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Sujet

Optimization of the bending and compressing behavior of cementitious mortar reinforced with hemp fibers by using the response surface methodology (RSM)

Présenté et soutenu publiquement, le **07/06/2023**, devant le jury composé de :

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• Dedications

To my beloved father, who has been my guiding light and unwavering support throughout my academic career. Your love, encouragement, and belief in me have been the driving force behind my success. I am forever grateful for your sacrifices, wisdom, and unwavering faith in me. This achievement would not have been possible without your constant support and guidance. Thank you for being my rock and for instilling in me the values of hard work, perseverance, and determination. I hope my graduation makes you proud, as it is a testament to your unwavering love and support.

I love you, Dad.

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My dear friends;

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Abstract

In recent years, cementitious composite materials reinforced with vegetable fibers have gained increasing interest due to the need for sustainable building materials. Vegetable fibers are a hierarchical structure material that can be used as reinforcement in cement-based composites. This research work aims to investigate the use of vegetable fibers as reinforcement in cement-matrix composites for building applications. This research presents the development of construction bio-composites consisting of a cementitious matrix reinforced with *hemp* fibers and to optimize its behavior under compressive and 3-point bending tests. The *hemp* fibers were treated with different concentrations of soda solution and cut into different lengths before distributing them in mortar, to study the influence of these factors in mechanical tests. Then bio-composites of *hemp*/cement were elaborated with an experimental design using response surface methodology (RSM). The results obtained from mechanical tests after 28 days of curing were analyzed with the analysis of variance technic (ANOVA) to optimize the mechanical characteristics of the bio-composites developed. Next, these results have been converted into a mathematical model to understand the effect of changing factors on the mortar behavior using Design Expert software.

Keywords: *hemp* fiber, biocomposite, mechanical behavior, mortar, optimization.

• ملخص

في السنوات الأخيرة ، اكتسبت المواد المركبة الأسمنتية المقواة بالألياف النباتية اهتمامًا متزايدًا بسبب الحاجة إلى مواد بناء مستدامة. الألياف النباتية عبارة عن مادة هيكلية هرمية يمكن استخدامها كتعزيز في المركبات القائمة على الأسمنت. يهدف هذا البحث إلى التحقق من استخدام الألياف النباتية كتعزيز في مركبات المصفوفة الأسمنتية لتطبيقات البناء. يقدم هذا البحث تطوير المركبات الحيوية للبناء التي تتكون من مصفوفة اسمنتية معززة بألياف القنب ولتحسين سلوكها في ظل اختبار الانضغاط والانحناء ثلاثي النقاط. تمت معالجة ألياف القنب بتراكيز مختلفة من محلول الصودا وتم تقطيعها إلى أطوال مختلفة قبل توزيعها في الملاط بهدف دراسة تأثير هذه العوامل في الاختبارات الميكانيكية. ثم تم تطوير المركبات الحيوية القنب / الأسمنت مع خطة تجريبية باستخدام منهجية سطح الاستجابة (RSM). تم تحليل النتائج التي تم الحصول عليها من الاختبارات الميكانيكية بعد 28 يومًا من المعالجة باستخدام تقنية التباين (ANOVA) من أجل تحسين الخصائص الميكانيكية للمركبات الحيوية التي تم تطويرها. بعد ذلك ، تم تحويل هذه النتائج إلى نموذج رياضي لفهم تأثير العوامل المتغيرة على سلوك الملاط باستخدام برنامج Design Expert.

الكلمات المفتاحية: ألياف القنب ، المركبات الحيوية ، السلوك الميكانيكي ، الملاط ، التحسين.

Summary

General introduction

Cementitious composite materials reinforced with vegetable fibers have gained significant attention in recent years due to their potential to provide sustainable and eco-friendly alternatives to traditional construction materials. These composites have shown promising mechanical properties, including high compressive and bending strength, making them suitable for various non-structural applications. The behavior of these materials under bending and compressive loads is of particular interest, as it can provide insights into their structural performance and durability.

The selection of fibers for fiber-reinforced cementitious composites mainly considers the tensile strength, modulus of elasticity, elongation, and bonding with the matrix. Several studies have investigated the mechanical properties of fiber-reinforced cementitious composites, including the relationship between bending strength, compressive strength, dosage, and shrinkage. The test results show that unreinforced cementitious materials are well known for possessing high compressive strength while being weak under flexural and tensile loads. However, the addition of vegetable fibers can improve the tensile and flexural strength of these materials, making them more suitable for structural applications.

This work aims to develop and investigate the behavior of cementitious composites reinforced with vegetable fibers under bending and compressive loads. The study will focus on the mechanical properties of these materials also the chemical treatment of the vegetable fibers. The research will also explore the effect of treated fiber content, the way that it has distributed in the matrix, on the mechanical properties of these composites.

The results of this study can provide valuable insights into the structural performance and durability of these materials, which can be useful for the development of sustainable and eco-friendly construction materials that can meet the demands of modern construction while reducing the environmental impact. Overall, this research will contribute to the growing body of knowledge on the behavior of cementitious composites reinforced with vegetable fibers.

The research findings can be useful for engineers and researchers working in the field of sustainable construction materials, as well as for stakeholders interested in promoting sustainable and eco-friendly building practices.

This work is structured of four chapters as follows:

The first chapter is devoted to a bibliographic study, which focuses on some generalities

about composite materials and a review of the characteristics of vegetable fibers, highlighting their main physical, thermal, and mechanical characteristics, including their advantages and disadvantages. with an overview of the manufacturing processes of composite materials.

The second chapter describes specifically the hemp fibers and in detail the materials used, the treatment of the fibers with different percentages of NaOH, as well as the development of bio-composites based on an experimental design using the response surface method (RSM) and presents the tests carried out in flexion and compression, as well the results obtained.

The third chapter is dedicated to identifying the experimental design methods to build and bring understanding closer to some explanations and concepts related to it.

The fourth chapter is concerned with the analysis of experimental data gained from 3-point bending and compression tests performed on bio-mortars developed by the RSM, to evaluate their bending and compression behavior and establish their mechanical characteristics after 28 days of cure. An ANOVA analysis was established to find the optimal choice of bio-mortar production parameters: length (mm), NaOH (%), and fiber volume (%), resulting in optimization of the mechanical characteristics of bio-composites exposed to bending and compressive stress.

1

Overview of composite materials and their applications

1.1 Introduction

In this chapter, we describe the composite material with various fibrous reinforcements with specific attention on vegetable fibers which represent the trend of industry anddevelopment, due to their abundance and distinctive characteristics. To comprehend the behavior of the resulting material and clarify the production procedures, we describe in this study the various elements that make up composites as well as the link between reinforcements and matrices. Furthermore, we shall go to great lengths when conceptually describing the fiber's structure, traits, chemical composition, extraction techniques, and applications.

1.2 Definition

A composite material is defined as a combination of at least two separate parts with a high degree of adaptability. The linked components' strengths work in concert to create a material with enhanced mechanical, thermal, electrical, and/or physical-chemical performance. Typically, a specific requirement drives the development of composite materials with one or more of these unique features. Thus, by combining a reinforcement (fibrous) and an organic resin, heterogeneous materials can be made that, for instance, enable bulk reduction of a part while enhancing its mechanical qualities. This material's versatility makes it an irrefutable asset, which accounts for its expanding use in a wide range of industries, especially sports and leisure, construction, and transportation (by air, sea, and train).

The composite material is made up of a reinforcement, which contributes to the mechanical properties of the part, and a binder called a matrix. The matrix's role is to transfer the flow of forces between the plies and guarantee environmental resistance (corrosion, wet aging), and temperature resistance while also maintaining cohesiveness between the reinforcement's parts. There are several composite materials available today that can be categorized using various

criteria **[1]**. The composition of the matrix is one of the factors that enable the division of the composites into three major families: Organic matrix composites (OMC), such as organic polymers (thermoset or thermoplastic resin), ceramic matrix composites (CMC), reserved to high-temperature applications and metal matrix composites (MMC). These materials can also be categorized based on the type of reinforcement used, which is described below, or based on the goal that is being pursued by their use. If cost optimization is the primary goal, one will refer to composites as having a "big diffusion." On the other hand, "high-performance" composites are those that are used to enhance mechanical or thermal characteristics to a reduction in weight **[2]**.

1.3 Constituents of composite materials

1.3.1 Matrix

The matrix (Fig. 1.1) is one of the fundamental components of composite materials, which has three major purposes: it distributes the mechanical stress across the entire reinforcement, promotes fiber cohesion for greater homogeneity, and protects the reinforcements from the outside environment (mechanical or thermal shocks). The polymer resins most used in composite materials are thermosetting resins (polyester, polyurethane. . .) and thermoplastic resins (PVC, PS, PET...)**[3]**.

