability during placement, at the possible expense of mechanical strength.
- $2.2 < FM < 2.8$: Preferred sand, suitable for achieving adequate workability and good mechanical strength, while reducing the risk of segregation.
- $2.8 < FM < 3.3$: Coarse sand, often used to obtain high mechanical strength; however, this is generally accompanied by negative effects, such as reduced workability and an increased risk of segregation.

III.5.3. Densities

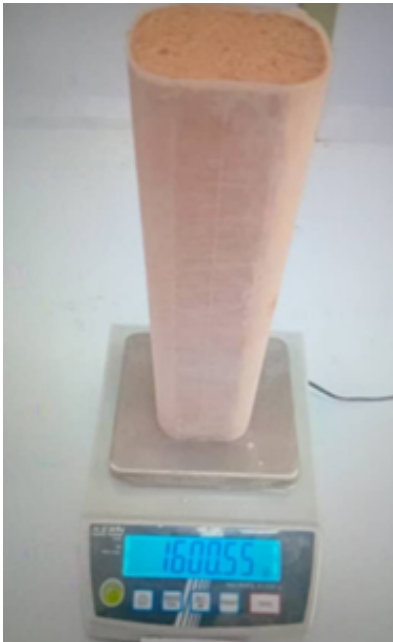
This characteristic, also called mass bulk density, makes it possible to determine the mass or volume of the different granular classes. The apparent bulk density (including voids) and the absolute bulk density (excluding voids) of a granular fraction are determined for the NS as well as for the rubber aggregate with different substitution rates.

III.5.3.1. Apparent Density (ρ_{app})

The apparent bulk density of a material is defined as the mass of one cubic meter of the material. For its determination, a quantity was taken in accordance with standard EN 1097-6[70]. The test involves filling a one-liter container with dry material, without compaction, and then weighing this quantity (M) using a scale with 0.1-gram accuracy, as shown in Figure III .7. The procedure is as follows:

- 1) Weigh the empty 1-liter container, denoted as M_1
- 2) Fill the container with aggregates (natural or recycled) by hand scoop until it overflows. The hand scoop should be at a height not exceeding 5 cm;
- 3) Level off the excess grains
- 4) Weigh the container with the grains, denoted as M_2
- 5) The value is calculated by the following relation:

$$\rho_{app} = \frac{M_2 - M_1}{V} \quad (III.2)$$



NS



CR

Figure III.7. Apparent density test for two types of sand used.

Where :

- M in (g): dry mass of the material.
- V = 1000 in (ml): represents the volume of the container.

III.5.3.2. Absolute Density (ρ_{abs})

This consists in measuring the mass per unit of volume of material by eliminating all the voids existing between the grains. The ratio of the different recorded volumes and masses of the material gives the absolute bulk density (See Figure III.8).

This method is very simple to perform using a graduated cylinder by measuring the difference in volumes.

- Fill a graduated cylinder with an initial volume V_1 of water.
- Weigh a dry aggregate sample of mass M (approximately 30g) and introduce it into the cylinder, ensuring that all air bubbles are removed. The water level will rise.
- Record the new volume V_2 .

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The density is then calculated as:

$$\rho_{abs} = \frac{M}{V_2 - V_1} \quad (III.3)$$

Where:

$V_2 - V_1$: represents the volume of the solid particles.

The absolute bulk density of the recycled sand (CR) was determined using the immersion method with toluene as the displacement liquid (Figure III.9).



Figure III.8. Method used to determine the absolute density of a sand.

The use of toluene (Figure III.9) is recommended for rubber-based materials due to its low polarity and negligible interaction with elastomeric particles, thereby minimizing absorption and swelling effects observed with water. This method enables an accurate evaluation of the true volume of CR particles and ensures repeatability and reliability of the measured absolute bulk density, which is essential for mix design and comparative analysis.

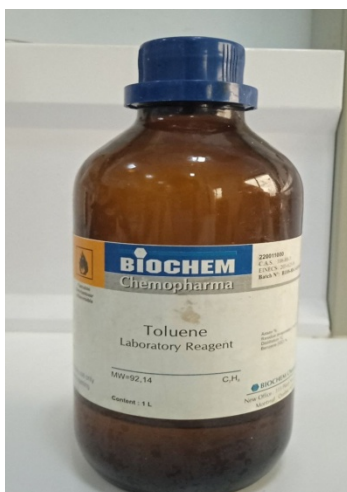


Figure III.9. Toluene used in the absolute bulk density test.

III.5.4. Sand Equivalent

The sand equivalent is a parameter used to assess the cleanliness of sand. This test is carried out in accordance with EN 933-8[71]. It provides information on the content of fine particles and the presence of excess materials of organic, vegetal, and mainly clayey origin, which constitutes the primary objective of this test. The presence of clay in the material adversely affects shrinkage behavior and leads to an increased water demand, thereby resulting in a reduction in mechanical strength (Figure III.10).

The test consists of introducing a specified quantity of sand into a standardized graduated cylinder previously filled with a deflocculating solution, whose role is to separate sand grains and release entrapped air bubbles after agitation. The operating procedure is carried out according to the following steps:

1. Preparation of a dry sand sample with a mass of 120 g, which is introduced using a funnel
2. into a graduated cylinder previously filled with deflocculating solution up to the lower calibration mark.
3. Firmly tapping the base of the cylinder several times with the palm of the hand in order to release and expel air bubbles.
4. Sealing the cylinder with a rubber stopper and placing it in an electrical shaker for 90 cycles over 30 seconds.

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5. Carefully washing the cylinder and the stopper to remove sand residues until the upper calibration mark is reached.
6. Allowing the sample to stand for 20 minutes, then visually measuring the heights.
7. For measurement using the piston, after the resting period, the piston is carefully inserted into the cylinder and the corresponding heights are recorded.

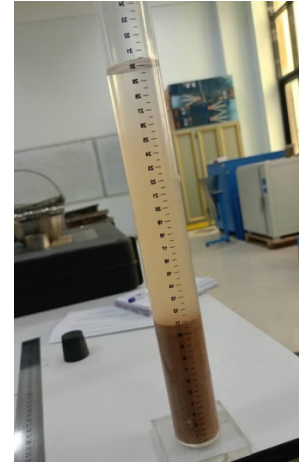


Figure III.10. Sand equivalent test.

The sand equivalent is determined using the following relationship:

$$Es = \frac{H_2}{H_1} 100 \quad (\text{III.4})$$

Where:

- H_2 (Sand height): Height of the clean sand sediment measured after 20 minutes of settling, usually using a piston.
- H_1 (Total height): Total height of the sand and flocculated material.

However, a complete lack of fine particles leads to concrete with poor workability and low compactness.

III.6.1. Mortar formulation

The objective of the formulation is to determine the required quantity of each mortar component. A standardized mortar was chosen with a weight-based sand-cement-water composition of 3:1:0.5. The NS will be replaced by CR at rates of 0, 10, 20, and 30% by volume. To maintain consistent workability for all mortars, 1% of superplasticizer is added, keeping a spread of 80% on the flow table, which ensures a plastic behavior in the mortar (TableIII.4).

TableIII.4. *Formulation and composition of plastic mortar.*

Substitution rate of NS by CR	0%	10%	20%	30%
Natural sand NS(g)	1345,5	1211	1076	941
Crumb rubber CR (g)	0	47	94	141
Cement(g)	450			
Water(g)	220.5			
Date palm fibers DPF(g)	4.5			
Superplasticizer(g)	4.5			

III.6.2. Sample Preparation

To ensure the production of a workable and homogeneous mortar, the mixing process plays a crucial role in achieving the desired properties. After determining the proportions of the various mortar constituents, the mortar preparation proceeds by mixing these materials. According to the EN 196-1 standard [15], the mixing is conducted over a total duration of 4 minutes, divided into following stages (see Figure III.11):

1. Dry mix the cement and sand for 1 minute to achieve uniform distribution;
2. Add two-thirds of the total mixing water and mix for 1 minute;
3. Add fibers with the remaining one-third of the water along with the superplasticizer and mix for 1 minute;

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4. Switch to the second mixing speed and continue mixing for 1 minute;
5. Proceed with flow/spread tests or calorimetric assays.



Figure III.11. Mortar mixing.

III.6.3. Spread test

After mixing the different mortars, we first checked the fluidity using the spread test conducted on a shaking table. The procedure for this test was carried out according to the ASTM C230 standard [72]. The test involves placing the prepared mortar in the conical mold and, after demolding on the shaking table, the mortar receives 15 blows in 15 seconds, and the diameter of the resulting disk is measured as illustrated in Figure III.12.



Figure III.12. Spread test for mortar

III.6.4. Molding of Specimens

Once the mortars were prepared, the specimens were molded in two layers. The first layer was placed into each compartment of the mold, after which the mold was positioned on the flow table (Figure III.13) and the layer was compacted with 60 drops. The second layer was then added and similarly compacted with 60 drops. Finally, the mold was removed from the table, and any excess mortar was leveled off using a trowel, as shown in Figure III.14.