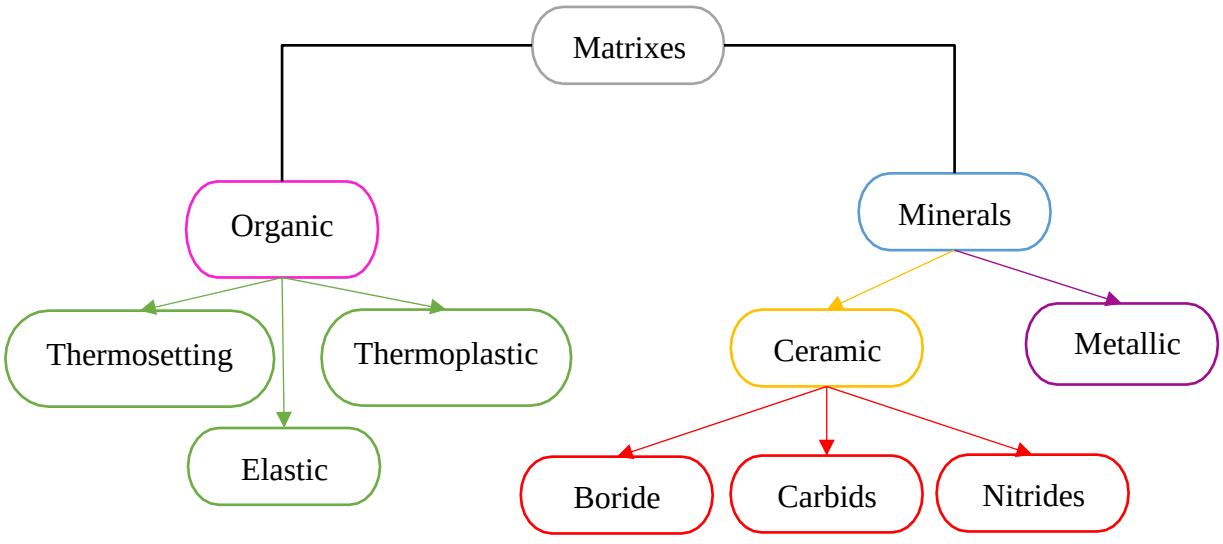


Fig. 1.1 Different types of matrixes **[1]**

1.3.1.1 Polymer matrix

Polymers have a significantly more complex structural structure compared to metals or ceramics. They are inexpensive and simple to process. Polymers, on the other hand, have reduced strength, modulus, and temperature use restrictions. The characteristics of polymers can deteriorate as a result of extended exposure to UV radiation and some solvents. Polymers are typically poor conductors of heat and electricity due to their predominance of covalent bonding and are generally more resistant to chemicals than metals **[4]**.

- **Thermoplastic polymer**

The linear chains that make up the thermoplastic matrices can change while they are still molten. The thermoplastic matrices are heated in the majority of production procedures, thenthe finished product is shaped using molding, injection, extrusion, or thermoforming before being cooled to maintain its shape. This procedure can be undone. Thermoplastics come in a broad variety of forms today, each with a unique set of intriguing features. For usage in a variety of products, they can be made as clear as glass, as rigid as metal and concrete, or as flexible as rubber. They do not oxidize, exhibit strong resistance to corrosion, and function superbly as thermal and electrical insulators. Thermoplastics are an excellent material for many applications due to their lightweight, great mechanical strength, and resistance to environmental influences. Common thermoplastic resins used as a matrix in composites are **[5]**:

- Polyethylene (PE);
- Polystyrene;
- Polyamides (poly hexamethylene sebacic);
- Polycarbonate (PC);
- Polysulfone;

- **Thermosetting polymer**

They are typically liquid at room temperature, but during the application, under the influence of heat and a substance called a hardener, they solidify. Cooking, a chemical change that creates a strong three-dimensional link between the molecules, transforms them in this way. The substance treated as a result of this irreversible procedure becomes infusible and insoluble in most solvents (alcohols, ketones, and hydrocarbons). They are useful for molding big parts with short, long, or woven fibers because they are generally more rigid than thermoplastic matrices, resist creep better, and are less brittle. Polyesters, phenoplasts, epoxy resins, polyurethanes, and

polyimides are the most widely used thermosetting matrices. Extremely used thermosetting are **[5]**:

- Epoxies;
- Polyesters (Polyethylene terephthalate);
- Polyurethanes;
- Phenoplasts;
- Silicones;

- **Elastomers**

Elastomers are characterized by long molecular chains that have been folded back on themselves to make up an elastic material at rest. The molecules can move about and change shape in response to a limitation. The base material is vulcanizedto give it good elasticity. It is a process of hardening that leaves the molecular chains'fluidity while producing a more or less rigid three-dimensional network. During vulcanization,the elastomer receives introductions of sulfur, carbon, and numerous chemical agents. Synthetic rubbers can be produced using various formulations for a variety of purposes. Elastomers are used to make tires, certain insulators, shoe bottoms, and cushions **[5]**.

1.3.1.2 Ceramic matrix

It is a material that exhibits outstanding thermal qualities as well as better mechanical capabilities, addressing the drawbacks of monolithic ceramic (such as toughness) and offering additional advantages. Turbine disks, combustor linear, and turbine aerofoils are a few examples of the potential uses of CMCs in aviation, which are often found in the hot portion of aero engines **[6]**.

1.3.1.3 Metal matrix

These materials offer several advantages over monolithic ones and are made of an oxide, nitride,or carbide matrix reinforced with aluminum or titanium. However, they are less durable, more expensive, and more challenging to make. Potential uses include heavily loaded surfaces like floor supports, turbine fan blades, and helicopter rotor blades **[6]**.

1.3.1.4 Cementitious matrix (Mortar)

Mortar is one of the building materials used to connect the components, ensure the stability of the construction, and fill in the spaces between the building blocks, in most cases, mortar is created by combining sand, a binder (usually cement or lime), water, and additives. Employed

for any type of masonry work, including construction, plastering, and restoration **[7]**. According to their composition, there are 5 types of mortars **[8]**:

- **Cement mortar:** white or grey artificial cement of the Portland type is used. It is extremely resilient.
- **Lime mortar:** lime is hydraulically produced. It is more flexible and allows the walls to breathe despite being less resistant and waterproof than cement mortar.
- **Bastard mortar:** made with a cement and lime combination. It offers qualities that fall between those of lime mortar and cement mortar.
- **Refractory mortar:** Based on "molten", for fireplaces and barbecues.
- **Rapid mortar:** Based on "prompt" cement, for sealing.

1.3.1.4.1 Composition of mortars

Mortars are mainly made of binder, sand, and water, as described below **[8]**:

1. **The binders**

❖ **Cement**

Little, distinct grains of various materials are used to make cement, but they must all have statistically homogenous compositions. A continuous mass manufacturing process, in particular appropriate grinding and homogenization procedures, results in a high degree of homogeneity in all the cement's qualities.

❖ **Lime**

It is produced by calcining, or heating limestone to a high temperature. Based on the underlying material's composition. We find:

- Air lime is obtained from very pure limestone.
- Hydraulic lime is obtained from limestone containing silicates, aluminates, and magnesium compounds.

2. **The sand**

Sand is a loose sedimentary rock with a size range of 0.063 to 2 mm that is made up of tiny fragments from the breakdown of other rocks. They have two origins:

- **Natural**: Sea, Wadi, Sahara. This case represents the quasi-totality of the constructions.
- **Industrial**: from the crushing of quarry aggregates, and more rarely from the recycling of construction waste.

3. The water

The quantity of water in the mortar depends on how it will be used; a sprayed coating requires a lot more liquid than a mortar used to assemble breeze blocks. Too-dry mortar loses homogeneity and is challenging to use (it does not "stick"). Too-wet mortar does not dry well and is challenging to apply thickly (it sinks).

1.3.2 Reinforcement

The reinforcement serves as the skeleton for the mechanical efforts and helps to increase the rigidity and mechanical resistance of composite materials. It is displayed as filaments, either organic or inorganic (Fig.1.2), such as glass, carbon, aramid, and natural fibers (*flax*, *sisal*, ramie, *jute*, *hemp*, *abaca*, and *olive pomace*) **[3]**.