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Figure III.13. Flow table



Figure III.14. *Filling of the molds.*

III.6.5. Curing Conditions of the Specimens

Immediately after molding, the molds were covered with plastic sheets and stored under laboratory ambient conditions. After 24 hours, the specimens were demolded and stored under conditions appropriate for each test (See Figure III.15).



Figure III.15. Curing tank for the different specimens

III.7. Tests Performed on Mortar

III.7.1. Normal Consistency

The purpose of the consistency test is to evaluate the fluidity or workability of fresh mortar, i.e., its ability to flow or deform under its own weight. This test is essential to determine whether the mortar has an appropriate consistency for its intended application (plastering, masonry, injection, etc.).

The test consists of inserting a standardized needle (Vicat needle) into a fresh mortar paste contained in a truncated conical mold (Figure III.16). The amount of mixing water is adjusted until the needle penetrates 6 ± 1 mm from the bottom of the mold. The test was carried out in accordance with NF P15-414[73].



Figure III.16. Vicat apparatus used to determine the normal consistency of a mortar.

III.7.2. Flexural strength

The three-point bending test is performed on prismatic mortar specimens (4×4×16 cm) at the ages of 2, 7, 28 and 90 days, as shown in Figure III.17. The flexural tensile strength is calculated using the following formula:

$$\sigma_f = \frac{3PL}{2a^3} \quad (\text{III.5})$$

Where:

σ_f = flexural strength, in Megapascals (N/mm²)

P = maximum load, in Newtons

L= span between the two support rollers, in millimeters

a= dimension of the specimen's cross-sectional area, in millimeters.



Figure III.17. Flexural tensile test on a mortar specimen.

III.7.3. Compressive strength

The CS was measured according to standard EN 196-1[15] by axial compression on prismatic specimens broken into halves at the ages of 2, 7, 28 and 90 days, using a 2000 kN capacity press (Figure III.18).

The CS corresponds to the average crushing stress of three (03) halves from three specimens.

The CS is calculated using the following formula:

$$\sigma_c = \frac{P}{S} \quad (\text{III.6})$$

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Where:

- σ_c : failure stress
- P: failure load (N)
- S: cross-sectional area of the specimen (mm²)



Figure III.18. Compression test on a mortar specimen.

III.7.4. Porosity

The P test (Figure III.19) was carried out in order to determine the volume of voids accessible to water in the mortar specimens. This parameter is important for evaluating the durability and permeability characteristics of plastic mortar. The test was performed according to ASTM C642[74]. Mortar specimens cured for 28 days were used for this test. The procedure consists of determining three masses of the specimen: the dry mass, the saturated mass, and the immersed mass in water.

The procedure is as follows:

Dry the specimens in an oven at 105 ± 5 °C until constant mass and record the dry mass (M_d). Immerse the specimens completely in water for 24 hours in order to saturate the accessible pores. Remove the specimens from water, wipe the surface with a cloth to remove excess water, and measure the saturated mass (M_s).

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Suspend the specimen in water using a thin wire without touching the bottom or the sides of the container and measure the immersed mass (M_i).

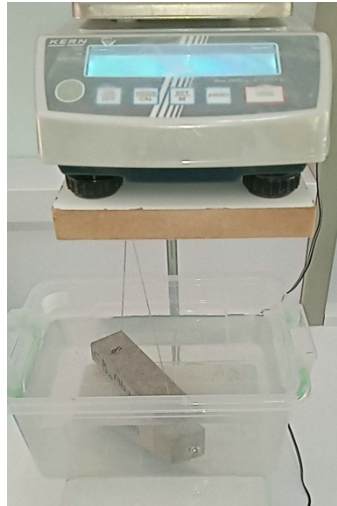


Figure III.19. *P test on mortar specimen.*

The porosity is calculated using the following relation:

$$P = \frac{M_s - M_d}{M_s - M_i} \times 100\% \quad (\text{III.7})$$

Where:

M_d : dry mass of the specimen (g)

M_s : saturated mass of the specimen (g)

M_i : immersed mass of the specimen in water (g)

III.7.5. Water Absorption by Immersion

WA by immersion in concrete is related to capillary movements within the open pores of the material. To evaluate this absorption, a mortar specimen is immersed in water until its mass stabilizes (Figure III.20). This method complies with the requirements of standard NF EN 206-1[75].

WA is expressed as a percentage of the dry mass of the specimen and serves as an indicator of P to assess the quality of the mortar.

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The absorption coefficient is defined by the following ratio:

$$Abs \% = \frac{M_h - M_s}{M_s} \quad (III.8)$$

Where :

- M_h : Mass of the specimen after immersion, in grams (g)
- M_s : Mass of the specimen after drying, in grams (g)

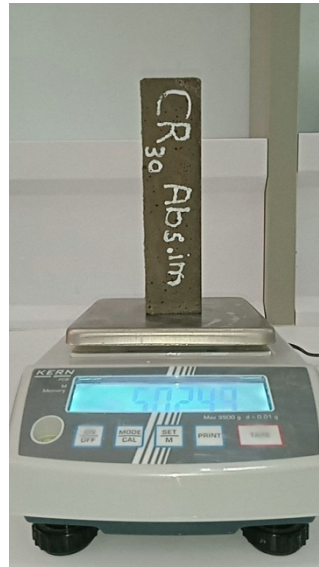


Figure III.20. Water Absorption by Immersion test.

III.7.6. Water Absorption by capillarity(sorptivity)

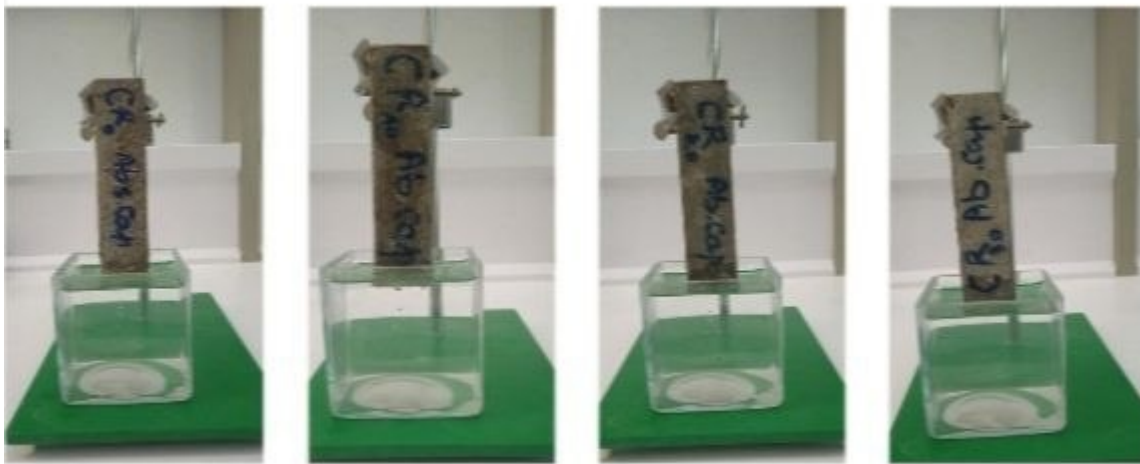
The S test was performed on mortar specimens with dimensions of $4 \times 4 \times 16$ cm after 28 days of curing under laboratory conditions (20 °C and 95 % relative humidity), following the procedure recommended by ASTM C1585-20[76]. Before testing, the surfaces not intended to be in contact with water were wiped to remove superficial moisture, and the initial mass (M_0) of each specimen was recorded, to ensure one-dimensional water penetration, all faces except the base were sealed with resin (See figure III.21). The specimen was then placed in a container with only its base immersed in water to a depth of approximately 5 mm, while the remaining faces were sealed to prevent lateral water penetration as presented in figure III.22. The mass of the specimen was measured at regular time intervals (1, 2, 5, 10, 20, 30, 60, and 120 minutes) after removing excess surface water. After each measurement, the specimen was immediately returned to the water. This

Chapter III:**Materials and experimental procedure**

test evaluates the capillary water absorption capacity of the mortar and provides information about its pore structure and durability.



FigureIII.21. Epoxy resin and sealed mortar specimens prepared for the sorptivity test



FigureIII.22. S test on different mortar specimens.

The S is defined by the following ratio:

$$S = \frac{\Delta M}{A\sqrt{t}} \quad (\text{III.9})$$

Where:

- $\Delta M = M_t - M_0$: mass of water absorbed (g)
- A: area of the immersed base (cm²) (for a 4 × 4 cm mortar specimen, A = 16 cm²)
- t: time (min or s)
- S: sorptivity (g/cm²·min^{0.5})

III.7.7. Weight Loss

After demolding, the specimens are stored in a room at a temperature of 20 ± 2 °C. The prepared specimens are then exposed to air at 20 ± 2 °C for 28 days. The specimen's weight is measured every 15 days over a period of 3 months (See Figure III.23).

It should be noted that dry room curing is absolutely necessary so that water contained in non-connected pores can leave the specimen. Otherwise, this water would be counted as absorbed water.

The WL is defined by the following ratio:

$$\Delta M = \frac{M_0 - M_i}{M_0} 100\% \quad (\text{III.10})$$

- ΔM : Percentage of mass loss
- M_0 = initial mass of the specimen
- M_i = mass of the specimen on day i

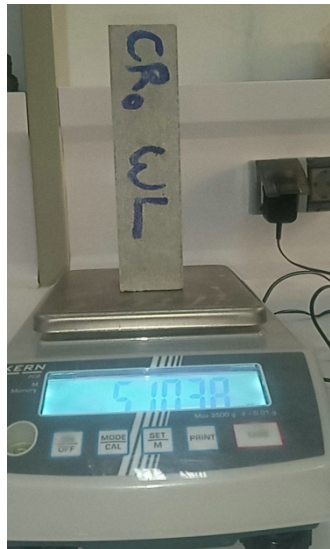


Figure III.23. Monitoring the weight of a mortar specimen during drying.

III.7.8. Carbonation

The C test was carried out to determine the depth of C in mortar specimens and evaluate their durability against carbon dioxide penetration. The test was performed according to NF EN 13295[77].

Mortar specimens cured for 28 days were first removed from the curing environment and allowed to dry under laboratory conditions. Each specimen was then split into two halves to expose a fresh surface. The exposed surface was sprayed uniformly with a phenolphthalein solution with 1% concentration in ethanol (See figure III.24).



Figure III.24. Chemical products used for preparing phenolphthalein solution for C test.

The non-carbonated zones turn purple, indicating a high alkalinity ($\text{pH} > 9$), while the carbonated zones remain colorless. The C depth was determined by measuring the distance between the external surface and the boundary between the colored and uncolored zones. The results are indicated in figure III.25.



Figure III.25. C test on mortar specimens after 28 and 90 days.

III.8. Conclusion

To characterize a material or mortar, multiple tests must be conducted and repeated due to the available equipment. Most of the tests were carried out under good conditions, and the results will be presented and discussed in the next chapter.

CHAPTER IV:
RESULTS AND DISCUSSIONS

IV.1. Introduction

In recent years, the construction industry has faced increasing pressure to adopt more sustainable practices due to the environmental impacts associated with conventional building materials. The accumulation of waste tires and the over-extraction of NF pose significant ecological challenges, including landfill overflow, pollution, and depletion of finite resources. Incorporating waste tire rubber and natural palm fibers into mortar production offers a promising solution to these issues by transforming waste into valuable raw materials, reducing environmental burdens, and conserving natural resources. This chapter presents and discusses the results obtained from experimental investigations aimed at evaluating the feasibility and performance of mortars enhanced with these sustainable additives, thereby contributing to the development of low environmental impact construction materials.

IV.2. Research Objective

The primary objective of this research is to explore and evaluate the potential of valorizing WTR and NF as sustainable additives in the production of mortars with reduced environmental impact. Specifically, the research aims to investigate how the incorporation of these materials influences the physical, mechanical, and durability properties of mortars, while contributing to waste reduction and the conservation of natural resources. Through systematic experimentation and analysis, this research seeks to contribute to the development of low environmental impact mortars that support sustainable building practices and resource conservation.

IV.3. Fresh state**IV.3.1. The Need for a Superplasticizer**

Figure IV.1 illustrates how CR incorporation affects mortar workability and consistency, as well as the resulting demand for superplasticizers. The results indicate that the spread values remain relatively stable for all mixtures, varying only slightly between approximately 80% and 85%. The consistency of the mortar mixtures slightly increased with increasing CR content, while the addition of SP ensured adequate workability for all formulations. The mortars' workability was successfully maintained despite the increase in CR content, as evidenced by this relatively constant spread.

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Moreover, the incorporation of NF generally has a negative impact on the workability of cementitious materials in the fresh state. This reduction in workability is mainly attributed to the high WA capacity and the rough surface texture of the fibers, which increase internal friction within the mixture and reduce its fluidity. Consequently, the fresh mix becomes more difficult to mix, place, and compact. Aziz et al. [78] have reported that the use of a SP is an effective solution to overcome this issue. Superplasticizers improve the dispersion of cement particles and enhance the fluidity of the mixture without increasing the water content. Therefore, the addition of a SP is often necessary in fibrous cementitious composites to ensure adequate workability while maintaining the desired mechanical properties. Ren et al. [79] explored how SP affects workability. Their findings indicated that SP greatly enhanced the workability of the cementitious mixture. In a similar vein, they found that increasing the fiber replacement percentage led to reduced workability in mixtures, but SP helped counteract the workability loss caused by fiber additions. Thus, adding SP improves the workability of the fiber-reinforced cement-based matrix.

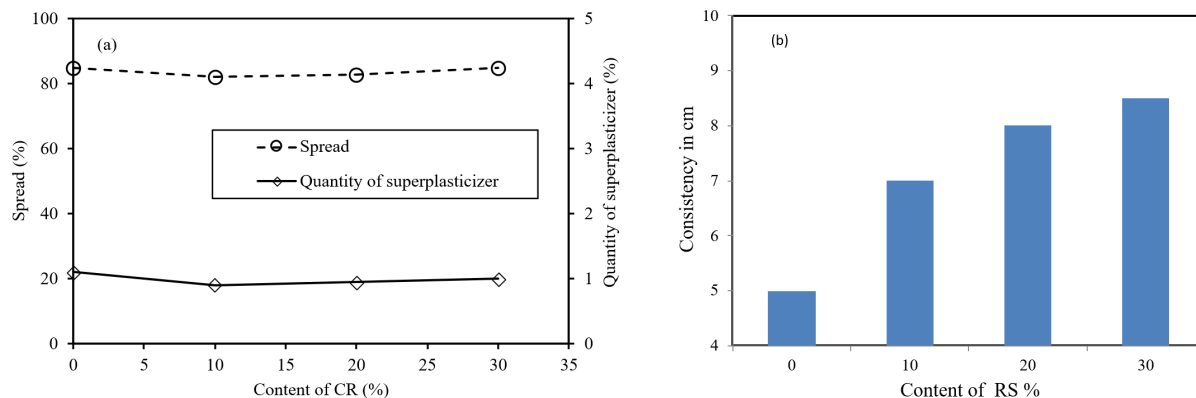


Figure IV.1. Variation of mortar spread (a) and consistency (b) with SP dosage as a function of CR content.

According to figure IV.1 it is clear that the incorporation of DPF increased the demand for SP. Consequently, a dosage of 1% was adopted to ensure proper dispersion of the constituents and to facilitate the flowability of the fiber-reinforced mortar.

Overall, the use of SP proved effective in maintaining satisfactory workability of the mortars containing CR and DPF, ensuring homogeneous mixtures and suitable casting conditions.

IV.3.2. The influence of varying CR percentages on the particle-size distribution of NS

Table IV.1 presents the FM values of the different sand mixtures used in this study. The results show that the FM increases progressively with the increase in CR content, rising from 1.8 for CR0 to 2.06, 2.17, and 2.3 for CR10, CR20, and CR30, respectively. This progressive increase shows that the FM is corrected and improved with the addition of CR, approaching the recommended range of 2.2 to 2.8 for fine aggregates.

Table IV.1. *Fineness modulus of different sands used.*

Sample	FM
CR0	1.8
CR10	2.06
CR20	2.17
CR30	2.3

Figure IV.2 illustrates the influence of varying CR percentages on the particle-size distribution of NS. The granulometric curves show that increasing the CR content slightly shifts the distribution curve, indicating a gradual modification of the aggregate grading. Despite this variation, all mixtures maintain a relatively continuous grading, which is favorable for mortar production.

Overall, the incorporation of CR slightly modifies the particle-size distribution of NS while improving the FM, leading to a granulometric composition that remains suitable for mortar mixtures.