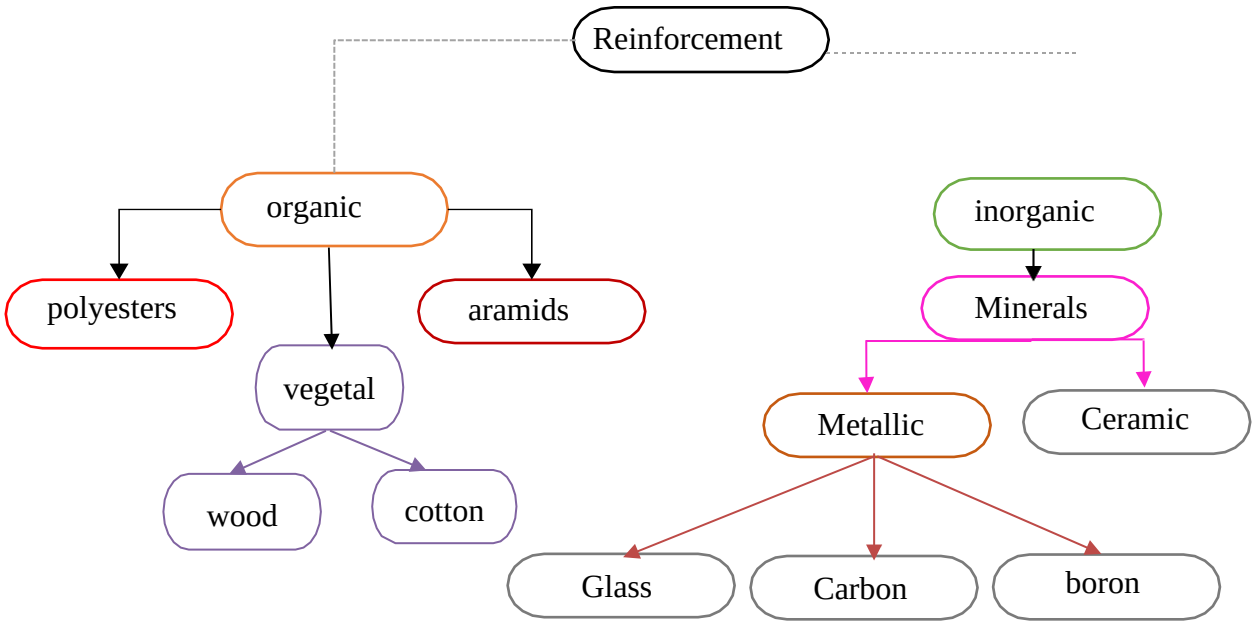


Fig.1.2 Different types of reinforcement **[5]**

1.3.2.1 Glass fibers

Glass fibers constitute the essential reinforcement used in composites due to their advantages which represent 90% of applications, they are employed in different forms such as longitudinal, woven mat, chopped fiber (distinct), and chopped mat (Fig.1.3), the form contributes to enhancing the mechanical and tribological properties **[1]**. There are many types of glass fibers, the famous and oldest type is the E-glass (electrical grade glass), and many other types including; A-glass or alkali glass, C-glass or chemical resistant glass, and the high strength R-glass and/ or S-glass.

All of these types resist tensile stress of about 7000 N/mm² and also have high physical, chemical, and mechanical properties as represented in (Tables 1.1, 1.2, and 1.3) **[1]**.

(a) **(b)**

Fig.1.3 Glass fibers reinforcement: **(a)** Unwound glass fibers; **(b)** Woven glass fiber **[3]**

Table 1.1 Physical properties of various classes of glass fibers **[1]**

Classes of GFs	Physical properties
A-glass	High durability, strength, electrical resistivity
C-glass	High corrosion resistance
D-glass	Low dielectric constant
E-glass	Higher strength and electrical resistivity
AR-glass	Alkali resistance
R-glass	Higher strength and acid corrosion resistance
S-glass	Higher tensile strength

Table 1.2 Chemical compositions (%) of glass fiber types **[1]**

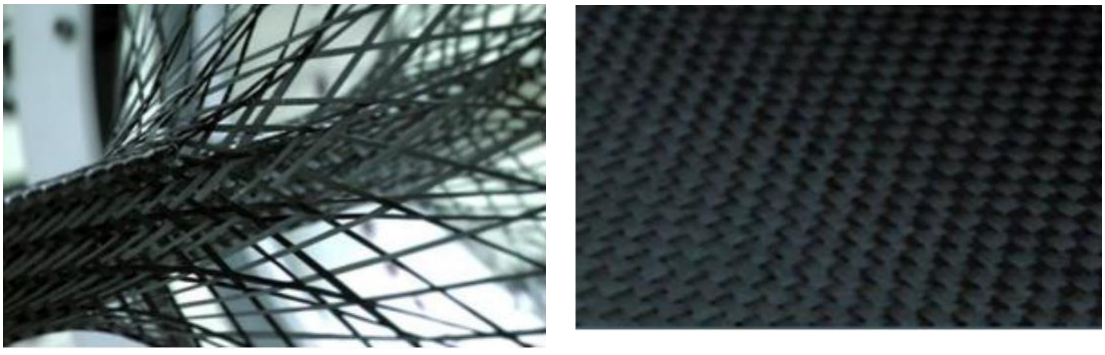
Type of GF	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	B_2O_3	CaO	MgO	Na_2O	K_2O
E-glass	55	14	-	0.2	7	22	1	0.5	0.3
C-glass	64.4	4.1	-	-	5	13.4	3.3	9.6	0.5
S-glass	65	25	-	-	-	-	10	-	-
A-glass	67.5	3.5	-	-	1.5	6.5	4.5	13.5	3
R-glass	60	-	-	-	-	9	6	0.5	0.1
EC-glass	58	12.4		-	-	23		-	-
AR-glass	61	1	-	-	-	5	1	14	3

Table 1.3 Mechanical and physical properties of GFs **[1]**

Type	Density (g/ cm^3)	Tensile strength GPa	Youngs modulus GPa	Elongation (%)	Coefficient of thermal expansion	Poison's ratio	Refractive index
E-glass	2.58	3.445	7.23	4.8	54	0.2	1.558
C-glass	2.52	3.310	6.89	4.8	63	-	1.553
S-glass	2.46	4.890	8.69	5.7	16	0.22	1.521
A-glass	2.44	3.310	6.89	4.8	73	-	1.538
R-glass	2.54	4.135	8.55	4.8	33	-	1.546
EC-glass	2.72	3.445	8.55	4.8	59	-	1.579
AR-glass	2.70	3.241	7.31	4.4	65	-	1.562

1.3.2.2 Carbon fibers

Carbon fibers known for their exceptional characteristics prepared from polyacrylonitrile (PAN) carbonization followed by graphitization offer high specific modulus and strength to the material, this is what makes it so widely used in several industries such as space construction, aeronautics, aircraft, and automobile. carbon marked by; High tensile strength with the ability to hold it at an elevated temperature, superior compressive strength, and rigidity **[5]**. Carbon fibers have various types of features with high strength and high modulus the most important and used is Polyacrylonitrile (PAN) offers an elastic modulus in the range of 2×10^6 - 4×10^6 N/mm^2, this modulus can ambit to three times from steel, the other classes of carbon represent a high modulus with low tensile strength (Table 1.4) and high thermal and electrical conductivities, all of these types are formed with PAN. The final product of carbon fibers may be produced as a finished product shaped as woven tubes or as a pre-product; involve short fibers, twisted or non-twisted yarns, and continuous filament tows (Fig.4.1) **[1]**.



a **b**

Fig.1.4 Digital images of **a)** unwound carbon fibers; **b)** woven carbon fiber **[3]**

Table 1.4 Mechanical properties of different types of carbon fiber tows **[1]**

Types	Ultra-high elastic modulus type(UHM)	High elastic modulus type (HM)	Intermediate elastic modulus type(IM)	Standard elastic modulus type(HT)	Low elastic modulus type(LM)
Tensile elastic modulus	600 GPa	350-600 GPa	280-350 GPa	200-280 GPa	200 GPa
Tensile strength	2 500 MPa	2 500 MPa	3 500 MPa	2 500 MPa	500 MPa

1.3.2.3 Aramid fibers (Kevlar)

Kevlar is a type of synthetic polymer of Poly-para- phenylen-terephtalamide (PPTA) (Fig. 1.5 and Fig.1.6) made up of a large chain of a large number of monomers tied to each other, stiff molecular structure, flame resistant and conserves its proprieties in high temperature, different types of Kevlar are available with excellent properties showed in (Table 1.5). Aramid is suitable for high-performance composite, used in applications that need lightweight, high strength, andstiffness such as ship hulls, bulletproof vests, and colling vehicles.

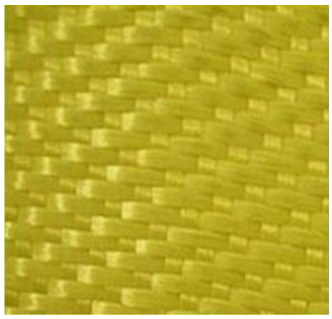


Fig.1.5 Woven Kevlar fibers **[1]**

Fig.1.6 Unwound Kevlar fibers **[1]**

Generally, aramid includes two groups; Meta-aramid; which is extremally good heat and radiation resistance, characteristic of excellent thermal. Used in the manufacturing of fire-retardant textiles, the Para-aramid is more strength and ductility used in high tensile strength applications, its fibers are more aligned in the long direction of the fibers, which differs from the Meta which is less or not aligned **[1]**.

Table 1.5 Properties of different types of Kevlar **[9]**

Yarn properties	Kevlar 29	Kevlar 49	Kevlar 100	Kevlar 119	Kevlar 129	Kevlar 149	Twaron
Tensile strength GPa	3.6	3.6-4.1	3.0	3.45	3.4	3.4	3
Elastic Modulus GPa	83	131	60	55	97	143	78
Elongation %	3.0	2,8	2.9	3.1	3.4	2.3	3.3
Density	1.44	1.45	1.44	1.44	1.45	1.47	1.44

1.3.2.4 Natural fibers

Natural fibers are the new generation of reinforcing composites that offer new advantages and efficiency, generated from natural resources which make them environmentally friendly, renewable, and biodegradable, they also present important mechanical properties such as stiffness and lightness. Additionally, natural fibers outperform synthetic fibers in terms of cost and processing capacity. The fact that they are a plentiful renewable resource also lessens reliance on both domestic and imported oil. Fibers can be divided into many categories based on their origin (natural, synthetic, or artificial), shape (straight, wavy, needle, etc.), size (macro or microfiber), and mechanical characteristics **[10]**. Usually, there are three divisions of natural fibers used to reinforce composites; animal, mineral, and vegetable classified according to theirnature.