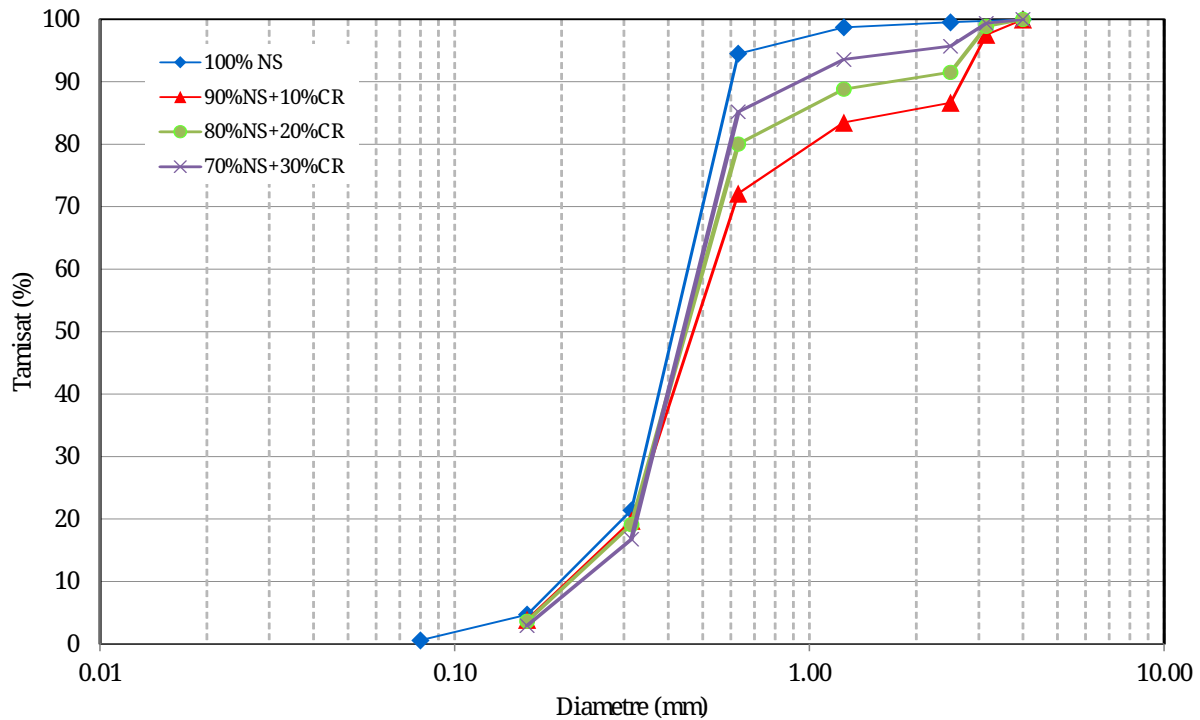


Figure IV.2. Particle-size distribution curves for different CR percentages.

IV.3.3. Apparent and absolute densities

According to figures IV.3 and IV. 4 it is clearly shown that fresh-state bulk density decreases as the substitution rate of CR increases. This trend is expected, as the bulk density of CR is lower than that of NS. This reduction is primarily attributed to the higher P which leads to an increase in void content and reduce the overall compactness of the mix. Additionally, variability in the quality of CR can lead to fluctuations in the mortar's bulk density. Similar results are observed by Bouldoum et al. [80], which confirms that the use of recycled concrete sand reduces the overall density of mortar mixes.

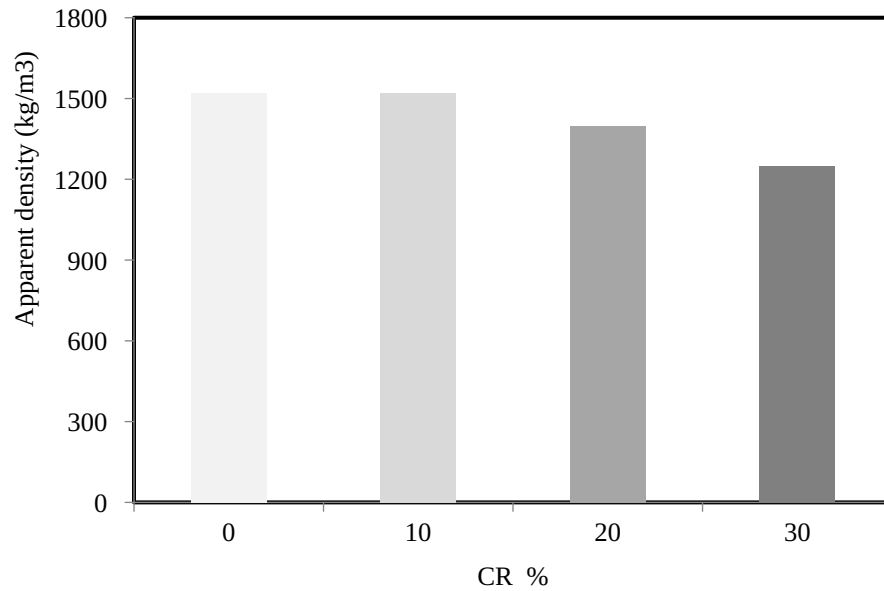


Figure IV.3. Apparent density for different CR percentages.

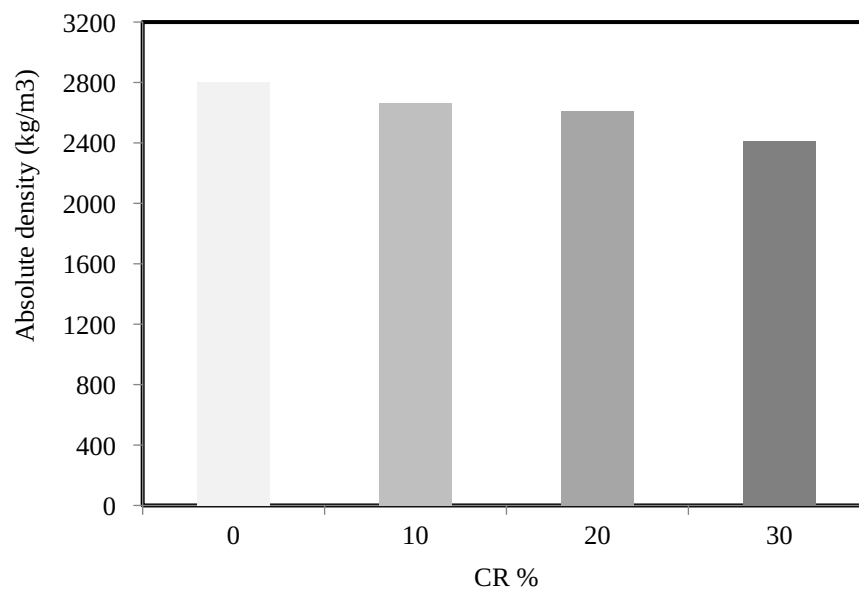


Figure IV.4. Absolute density for different CR percentages.

IV.4. Hardened state

IV.4.1. Mechanical properties

IV.4.1.1. Compressive strength

The figure IV.5 shows the evolution of CS with curing age for mortars containing different percentages of CR. As expected, strength increases over time for all mixtures due to cement hydration. However, the substitution of CR leads to a reduction in strength at all ages, and this decrease becomes more significant as the CR content increases. The control mixture CR0 exhibits the highest values, while the 30% CR mixture shows the lowest. This behavior is mainly attributed to the low stiffness of rubber particles, their weak bonding with the cement matrix, and the increase in P, which reduces the overall compactness of the material.

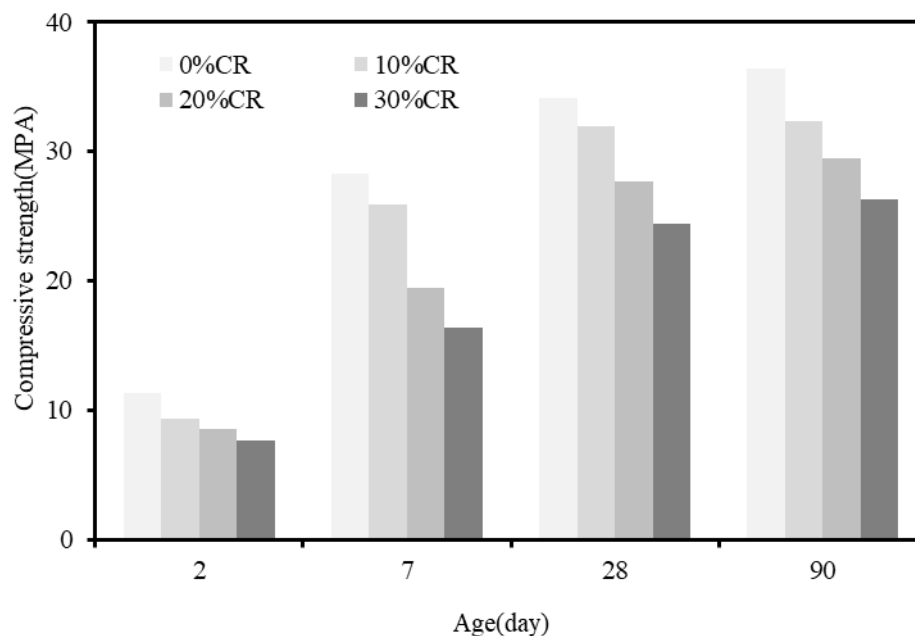


Figure IV.5. Effect of CR content on the CS of mortar.

Despite this reduction, mixtures with less than 30% of CR content still maintain acceptable strength levels for certain applications.

Nugroho et al. [81] showed that the CS of traditional mortar with no waste tire rubber powder (0% WTRP) is significantly higher than that of mortar containing 30%. This reduction in strength is consistent with previous findings by Faizah et al, who suggested that the decrease in CS is mainly due to the weak interfacial bond between rubber particles and the surrounding cement matrix. The chemical makeup of WTRP, which includes hydrophobic and non-polar substances, interferes with the hydration process and weakens adhesion within the composite structure. According to experimental results, the mortar with no WTRP achieved average CSs of 8.102 MPa at 7 days, 10.178 MPa at 28 days, and 14.747 MPa at 56 days. In contrast, the mortar with a 30% WTRP substitution showed CSs of only 3.941 MPa at 7 days, 3.962 MPa at 28 days, and 5.745 MPa at 56 days. The findings suggest that incorporating rubber powder from scrap tires adversely affects the mechanical properties of mortar, particularly its CS. While this approach supports sustainability by recycling rubber waste, it may limit the structural use of the resulting mortar unless further enhancements, such as surface treatments or hybrid modifications, are applied.

IV.4.1.2. Flexural strength

The figure IV.6 represents the evolution of FS as a function of curing age for mortars containing different percentages of CR. The results show that FS increases with curing age for all mixtures, due to the progressive development of the cement matrix. However, a gradual decrease is observed with increasing CR content, with the control mixture CR0 exhibiting the highest values and the 30% CR mixture the lowest. In the case of CR0, the presence of DPF plays a beneficial role by improving the flexural behavior. The fibers act as crack-bridging elements, delaying crack propagation and enhancing the post-cracking resistance of the mortar. This results in improved toughness and a less brittle failure mode compared to plain mortar.

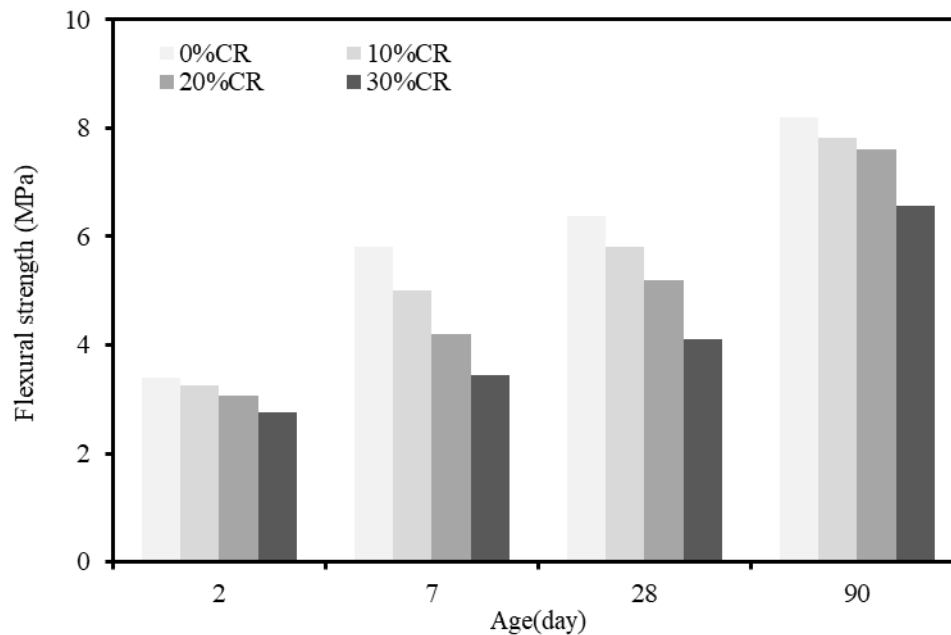


Figure IV.6. Effect of CR content on the FS of mortar.

With the incorporation of CR, although the overall FS decreases, the combined effect of fibers and rubber contributes to improved deformability. Rubber particles increase the material's elasticity, while fibers help control crack development. This synergy leads to a more ductile behavior, which is advantageous in applications where resistance to cracking and energy absorption are required. Even so, the reduction in strength, the obtained values remain acceptable even at higher substitution levels, confirming that these mortars can be effectively used in non-structural applications requiring enhanced flexibility and crack resistance, such as coatings, repair mortars, and lightweight elements.

Shen et al. [82] discovered that CR boosts the FS, toughness, and ductility of concrete, especially at moderate replacement levels. Rubber particles function as crack arrestors and aid in energy dissipation during loading, enhancing resistance to flexural and impact loads. Improvements in FS of 15–20% have been observed with the addition of rubber fibers. In rubberized mortars, substituting sand with 30% CR has resulted in a 10–12% decrease in drying shrinkage while maintaining adequate FS, suggesting its suitability for applications that require crack resistance and dimensional stability [83].

IV.4.2. Durability parameters

IV.4.2.1. Weight Loss

Figure IV.7 shows the evolution of WL as a function of curing age for different percentages of NS substitution by CR (0%, 10%, 20%, and 30%). It can be observed that the WL decreases with increasing age for all mixtures, particularly at early ages between 0 and 14 days, after which it tends to stabilize.

It is also clear that as the substitution rate increases from 0% to 30%, the WL decreases progressively. CR0 exhibits the highest values of WL throughout the curing period, whereas mixtures containing higher substitution percentages show lower values. This behavior can be explained by the incorporation of CR particles and DPF within the mortar matrix. These materials may contribute to modifying the internal structure of the composite by limiting the detachment of particles and improving the cohesion of the matrix. Furthermore, the presence of CR additions may influence the pore structure of the mortar, which can reduce material degradation and consequently limit WL over time. As the cement paste sets, the water that aids in workability is held within its capillary pores. Eventually, this water evaporates, leading to a reduction in the weight of mortars and concrete. Bouldoum et al [80], observed that recycled concrete sand speeds up this weight reduction due to its high P and the presence of residual mortar, which becomes completely saturated with water during mixing. The most significant WL occurs in the initial days and levels off after the second week. This phenomenon is likely due to the evaporation of moisture from the cementitious matrix. Other researchers [84-86] have reported similar findings, confirming that the use of recycled aggregates in concrete markedly increases WL.

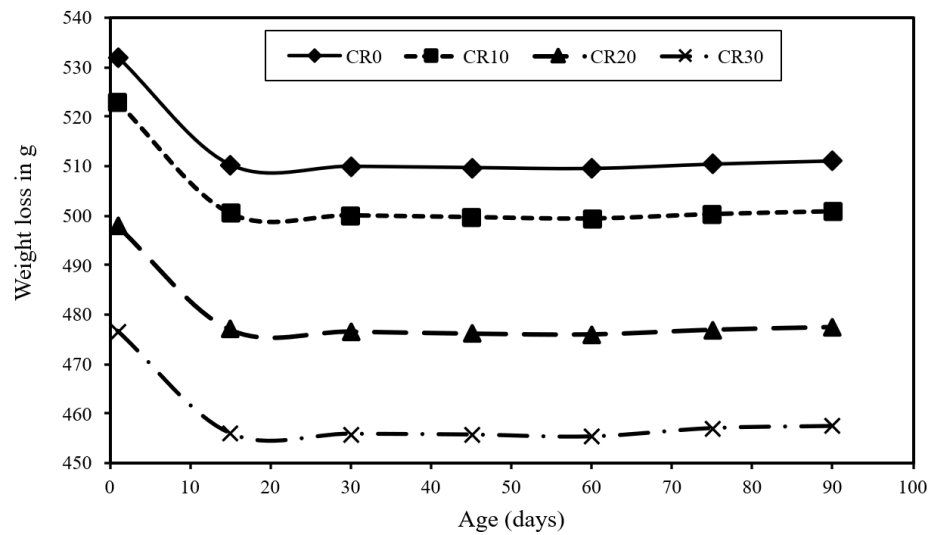


Figure IV.7. Effect of CR content on the WL of mortar specimens

IV.4.2.2. Sorptivity

Figure IV. 8 presents the variation of capillary water absorption as a function of the square root of time for mortars containing different percentages of CR. As expected, the measured values increase with time for all mixtures, reflecting the progressive penetration of water through the pore network of the cementitious matrix.