1.3.2.4.1 Animal fibers

Natural fibers made mostly of particular proteins are known as animal fibers. Silk, hair/fur (including wool), and feathers are among examples. Domestic sheep's wool and silk are the two types of animal fiber that are most frequently utilized in production and by hand spinners. Alpaca fiber and mohair from Angora goats are both in high demand. There are other unusual fibers, such as Angora wool from rabbits and Chiengora from dogs, although these are rarely employed in large-scale manufacturing **[11]**.

1.3.2.4.2 Mineral fibers

They include a variety of fibers (asbestos, alumina, etc.), and they are extensively employed in many conventional applications. The asbestos fibers that were once utilized for insulation and fire proofing are now known to cause cancer **[1]**.

1.3.2.4.3 Vegetable fibers

The use of natural fibers of vegetable origin in the construction goes back to the period when bricks reinforced with straw or reeds were made. The first composite binding with vegetables in modern times (since the year 70) was plaster. Plaster that has been strengthened with plants is used in many works. The world is currently aware of recent advancements in the field of using vegetable fibers to reinforce cement and concrete. Numerous studies are being done to replace asbestos with vegetable fibers as a result of the health risks that asbestos fibers present **[1]**.

1.3.2.4.3.1 Structural of vegetable fibers

vegetal fibers are biological structures composed mainly of cellulose, hemicelluloses, and lignin. In a much smaller proportion, they also contain extractable, proteins and some inorganic compounds. The structure of cellulose is generally crystalline, in contrast to other fiber constituents, which have an amorphous structure. The cellulose chains are linked together inside the fiber to produce microfibrils, which aggregate to form fibrils on many layers. The fiber's stiffness is determined by the angle formed by these highly organized components and its axis. A stack of composite plies is a plant fiber. It is made up of a primary wall and a secondary wall, each of which is made up of three layers S1, S2, and S3 (Fig.1.7). If the cell did not fill during development, a cavity named a lumen may exist in the center (filling of the outside of the cell towards the inside). About 80% of the section is made up of the secondary wall's S2 layer, which influences mechanical behavior **[12]**.

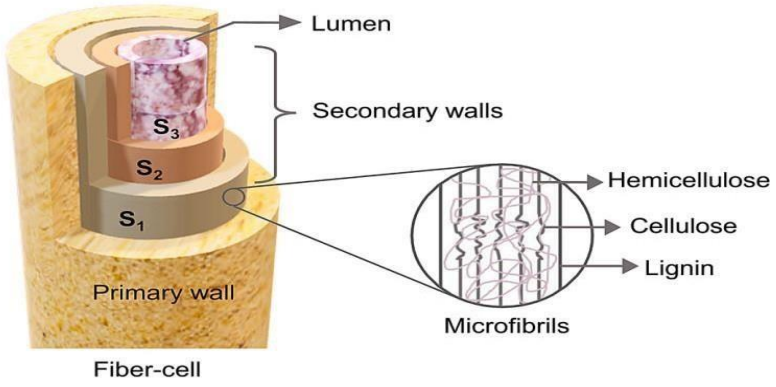


Fig.1.7 Structural of vegetable fiber **[10]**

1.3.2.4.3.2 Composed of vegetable fibers

Most vegetable fibers are composed of cellulose and frequently contain lignin; examples include cotton, hemp, jute, flax, ramie, sisal, bagasse, and banana. Paper and textiles (clothes)

are made from plant fibers, and dietary fiber is a crucial part of human nutrition. They are mainly composed of cellulose, hemicelluloses, and lignin **[4]**.

- **Cellulose**

It is the primary and most important part of vegetable fibers. It is a linear polymer made up of glucopyranose rings connected by glycosidic bonds in the range of one to four (β1-4) as shown in (Fig. 1.8). The degree of polymerization varies depending on the origin of the fibers and these paration process; for cotton, it is greater than 3000, while for wood pulp produced withthe least degradation, it is equal to 1500 **[4]**.

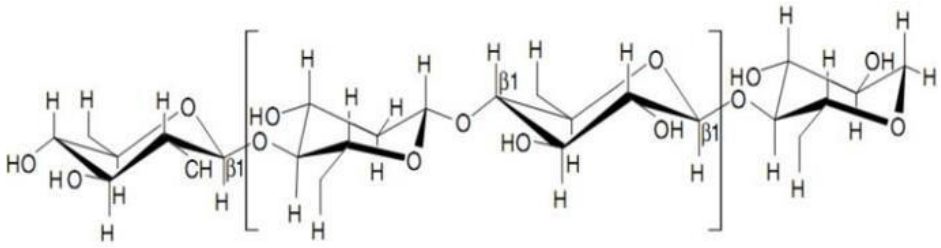


Fig.8.1 Structure of cellulose **[3]**

- **Hemicellulose**

Hemicellulose (Fig. 1.9) is not a cellulose variant. It consists of a collection of polysaccharides with a mix of 5 and 6-carbon rings. For cellulose microfibrils, hemicellulose serves as the support matrix. Three things set hemicellulose apart from cellulose. First off, it only comprises 1,4-D-glucopyranose units in comparison to cellulose's neutral sugars, which include xylose, arabinose, galactose, glucose, mannose, and ironic acids. Its non-crystalline structure is secondly explained by a significant amount of side-group-containing branching. Finally, native cellulose has a degree of polymerization that is 10-100 times higher than what it has in its natural condition, which is between 50 and 300. Hemicellulose rapidly hydrolyzes in acids, is soluble in alkaline media, and has high hydrophilicity **[3]**.

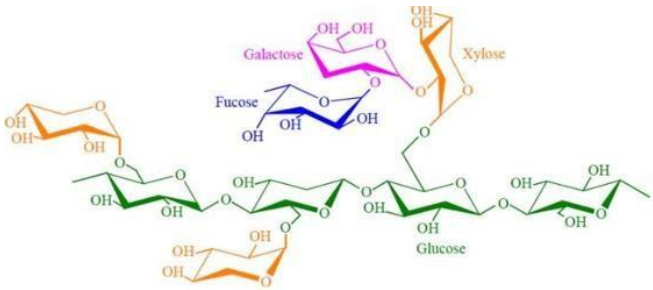


Fig.1.9 Structure of hemicellulose **[3]**

- **Lignin**

Lignin is the second-most prevalent renewable organic substance after cellulose. It has no repeating units and is made of three-dimensional phenolic polymers (Fig. 1.10). Lignin's high reactivity is explained by its complex structure, which includes a wide variety of phenolic, hydroxyl, and ether functionalities. However, their accessibility is constrained by the molecular network's three-dimensional shape **[3]**.

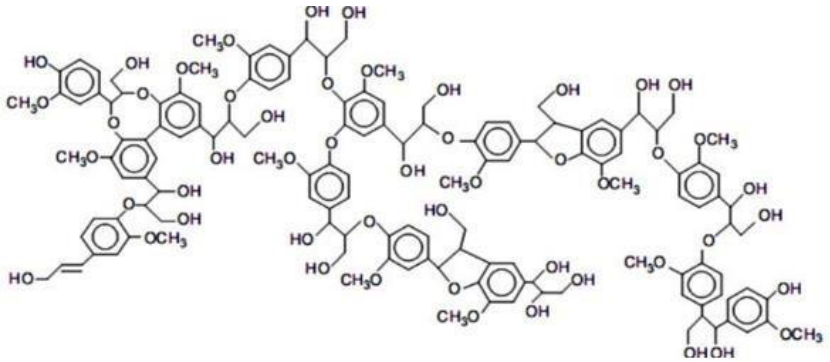


Fig. 1.10 Structure of lignin **[3]**

1.3.2.4.3.3 Classification of vegetable fibers

As shown in the illustration (Fig.1.11), these fibers are categorized according to where they originate from: leaves (*sisal, banana, palm, esparto* fibers), seeds (*cotton, kapok* fibers, etc.), stems (*flax, hemp, jute, kenaf, ramie, bamboo,* etc.), and fruits (*coir* fibers, etc.) **[13]**.

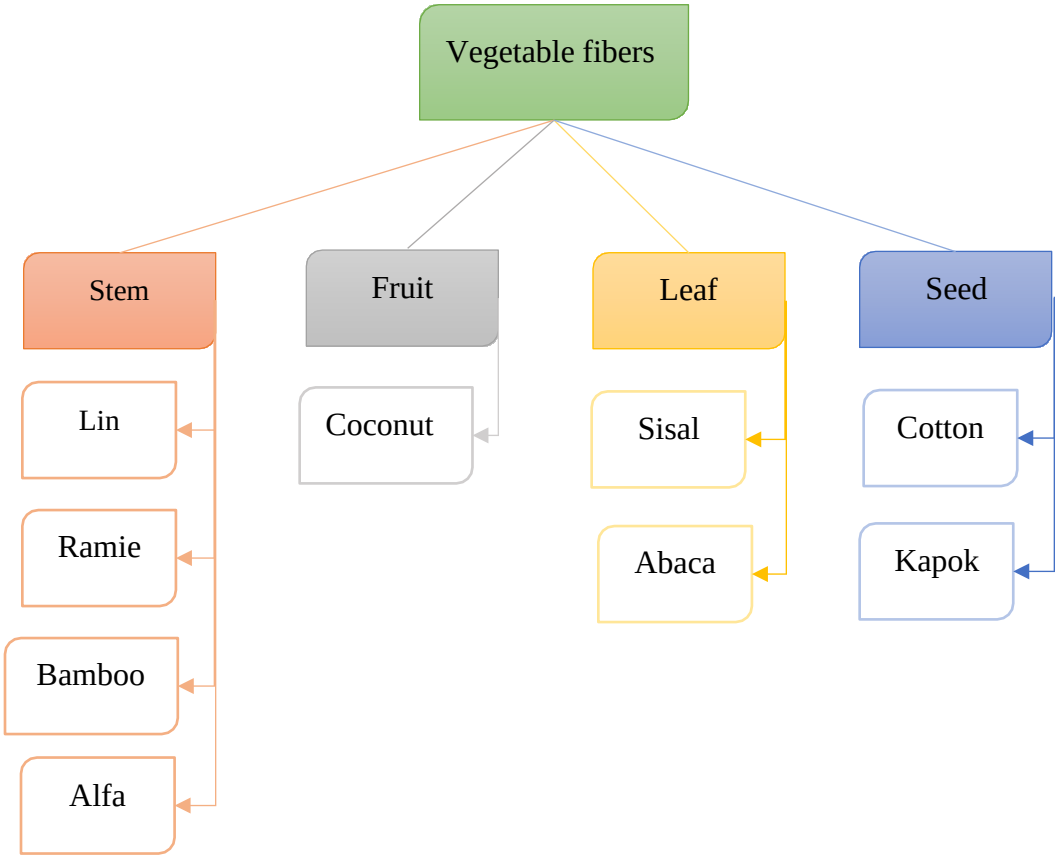


Fig.1.11 Classification of plant fibers by origin **[13]**

1.3.2.4.3.4 Characteristics of vegetable fiber

Vegetable fibers are distinguished by their fineness and elongated form relative to their diameter.The majority of vegetable fibers have a diameter of 10 to 50 micrometers and range in length from10 to 150 mm. The numerous benefits they provide account for their reappearance as prospective materials. Of course, the impact of these benefits differs from fiber to fiber and depends on factors such as chemical and physical composition, structure, the proportion of cellulose, microfibrillar angle,cross-section, and degree of polymerization **[13]**.

1.3.2.4.3.5 Advantages and disadvantages of vegetable fibers

Vegetable fibers offer many advantages, they are considered a renewable resource and biodegradable that's what makes them easy to recycle (combustion) plus, it doesn't leave residue after incineration, also for its production requires low energy and cost to produce and feature high specific mechanical properties (strength and stiffness). In addition, it is a good thermal and acoustic insulator. More importantly, it does not constitute a risk of irritation on the skin when handling fibers. On the contrary, fibers have disadvantages, they depended on the

place of growth growing location, and weather conditions are the influencing factors on the quality which makes it variable, they also have the property of absorbing water which reduces the resistance of the composites made of it, moreover, poor dimensional stability that result from anisotropic behavior, low thermal resistance (200 to 230 °C) **[3]**.

1.3.2.4.3.6 Methods of fiber extraction

Vegetable fibers are often acquired from the processing chain of the textile industry for use as reinforcement in composite materials. This industry has been developing methods to separate thefibers from the remainder of the plant for many years. The fibers are separated and presented usingmechanical, chemical, and biological extraction procedures **[14]**.

1) **Mechanical extraction**

- **Scutching**

This technique entails mechanical activity, such as grinding and threshing, to separate the fibers (stalks). It is done using automated machines that use a series of fluted wheels with initially hugeteeth and then later on with finer teeth to automatically engage, hold, and release the stalks. To increase the effectiveness of the shredding, the fibers are then fed beneath the flute of the rollers at a nearly 90° angle. The foot side and the head side of the procedure are completed one after theother. The shives and short, less resistant fibers, also known as tow, are retrieved by suction and separated **[14]**.

- **Deflection**

Scraping and beating are used in conjunction to extract the fibers. The plant's leaves are grated using devices referred to as "raspadors" (scraper in Spanish), which release the fibers. The fundamental component of these devices is a rotating shaft that is powered by a motor and fixed supports holding steel beaters. The rods entered in front of the machine are grated and steered to the opposite side after becoming stuck between these beaters and a scissor table. Wood and powder are put through screening. Depending on the batch, the distance between the blades can be changed **[14]**.

- **Laminating**

The stalks are divided into smaller pieces, which are then either crushed using a press, rolled, or amixture of the two processes. Up until the fibers are adequately separated, this is repeated multipletimes in a row **[14]**.

2) Chemical extraction

- **Kraft process**

This alkaline technique uses a solution of sodium hydroxide (NaOH) and sodium sulfide to remove lignin, pectins, and hemicelluloses from the material (Na_2S). The latter is a reducing agentthat protects cellulose and stops it from oxidizing. For a period of 2 to 4 hours, the cooking temperature is between 170° and 175°C **[14]**.

- **Bisulfite process**

Using different sulphuric acid salts, the lignin is separated from the cellulose fibers in this method.Sulfites ($SO3^{2-}$) or bisulfites are the salts utilized in the reduction process, depending on the pH. (HSO^{3-}). It is based on the reaction of free sulfur dioxide in calcium, sodium, ammonium, or magnesium hydrogen sulfite. Sulfites or bisulfites have a pH between 1.5 and 5, last for between 4 and 14 hours, and have a temperature range of 130° to 160°C, all of which depend on the base employed **[14]**.

- **Acid process**

An ideally strong acid is used to dissolve the non-cellulosic components (sulphuric acid). The acidtransforms the lignin into hydrochloric acid, or soluble lignosulphonate acid, which creates chlorolignins that are soluble in sodium hydroxide with the help of its chlorate ions **[14]**.

- **Soda process**

Lignin, pectin, and hemicellulose, as well as the different elements making up the reserve and outer wall of the plant stem, are all disintegrated using only NaOH in this method. To avoid degrading the cellulose fibers, the temperature, pressure, concentration, and length of the treatment should be decided upon based on the batch, age, and type of plant. It is recommended to regulate the pH of the solution and set it at about 7. Reducers can be added to stop the cellulose from oxidizing **[14]**.

3) Biological extraction

- **Retting on land**

Retting is a natural process intended to encourage the extraction of fiber. After harvesting, it entails spreading the stalks (of flax, for example) in a field to take advantage of the sun's and rain's combined effects, which encourage the growth of microorganisms that can separate the non-cellulosic components from the fibrous section of the plant. Depending on the weather, this procedure could take anywhere between 6 and 8 weeks. Although this approach is effective, it

has several drawbacks caused by its complete reliance on meteorological conditions (excess humidity, very strong wind). As a result, air retting is an effective techniquein excellent weather, but it is very slow and hence random **[14]**.

- **Water retting**

The difference between this kind of retting and air retting is that the stalks (such as those of hemp, for example) are submerged in water for several days. Anaerobic microorganismsare exposed to the 5–7 kg bundles. The plant is removed from the water and dried as soon as the fibers have completely detached along their entire length. This method has the significant drawback of water contamination but produces fewer random outcomes than the first one. *Flax* and *hemp* retting are fairly common in northern Europe (France, Belgium,and the Netherlands). Once the fibers are dignified and no longer sticky, the water-retting process is continued in a vat with water that is (37°C). This method favors continuous regression and regressing on land **[14]**.

- **By microbial action**

Bacteria, protozoa, and fungi are three types of microbial organisms that can break down the non-cellulosic parts of plant stems or leaves. Three types of bacteria fall into the first group; one has depolymerase activity and the other has glycosidase activity. These bacteria can hydrolyze the main chain and cut the side chains utilizing the released oligosaccharides and oses. The second cannot exploit the hemicelluloses' hydrolysis byproducts since it only possesses depolymerase activity. And the third, which lacks depolymerase activity but has glycosidic activity. Numerous protozoan species can depolymerize hemicelluloses and pectic materials, however, they can only partially use the hydrolysis products as an energy source. Fungi can partially solubilize lignin and depolymerize hemicelluloses to utilize the oligosaccharides and oses that are liberated. Pectins, however, cannot be depolymerized by them **[14]**.

1.3.2.4.3.7 Field of application of vegetable fibers

Vegetable fiber production hasn't been able to keep up with demand, which has been steadily risingalong with industrialists' increased interest, since 2002. This is hardly surprising given that new markets for vegetable fibers that were previously limited to the paper and textile industries have been able to emerge in recent years due to economic and environmental factors. These fibers, which have several benefits for the production of composite materials alloyed with polymers andare finding new uses in the plastics, construction, and automotive industries, are drawing increasing interest from the industrial sector. These composites are widely used in

many other industries, including packaging, cosmetics, pharmacology, and home appliances. (Fig. 1.12) shows the distribution of biocomposites by application. In a report released during the International Year of Natural Fibers in 2009, the Food and Agriculture Organization of the United Nations (FAO) stated that one of the most popular uses forplant fiber nanocomposite membranes in recent years has been in electro-acoustic devices that produce high-quality sound, fuel cell membranes (hydrogen), ultra-filtration membranes (water purification), and membranes used to recover minerals and oils **[13]**.

Fig. 1.12 Interior parts of vehicles of German manufacturers made of biocomposites **[13]**

1.4 Composite manufacturing processes

Several methods are currently used in the industry to manufacture composites, the most commonly used being contact molding, prepreg molding, resin transfer molding, and infusion molding **[15]**.