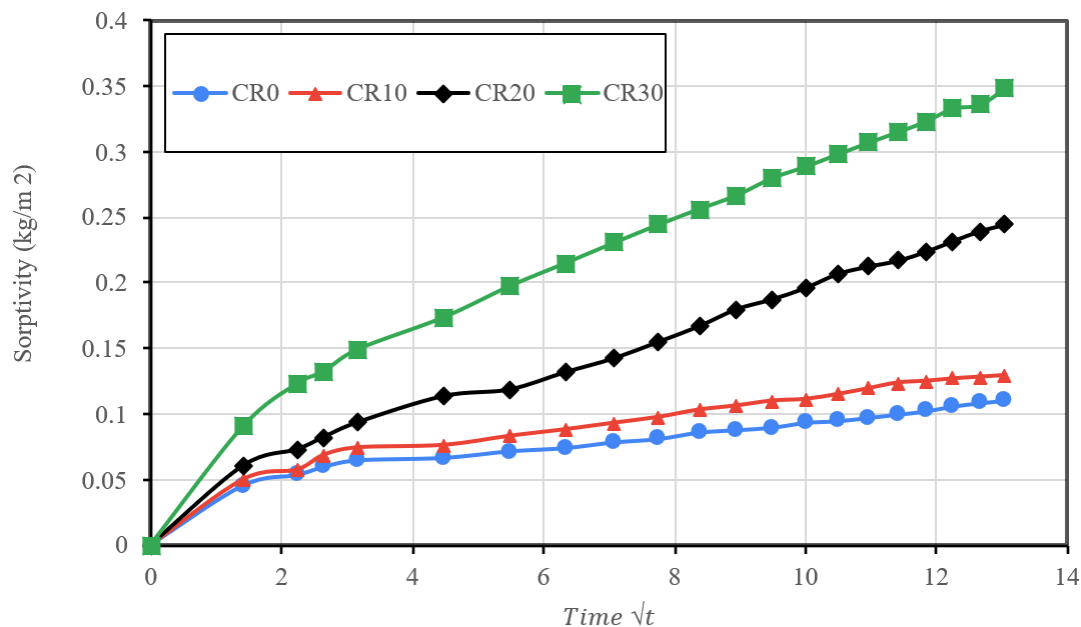


Figure IV.8 Variation of S as a function of time for mortars containing different percentage of CR

A clear influence of CR incorporation can be observed. CR0 exhibits the lowest values over the entire testing period, indicating a relatively denser microstructure. The introduction at 10% of CR leads to a slight increase of 16 % compared to the CR0. This increase becomes significantly more pronounced for CR30, which presents the highest values among all mixtures. For CR20, the results remain higher than those of the control mixture but slightly lower than those obtained for CR30.

Based on result obtained by Bušić et al. [87], it can be noted that incorporating WTR into both self-compacting concrete (SCC) and conventional concrete leads to an increase in WA. This phenomenon is due to the air entrapment in the interfacial transition zone (ITZ) between the rubber particles and the cement paste, as well as the irregularities of the rubber particles, such as their rough surfaces. Additionally, raising the water-to-powder ratio can further enhance WA.

IV.4.2.3. Water absorption by immersion

Figure IV.9 shows how WA by immersion changes at 28 and 90 days based on the CR content. It highlights a slight increase in absorption at 10% CR compared to CR0, followed by a minor decrease at 20% CR, and then a notable rise at 30% CR after 28 days. After 90 days, all mixtures exhibit an increase in WA.

This behavior is closely related to the evolution of P in the mortar matrix. CR is hydrophobic in nature and does not absorb water. However, the addition of CR modifies the internal structure of the mortar by increasing the volume of voids and reducing the interfacial transition zone between the rubber particles and the cement paste. These breaks allow water to penetrate into the material. Furthermore, the inclusion of DPF provides WA ability due to its natural porous structure and moisture absorption and retention capacity. The fibers could thus provide preferential pathways for water movement, in particular when associated with high CR contents. This effect becomes pronounced as the matrix becomes less compact.

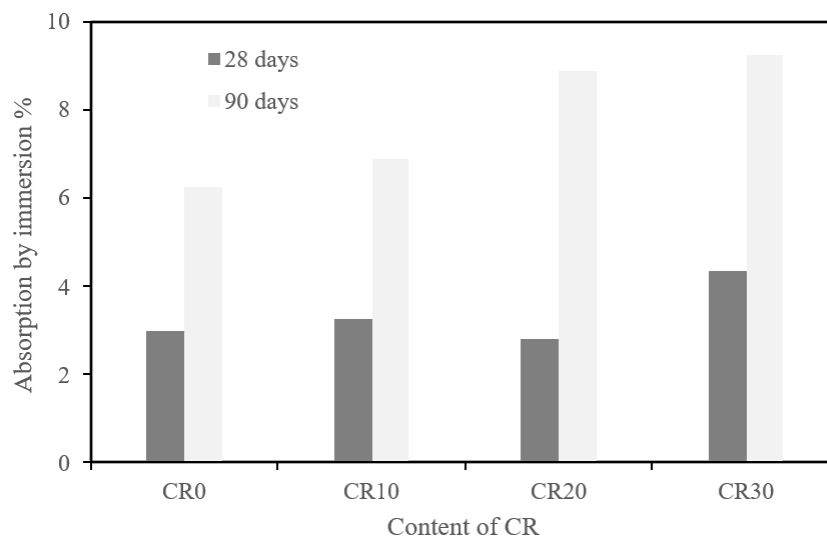


Figure IV.9. Effect of different percentages of CR on WA.

As reported by Syafiqah et al. [88], CR is characterized by its low ability to absorb water. Therefore, when larger CR particles are used in combination with a higher water–cement ratio in

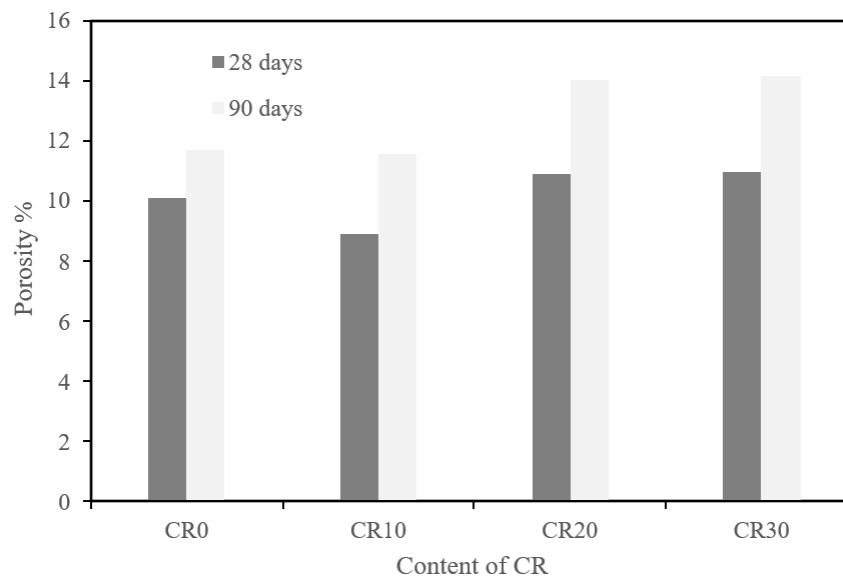
concrete, the result is an increase in the concrete's capacity to absorb water, which subsequently enhances its P.

IV.4.2.4. Porosity

The figure IV.10 illustrates how P changes with varying levels of CR in mortar mixtures. It is evident that at a 10% CR content, P slightly reduces compared to CR0, indicating an improvement in particle arrangement and matrix compaction at lower levels of substitution. However, when the CR content exceeds this percentage, P progressively increases for CR20 and CR30.

The reduction observed at 10% CR may be attributed to a more favorable particle arrangement, leading to a denser microstructure. However, at higher substitution levels, the incorporation of CR increases the volume of voids within the mortar due to its low density, irregular shape, and weak adhesion with the cement matrix. These characteristics promote the formation of interfacial voids and reduce the compactness of the granular skeleton.

The presence of DPF may also influence the pore structure. Although they contribute to crack bridging and improve the toughness of mortar, their rough surface texture and WA capacity can locally increase P, particularly when combined with high CR contents. Consequently, the combined incorporation of CR and DPF tends to increase the overall pore volume at high substitution rates.



FigureIV.10. Effect of CR content on the P of Mortar Mixtures.

According to the research by Marie [89], Bisht and Ramana [90], and Bignozzi and Sandrolini [91], incorporating a high-water content in the mixing of CR with concrete can result in concrete with low strength and numerous pores. Additionally, the P of the concrete rises as the proportion of CR increases.

IV.4.2.5. Carbonation

The figureIV.11 shows the C test performed on mortar specimens with different percentages of CR after 28 and 90 days of exposure. The phenolphthalein indicator produced a uniform pink coloration throughout the entire cross-section of all specimens, indicating the absence of a carbonated zone. This confirms that the cementitious matrix maintained a highly alkaline environment and that carbon dioxide penetration remained very limited for all mixtures, including those containing high CR contents.

These results suggest that the incorporation of CR and DPF did not significantly affect the C resistance of the mortars at early ages. Despite the increase in P observed in some mixtures, the internal microstructure remained sufficiently compact to limit CO₂ diffusion.

Chapter IV :**Results and Discussions**

It should be noted that a period of 28 days, and even 90 days, is generally not sufficient to fully evaluate C behavior under natural exposure conditions. However, the present study was conducted under accelerated C conditions, which intensify CO₂ penetration and provide a preliminary assessment of durability performance in a shorter time. Therefore, the obtained results can be considered promising, demonstrating the potential durability of these eco-friendly mortars against C.