1.4.1 Contact molding

Contact molding is carried out on an open mold and consists of laying the first reinforcement ply on the mold, adding the resin, and spreading the resin with a roller (Fig. 1.13). The action is repeated for each ply added to the composite. Contact molding's inability to manage resin volume results in laminates of inferior quality. The laminates also have a higher amount of trapped air because thisassembly is not under a vacuum, which makes them more brittle **[15]**.

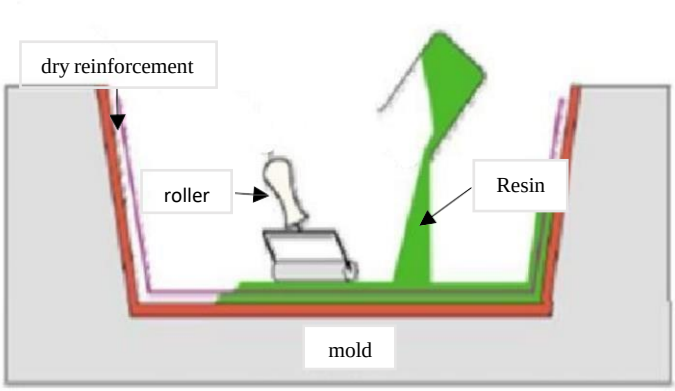


Fig. 1.13 Manufacturing of composites with contact molding **[15]**

1.4.2 Resin transfer molding (RTM)

Takes place in a closed mold made up of two stiff molds. The composite's thickness can be managed thanks to this functionality. A mixer with a pump is used to inject the resin and catalyst, and then the mixture is injected into the mold (Fig. 1.14). By applying pressure while working in a vacuum, this manufacturing technique reduces the amount of air that is trapped in the mold. It makes parts of the highest quality. Due to the need for two hard molds and a more intricate injection system, this process is more expensive than infusion and contact molding **[15]**.

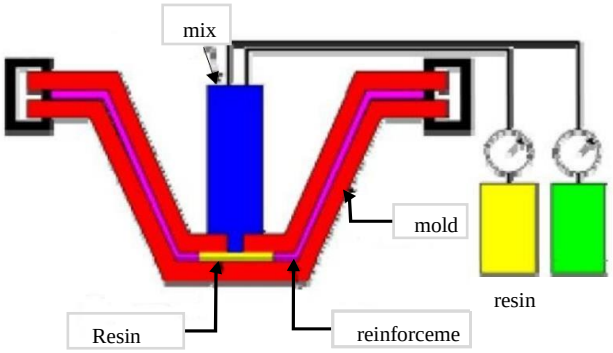


Fig. 1.14 Manufacturing of composites with resin transfer molding **[15]**

1.4.3 Infusion molding

The process is carried out in a closed mold made up of a stiff and flexible mold. A vacuum pump that draws the resin through the reinforcement introduces the resin into the system (Fig. 1.15). Composites made using this process are of high grade. Additionally, it costs less than resin transfer molding. The laminate thickness cannot be better controlled, though **[15]**.

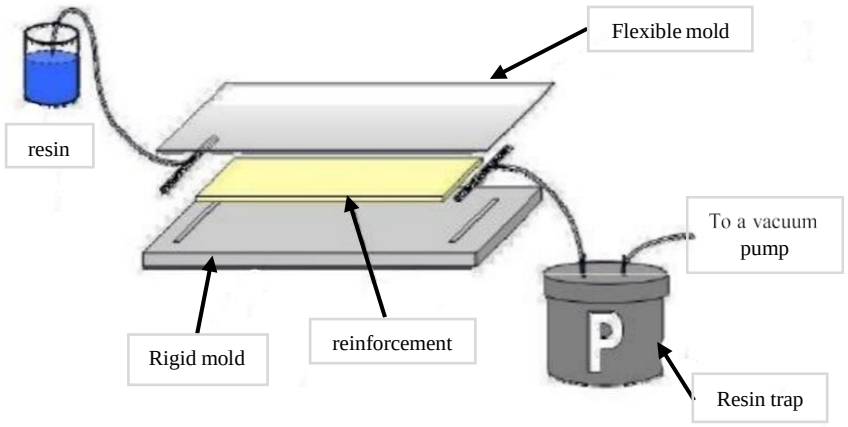


Fig. 1.15 Manufacturing of composites with infusion molding **[15]**

1.5 Conclusion

In conclusion, this bibliographic part focused on presenting the composite materials and their components including matrix, and reinforcement with mentioning their types. also touched on the description of the natural fibers especially the vegetable fibers and their structure, categorization, and characterization. Additionally, we discussed the various extraction techniques and their impact on the composite quality, the composites' application areas, and manufacturing procedures.

Bibliography

1. Prashanth, S., et al., *Fiber reinforced composites-a review.* J. Mater. Sci. Eng, 2017. **6**(03): p. 2-6.
2. Benarab, H., *Matériaux Composites Renforcés par les Fibres Naturelles Utilisées dans l'Isolation Thermique,* Université Saad Dahlab Blida Algérie. p. 89.
3. Anouar, M., *Effet des traitements chimiques sur les propriétés mécaniques des composites polychlorures de vinyle/fibre de palmier.*
4. Krishan, K.C., *Composite materials: science and engineering*2012: Spinger.
5. Hsissou, R., et al., *Polymer composite materials: A comprehensive review.* Composite structures, 2021. **262**: p. 113640.
6. Mrazova, M., *Advanced composite materials of the future in aerospace industry.* Incas bulletin, 2013. **5**(3): p. 139.
7. Saidi, S.M., Louiza; Zembouai, Idris;promoteur, *Matériaux Composites à base de fibres pour des Applications en Génie Civil*, 2022, Université a. Mira de Bejaia.
8. Mougari, B., *Etude expérimentale d'un mortier renforce par des fibres végétales*, 2016, Université Mouloud Mammeri TiziOuzou.
9. Prasad, V.V. and S. Talupula, *A review on reinforcement of basalt and aramid (Kevlar 129) fibers.* Materials Today: Proceedings, 2018. **5**(2): p. 5993-5998.
10. Laverde, V., et al., *Use of vegetable fibers as reinforcements in cement-matrix composite materials: a review.* Construction and Building Materials, 2022. **340**: p. 127729.
11. https://en.wikipedia.org/wiki/Animal_fiber.
12. BENMADDOUR, D., *Elaboration et caractérisation d'un matériau composite à base de fibre végétale: Application à la plante d'Alfa*, 2012, Université de Sidi Bel Abbès-Djillali Liabes.
13. AMEL, K. and B. MARIA, *Effet des techniques d'extraction des fibres végétales sur leurs caractéristiques physiques chimiques et mécaniques*, 2019, UNIVERSITE MOHAMED BOUDIAF-M'SILA.
14. DJEBLOUN, Y., *Contribution à la caractérisation des matériaux composites renforcés de fibres végétales*, 2018, UNIVERSITE MOHAMED KHIDER BISKRA.
15. Lavallée, C., *Étude de l'influence des caractéristiques des tissus sur la résistance à l'impact des matériaux composites*, 2010, École de technologie supérieure.

2

Materials and experimental procedures

Part I : Used materials

2.1 Introduction

Cementitious composites are composite materials made from cement and other materials such as sand, gravel, fibers, and admixtures. They are widely used in the construction industry for building structures like walls, floors, and roofs. These composites offer superior tensile strength and resistance to external elements like weathering and corrosion. Moreover, they also provide excellent fire resistance properties which make them ideal for use in fire-resistant buildings. Furthermore, they can be used to create green composites with high environmental sustainability due to their low carbon footprint. Cementitious mortars are also becoming popular due to their ability to provide increased durability and flexibility in construction applications.

2.2 *Hemp* fiber

2.2.1 History

Human has used *hemp* since the Neolithic era. One of the earliest plants that man tamed was possibly for its fibers. The seeds of *hemp*, which are extremely rich in polyunsaturated fatty acids, were consumed together with the initial applications of the fibers by man, which were for clothing. The paper was later made from *hemp*. In actuality, the earliest known piece of paper dates from between 140 and 87 BC and was made from a combination of *hemp* fiber and mulberry bark. With *hemp* paper, Gutenberg would have printed the first edition of the Bible. *Hemp* fiber was mostly utilized for apparel during the middle ages. *Hemp* and *linen* fibers were combined to create the regal clothes of the West. In the beginning, *hemp* fibers were used to make the sails and ropes that were used on sailing ships. *Hemp*'s farmed areas significantly decreased as cotton gradually overtook it in the textile business around the turn of the 20th century (from 176,000 ha in France in the middle of the 19th century to 700 ha in 1960). With the conflicts, *hemp* has seen a rise in popularity. Military uniforms were made with durable fibers. Because of its usage as a narcotic during the 1900s, *hemp* was gradually outlawed in

several nations. *Hemp* eventually nearly vanished with the development of the motorized fleet and synthetic fibers. Yet since the 2000s, *hemp* cultivation has stabilized, with new outlets reaching 8000 ha in France in 2006 **[1]**.

2.2.2 Plant

Industrial *hemp* (*Cannabis sativa*) belongs to the *Cannabinacae* family **[1]** (Fig.2.1), is an annual plant with a stem that, depending on the species, can grow to a height of 2 to 4 m and a diameter between 1 and 3 cm **[2]**, it is hollow, fluted, and extremely infrequently branching **[1]**. The morphology of the vegetative apparatus can change according to the species and the environment **[3]**. *Hemp* fiber has a considerable opportunity for use as reinforcement in composite materials because it is one of the strongest and stiffest natural fibers currently on the market, and possesses a certain rigidity that is equivalent to glass fibers. Normally is ready to harvest in two to three months after seeding **[1]**.

Fig. 2.1 *Cannabis sativa* plant **[4-5]**

It is commonly accepted that the *Cannabinacae* family consists of a single genus *Cannabis* associated with the species *Cannabinus sativa*, the latter is divided into three subspecies:

- *Cannabis sativa* : industrial *hemp*.
- *Cannabis ruderalis*: wild form of the plant.
- *Cannabis indica* : psychotropic form of the plant characterized by a high level of tetrahydrocannabinol (THC).

According to the regulations in place, *hemp* for industrial uses consists of non-psychotropic strains of the plant species *Cannabis sativa* with a THC concentration of less than 0.2% **[6]**.

2.2.3 General morphology of hemp

Hemp is an *angiosperm dicotyledonous* plant, known as *angiosperm dicotyledons*, whose seeds develop within plant tissues (dictionary terminology dictionary) **[6]**.

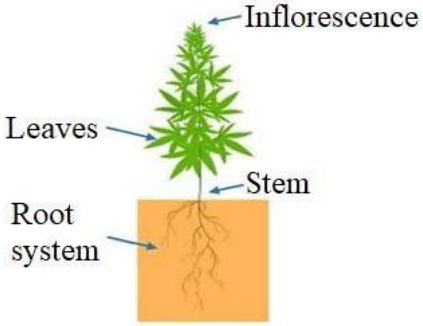


Fig. 2.2 General morphology of *hemp* **[6]**

- **Root system**

Hemp's root system is less developed than other annual plants' (8 to 9% of the total biomass) compared to its aerial biomass. It is a taproot made up of a primary root, also known as the central pivot, that can swell to a depth of about 2 meters and a network of secondary roots that occupy a soil profile between 10 and 90 centimeters high (Fig. 2.2). Depending on the properties of the soil or the methods of cultivation, this root volume will change. Hence, if a plant is experiencing water stress, its root system will often be more developed. Moreover, the sexual phenotype will have an effect because male plants have less developed roots than females (Fig. 3.2). Via its root system, *hemp* can remove heavy metals from the soil. When a plant is uprooted, the heavy metals that build throughout the entire plant are eliminated **[6]**.

Fig. 2.3 Male (A) and female (B) flowers of *Cannabis sativa* **[4]**

- **Leaves**

The leaves are made up of a petiole that is topped by 7 to 10 unequally sized lanceolate leaflets. The petioles are put on the stem in the vegetative phase in the opposite direction, with an interval of between 10 and 30 centimeters (in the first 8 to 10 nodes of the stem). Yet, the arrangement of the leaves on the stem will change to an alternate form at the start of flowering. The top of the plant will be where you can see this trait. The percentage of leaves is rather high (30%), and the number of leaflets gradually increases till the center of the stem **[6]**.

- **Inflorescence**

In the case of *hemp*, the flowers cluster together to create inflorescences, or clusters, which are centered on one of the plant's axes. Both male and female inflorescences are possible. *Hemp* is a naturally dioecious species, which implies that male and female flowers are produced by different plants. Male inflorescences (A) are typically arranged at the top of the stem in more or less loose clusters that resemble panicles and have few leaves, as seen in (Fig. 4.2). At the top of the stem, the female inflorescences (B) take the shape of a cyme with multiple spikes developed at the branching of numerous leaves. There are no petals on these female blooms. Typically, dioecious types of crops contain equal amounts of male and female plants **[6]**.

Fig. 2.4 Inflorescences of male and female *hemp* plant (A) male, (B) female **[2]**

- **Stem**

Hemp stems (Fig. 2.5) can be thought of as hollow cylinders with a range in size. Indeed, the growing conditions, plant types, or stage of development of the plant have a significant impact on the morphological properties of *hemp* stems. The stem often has a smooth base and an upper portion with flutes. The stem is made up of internodes that vary in length, and the diameter of the stem increases from the apex to the base rather than lengthening uniformly **[6]**.

Fig. 2.5 *Hemp* stem **[7]**

This structure is also found in the flax stem or one of its various varieties. We separate five crucial components (Fig. 2.6) **[7]**:

- The **epidermis** is composed of a layer of cells with cellulose walls, as well as a continuous deposit of lipidic nature that forms the cuticle, which serves as the stem's protective coat.
- The **cortex**, which contains cortical fibers grouped in clusters (called bundles), themselves embedded in various tissues.
- The **wood** is composed of fibers, conducting vessels, and parenchyma cells, and it is this section of the stem that provides the *hemp* utilized in the building.
- The **marrow** is constituted of medullary parenchyma.
- The **hollow** space, this latter one, also known as a medullary lacuna, can take up more than half of the diameter in older plants.

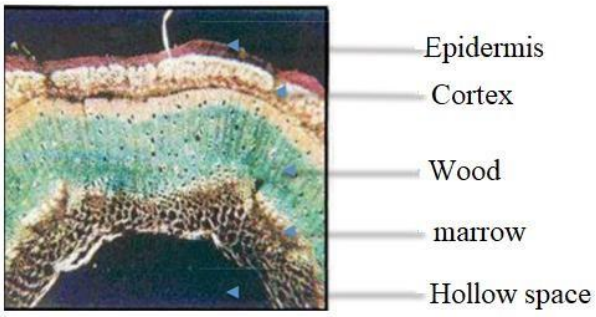


Fig. 2.6 Micrograph of a cross-section of *hemp* stem **[7]**

2.2.4 Cultivation

Growing *hemp* is not very labor-intensive. In Europe, it only needs a small amount of fertilizer (80 to 110 kg of nitrogen/ha), a small amount of water (approximately 450 mm/year), and no pesticides. If the conditions are right for the plant to emerge, *hemp* swiftly covers the ground, strangling its competitors. *Hemp* is a "weed" by nature, which makes it simple to grow. A reasonable output is between 0.5 and 1.2 tons of fiber per hectare. The steps for a typical *hemp* harvest are listed below. A combine harvester is used to first harvest the *hemp*. The *hemp* is then cut into a swath and dried. The straw is then compressed and kept. The retting time must be completed before harvesting the straw. Retting is a biological process that separates the fiber from the other components of the *hemp*. During this period, the humidity level increases which causes certain bacteria or fungi to grow and attack the *hemp*. Finally, there is a series of mechanical transformations to create usable fiber **[1]**.

2.2.5 *Hemp* fiber extraction

Retting of the stem, which usually involves arranging it in windrows on the cultivation field and exposing it for many weeks to the heat and humidity of the sun, is the first step in the extraction of the plant's fibers from its stem. Via this retting, the pectose can be enzymatically broken down, making it easier to extract the fiber bundles. When the stems have been retted, the fiber bundles need to be separated, dyed and then combed coarsely and finely (Fig. 2.7.a, 2.7. b) (Fig. 2.7.c, d). The fiber bundles can be aligned with the final combing **[8]**.

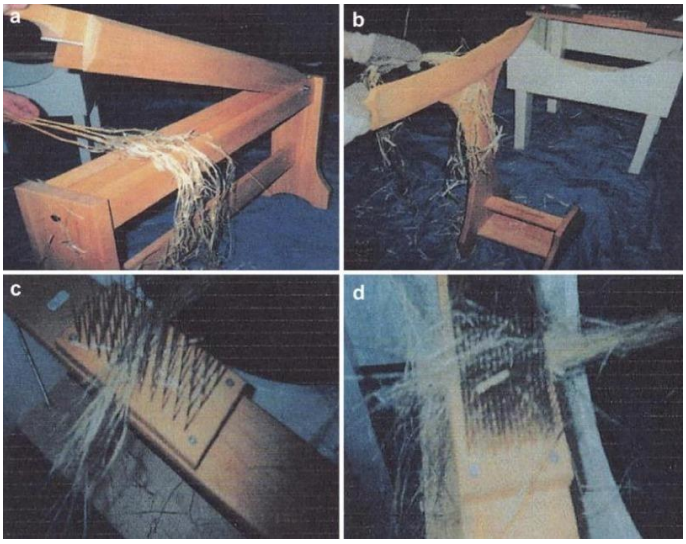


Fig. 2.7 Preparation of *hemp*: **a)** Separation of bundles, **b)** Scutching of fibers, **c)** Coarse combing, **d**) Fine combing **[8]**

Contrary to synthetic fibers, natural fibers have a limited length. The length of the primary fibers of *hemp* ranges from 8 to 14 mm, which is a major factor. Continuous reinforcing is not possible with this length, which is enough to create short fibers or felts. Thus, spinning is a crucial step in obtaining continual reinforcement. All fibers and bundles of fibers are given a torsion angle about the yarn's axis during the spinning process. This angle is crucial because it needs to be both substantial enough to guarantee the yarn's cohesiveness and manageable enough to maintain the mechanical characteristics of the fibers in their longitudinal orientation. To create a continuous woven reinforcement, the yarns can then be weaved **[8]**.