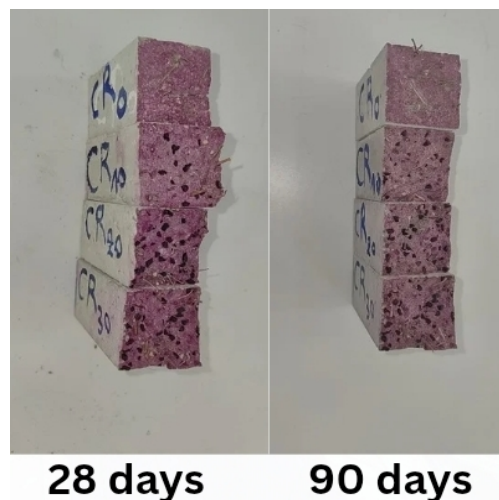


Figure IV.11. C depth of mortar with different mix proportions and different ages.

Mallek et al. [92] demonstrated that the depth of C increases with prolonged exposure to CO₂. Additionally, it has been observed that the level of C decreases as the CR content increases. Specifically, when the CR concentration is raised from 0% to 15%, there is a reduction in C depth by 3.0%, 5.6%, and 4.8% after 30 to 45 days of CO₂ exposure. This phenomenon may be attributed to the hydrophobic nature of CR, which slows down the C process. Rubber is known for its hydrophobic properties, meaning it does not easily interact with water. This characteristic can limit the amount of liquid CO₂ around these CR particles, thereby reducing the depth of C.

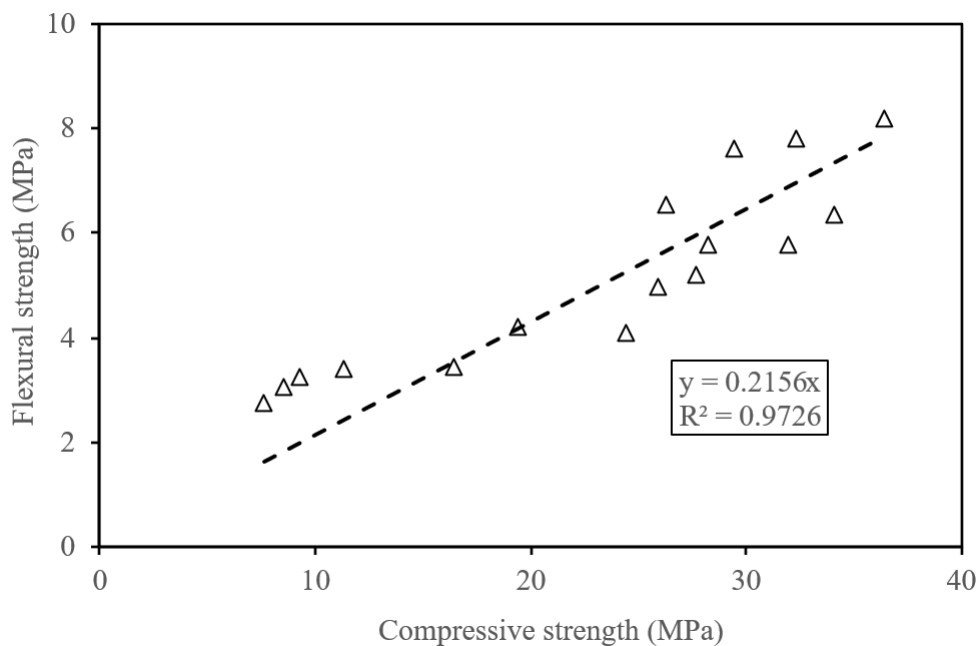
IV.4.3. Correlation between several parameters

IV.4.3.1. Correlation between CS and FS

As depicted in Figure IV.12, a strong relationship between the CS and FS of mortar mixes with different levels of CR aggregate replacement. The linear correlation clearly indicates a positive

association between CS and FS. With the coefficient of determination, R^2 , approaching 1, a strong correlation between these strengths is evident. Consequently, the linear equation is very reliable for predicting FS based on CS.

A recent study done by Boubekeur et al. [93] where they investigated the effect of recycled aggregate pavement (RAP) on the properties of SCC, found the same results and discovered that for every SCC sample, an increase in Sc leads to a rise in Sf. Here, Sf denotes the FS, while Sc represents the CS.



FigureIV.12. Correlation between CS and FS.

IV.4.3.2. Correlation between WA and P

The relationship between WA and P can be observed in Figure IV.13. Through multiple comparisons of the experimental data, we were able to establish a strong linear correlation that demonstrates the reliability of the experimental values of the P and WA of mortar mixes. This was validated with a correlation coefficient of 0.9385. This suggests that as P rises, WA also increases. The substantial coefficient of determination highlights a strong link between these properties, affirming that a rise in pore volume boosts the mortar's ability to absorb water.

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According to Singh et al. [95], the relationship between P and WA in concrete constructed from recycled aggregates such as recycled asphalt aggregates (RAA) or concrete waste shows that as P rises, so does WA. The findings show that adding RAA to concrete increases its P since the material may have irregularities and porous areas, which increases the voids in the concrete matrix.

As a result, mixes with larger percentages of RAA have a better capacity to absorb water. For instance, samples created with 100% RAA exhibited higher absorption rates than those made with NA, proving the connection between WA and P.

Lower P, which results in less WA, is linked to improved resistance to chemical and physical attacks in terms of durability, according to studies by Ali Kouadri et al [94]. Because of their increased P, mixes with higher RAA percentages originally absorbed more water, however, prolonged curing at higher temperatures allowed for matrix densification, which decreased WA and increased the concrete's durability. These findings imply that controlling P is essential to creating long-lasting concrete, particularly in humid or aggressive environments.

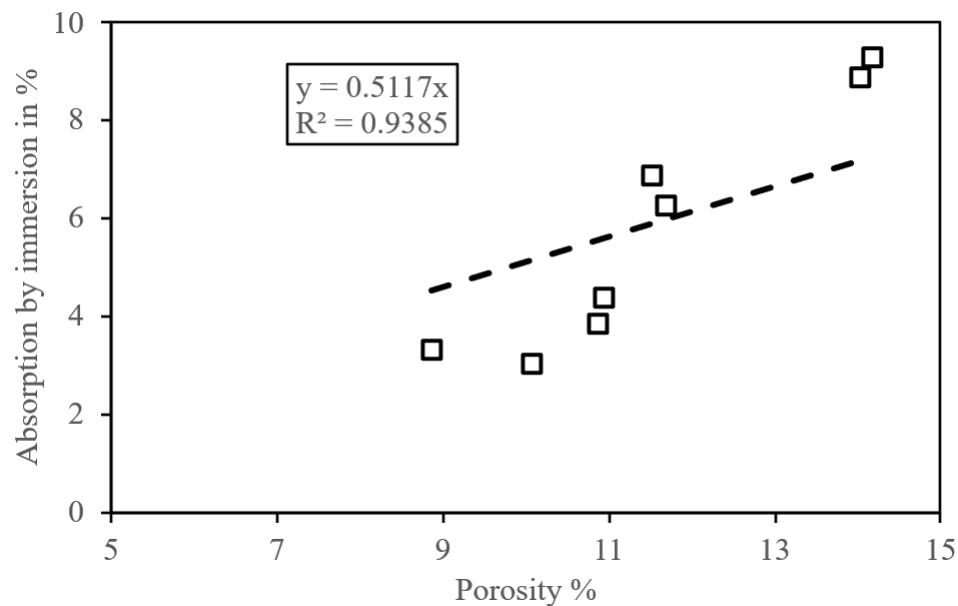


Figure IV.13.Correlation between WA and P.

IV.4.3.3. Correlation between CS and WA

The CS is strongly inversely proportional to WA in mortar mixes that have varying percentages of CR aggregate replacement, as indicated by an R^2 value of 0.8298, shown in figure IV.14. The two parameters are strongly correlated, as demonstrated by the high coefficient of determination. The inverse relationship between CS and WA observed in the present study aligns with the findings of Barik et al. [96], who reported that incorporating recycled fine aggregate significantly influenced both the CS and WA of mortar mixtures. An increase in recycled aggregate content led to higher WA and lower CS, attributed to the porous nature of the recycled particles.

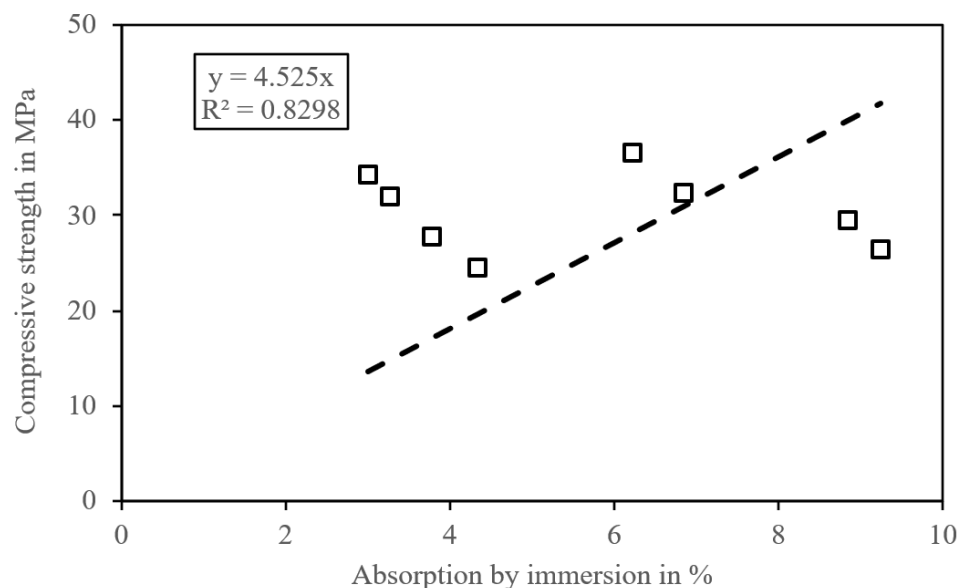


Figure IV.14. Correlation between CS and WA.

IV.4.3.4. Correlation between CS and P

A linear relationship between P and CS was found, as shown in Figure IV.15, with an R^2 value of 0.9516. There is a relatively strong correlation between the two parameters, as indicated by this coefficient near 1. Also, the microstructure of cementitious matrix affects directly the mechanical properties of mortar, when the number of the pores increase the CS decrease significantly for all mixtures. Same results were obtained in a study conducted by Boubekeur et al. [93] where they

found that SCC containing recycled aggregate pavement (RAP) present the similar behavior with the mortar studied where they demonstrated the linear relationship between P and CS.

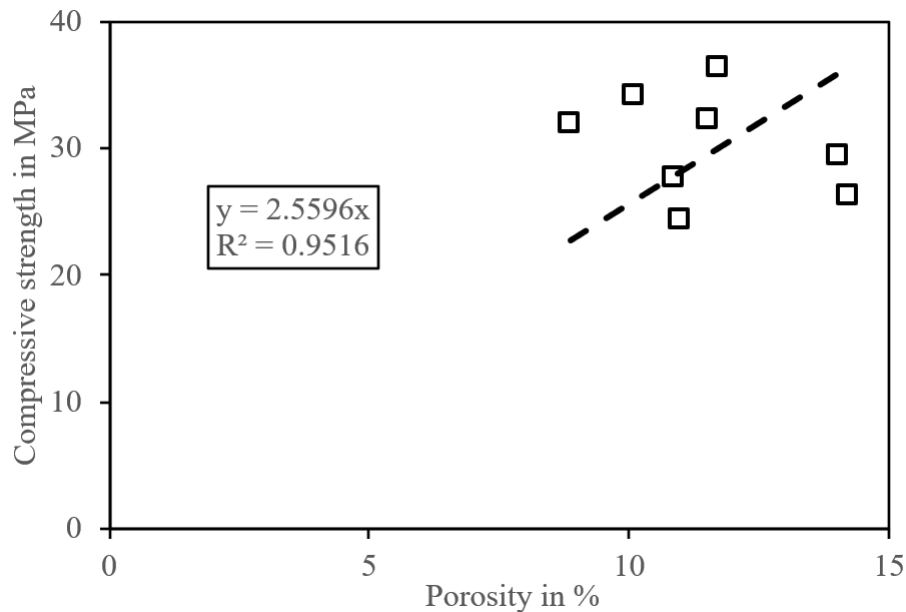


Figure IV.15. Correlation between CS and P.

IV.5. Conclusion

This research evaluates the effect of CR on the properties of a DPF reinforced mortar; the following findings can be drawn:

- In comparison to the reference mortar with DPF (CR0), adding CR led to a gradual reduction in CS by about 5%, 8%, and 24% for CR10, CR20, and CR30, respectively. Despite this, the MP remained acceptable for non-structural and lightweight construction applications.
- The WL of the mortar mixtures increased by about 2%, 6.5%, and 10% in comparison with CR0 for CR10, CR20, and CR30, respectively. This implies that the higher the CR, the more likely moisture is to evaporate.
- Initially, P decreased by approximately 12% with 10% CR replacements, indicating improved particle dispersion at lower replacement levels. However, further increases in CR

content led to elevated P, rising by about 7.81% and 8.71% for CR20 and CR30, respectively, indicating the formation of a more porous microstructure.

- The increase in CR content considerably boosted the S, resulting in increases of approximately 17%, 121%, and 214% for CR10, CR20, and CR30, respectively, compared with the DPF-reinforced reference mortar. This behavior is due to superior capillary water transport caused by the higher pore connectivity of the CR-containing mixtures.
- The optimal CR replacement range based on the tested properties is 10% by volume of NS, as CR10 exhibited the most balanced combination of fresh-state, mechanical, and durability properties.

GENERAL CONCLUSION

This dissertation has thoroughly explored the use of CR and DPF in cement-based mortar as an eco-friendly strategy to reduce environmental harm and enhance the use of industrial and agricultural waste. From the experimental methods employed and the results achieved, several significant conclusions can be reached:

- The use of DPF results in a reduction in workability, this reduction necessitates a certain dosage of SP to achieve the desired workability level.
- CR derived from waste tires can partially substitute NS in plastic mortar to make it more sustainable and environmentally friendly.
- Using CR with DPF in mortar significantly affects various properties, and its impact can be both beneficial and challenging depending on the CR content.
- The CS and FS of mortar containing CR decreased substantially as the percentage of NS replaced with CR increased.
- The combination of 1% of DPF and 10% of CR yielded the most optimal results.
- CR can be used in several applications. However, to maintain appropriate mechanical properties, the substitution level should not exceed 10% by volume of NS.
- Based on the results of C test, the use of CR with 1% of DPF improves the C resistance of mortar.
- The strong correlations between different parameters shown by the correlation analysis demonstrate a close association between the microstructural features and MP of mortar.
- Higher CR content results in a more porous matrix and an increase in WA and S of mortar.
- It's recommended to use CR with precisely measured amounts for better mortar production, which allows the recovery, recycling and valorization of substantial quantities of CR.

PERSPECTIVES

Building on the existing research into the production of eco-friendly mortars from DPF and recycled tires CR as alternative material for sand, several potential areas for future research work

General conclusion

are proposed in this paper, seeking to expand the knowledge about the properties of such materials, find sustainable ways of producing them, and solve all the problems associated with incorporating recycled materials in construction works. Specifically, attention is paid to testing procedures that could allow analyzing the durability, microstructure, and resistance to external impacts of the materials under consideration in order to manufacture environmentally friendly material suitable for use according to green building standards. The required tests to be carried out within this research framework are the durability tests involving accelerated aging tests to assess resistance to sulfate attacks, resistance to chlorides, and acids, and freeze-thaw cycle test. The microstructure of samples is analyzed using X-ray diffraction (XRD) and thermogravimetric analysis (TGA). In addition, dynamic modulus of elasticity and impact strength tests, as well as abrasion testing are required. Finally, the environmental performance of the materials can be assessed by implementing life cycle assessment (LCA) followed by field-scale validation in order to measure ecological footprints. The aim of such future studies will be to optimize the utilization of CR-palm fiber composite material while at the same time fostering the development of circular economy and sustainable building materials.

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Combined Effect of Natural Fibers and Rubber Tire Waste on the Properties of Sustainable Mortars

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Abstract

Faced with the economic challenges caused by the rising cost of natural resources such as palm fibers and waste rubber from tires, as well as their depletion and the environmental impact of their production and use, the construction industry is turning toward biomaterials. This research aims to investigate the potential valorization of natural palm fibers and recycled rubber waste in the formulation of environmentally friendly mortars with a low ecological footprint. Palm fibers, abundant in arid regions and often considered agricultural residues, exhibit interesting mechanical properties that can enhance the toughness and ductility of cementitious materials. At the same time, the incorporation of rubber—used as sand-sized particles derived from the recycling of waste tires—helps reduce the mortar's density and improve its energy absorption capacity, while contributing to the sustainable management of a difficult-to-dispose waste stream. The study examines the combined effect of these two types of additives on the fresh and hardened properties of mortars, including workability, mechanical strength, and durability. The results show that an optimal amount of palm fibers improves cohesion and limits crack propagation, while rubber particles provide greater resilience and reduce the material's brittleness. However, excessive incorporation may lead to a significant decrease in compressive strength.

Keywords: Eco-Friendly, Palm fibers, Waste rubber, Natural Sand, Strength



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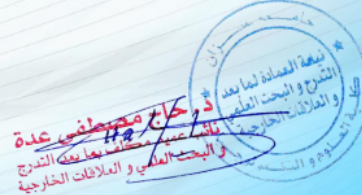
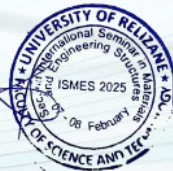
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