2.2.6 Structure of *hemp* fiber

The cross-section of *hemp* fiber has an unusual form that varies along its length. The major bast fibers are located in the phloem (Fig. 2.8 a) and are made up of roughly 70–74% cellulose, 15-20% hemicellulose, 3.5–5.7% lignin, 0.8% pectin, and 1.2–6.2% wax. This bundle of fibers spans the entire length of the plant stem. Secondary bast fibers that develop from the cambium are also present in the phloem. The multi-celled structure of *hemp* fiber can be thought of as a composite material with several lumens placed side-by-side (Fig. 2.8 b). The illustration below depicts a simple fiber structure made of *hemp* (Fig. 2.8 c). The multi-layered cell wall of *hemp* fiber is made up of the primary cell wall, which is the first layer to form during cell formation, and the secondary wall (S), which consists of three layers (S1–S3). Lignin, which makes up

about 90% of the middle lamella and holds the elementary fibers together, is present. On the other hand, the S2 layer has the highest percentage of cellulose, at roughly 50%. S2, which is also the thickest layer and has a larger proportion of cellulose, regulates the characteristics of the fiber. The link between the fibers is weakened by a microbiological process called retting. Following retting, the fibers are mechanically or manually removed from the *hemp* stem [3].

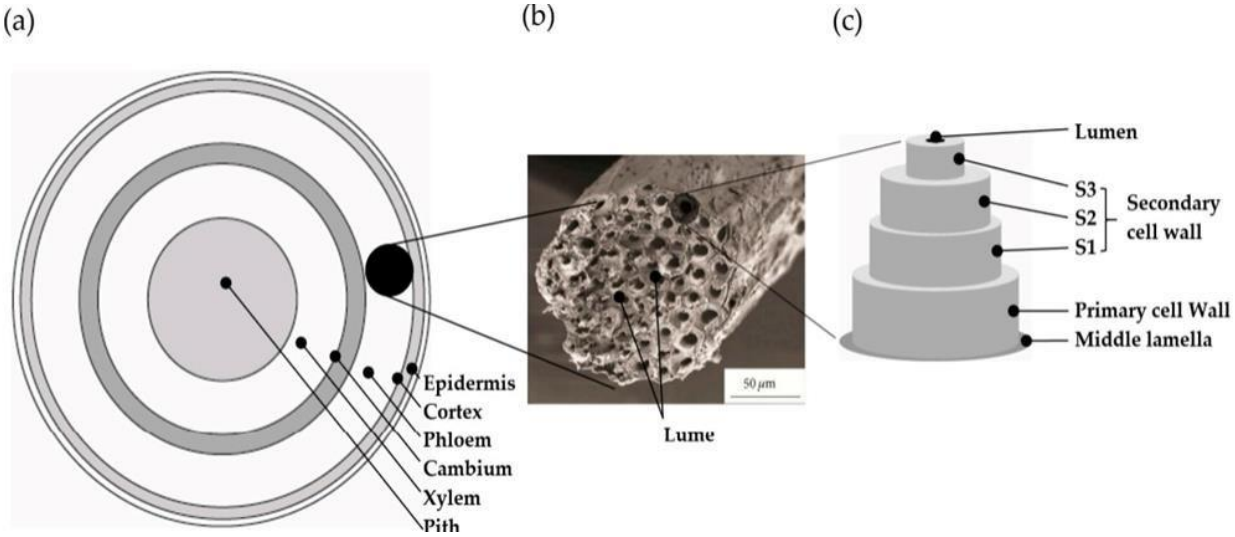


Fig. 2.8 Structure of *hemp* fiber: **(a)** Transverse stem section, **(b)** Cross-section morphology of *hemp* bundle, **(c)** Elementary fiber [3]

2.2.7 Field of application for *hemp* fibers

Two areas of application for the use of *hemp* are currently booming for thermoplastic and cemented composites:

- Building
- Transportation (e.g.; automotive)

In the construction industry *hemp* is inserted in a cement or lime matrix to create insulating blocks or walls. The majority of the time, they are utilized as filler for timber frames because their mechanical characteristics prevent them from being employed to create load-bearing walls.

Hemp is frequently employed in this industry for its thermal qualities. *Hemp* is utilized in place of glass wool because it is a great insulator. More sophisticated components with improved mechanical properties can be produced in the coming years and decades for use in systems that are much more effective. Regarding the automotive sector, there are two manufacturing methods for *hemp* fiber-reinforced polymer matrix composites:

Using injected thermoplastic composites can be made armrests, mirrors, and even fuel tank covers, which are most frequently built on polypropylene PP and reinforced with 30% mass of fibers. Thermoforming is used to create non-woven carpets that combine *hemp* and synthetic fibers to create interior car elements (for example the dashboards, and door interiors) **[9]**.

2.2.8 *Hemp* uses

Cannabis was used in the twentieth century B.C in Egypt in the treatment of sore eyes, and used in India before the tenth century B.C under the name Bhang (a *cannabis* drink) as an anesthetic and anti-phlegmatic **[10]**, in addition, it is applied in both Hindu and Islamic drug system, Being a spasmolytic, hypnotic, analgesic, and sedative for mental problems as well as to boost resistance to extremely stressful physical situations **[11]**, furthermore, it was discovered that it may be used to cure a variety of human diseases, including leukoderma, leprosy, smallpox, burns, allergies, cuts, scabies, and inflammation **[10]**.

- **Common use**

Past research on the *hemp* plant has shown that it has been used to treat a wide range of ailments and diseases because of its psychoactive qualities. They included stomach issues, sleeplessness, and headaches. *Cannabis* was frequently used to ease the agony of childbirth **[12]**.

Oil extracted from the *hemp* seed can be used in light cooking as well as in baking. unlike that, it will start to smoke even at very low temperatures. Thus, it is best to use *hemp* oil as “finishing oil”, used for salad dressing for example. Researchers investigated *hemp* for its ability to repel insects, and they discovered that it is effective **[13]**.

- **Pharmacological use**

When it was discovered that *hemp* herb had therapeutic effects, it has been utilized for this purpose for millennia. At the end of the nineteenth century, it was used to treat depression, pain, asthma, spasms, sleep issues, and appetite loss throughout Europe **[14]**.

- **Cannabis as medicine**

Researchers have speculated that *cannabinoids* may be the "aspirin of the twenty-first century" due to their extensive clinical potential. Unfortunately, the majority of the evidence for *cannabinoids*' therapeutic uses is based on anecdotes, and it is presently exceedingly difficult to confirm these results using different clinical investigations. Also, it is frequently uncertain which chemical component of *hemp* herb causes the observed impact after taking an herbal extract. The understanding of the underlying biology between the chemical components of *hemp*

herb and the human system is slowly expanding, but there are yet no adequate animal models or samples that are as complicated as the human brain. Nonetheless, several *cannabinoids* have been created for use in medicine despite these restrictions. Today, *cannabis* medicines are utilized to treat many ailments. Several researchers have validated or confirmed its usage in treating Tourette's syndrome as well as its effects as an antiemetic, analgesic, muscle relaxant, and appetite enhancer **[15]**.

- **Cannabis negative effects**

The severe and prolonged use of *cannabis* has some negative side effects, such as lung irritation, an increased chance of developing chronic bronchitis and respiratory tract cancer, as well as an increased risk of cardiovascular diseases due to an elevated heart rate. Moreover, it affected short-term memory and made learning and thinking challenging. *Cannabis* dependence, a compulsive drive to take the drug, as well as issues associated with long-term drug use **[10]**.

2.3 Cementitious matrix

This work was assisted by the company of cement in Tebessa so, we used the cement produced by this company. This product is subject to the Algerian standard (NA 442\2013) under the name Portland Composite Cement (CEM IIA/M-(P-L) 42.5 R). The table below (Table 2.1, 2.2) presents the characteristics of the cement used according to the standard (NA 234) **[16]**.

Table 2.1 Technical data sheet (CEM IIA/M-(P-L) 42.5 R) **[16]**

Mechanical tests (NA 234)			
	Expiry in days	Guarantees	Measures
Bending resistance MPa	02 days		4.7
	07 days		7.2
	28 days		8.1
Compressive resistance MPa	02 days	≥20	23.3
	07 days		40.0
	28 days	≥42,5	51.0

Table 2.2 Components of cement used

Components	(%)
SiC_2	21.69
Al_2O_3	6.19
Fe_2O_3	3.90
CaO	62.44
MgO	1.59
K_2O	0.67
NaO_2	0.314
SO_3	2.37
Cl	0.004
Sum	0.992

Part II : Characterization and experimental analysis of the mechanical behavior in bending and in compression of *hemp* fiber reinforced cementitious bio-composites

2.4 Preparation of cement composites based on *hemp* fibers

2.4.1 Fiber preparation

Natural fibers are advantageous since they are inexpensive, low-density, and biodegradable. However, the poor compatibility between fiber and matrix and the relatively high moisture sorption is the fundamental drawbacks of natural fibers in composites. Chemical treatments are therefore thought of as a way to change the surface characteristics of the fibers. The chemical modification of fiber that is intended to increase the fiber's strength may also change the fiber surface's ability to adhere to the matrix. Composites have less water absorption and better mechanical qualities **[17]**.

2.4.1.1 Alkaline treatment

One of the most popular chemical treatments for natural fibers is the alkaline treatment or sodium hydroxide (NaOH) treatment. The breaking of hydrogen bonds in the network structure caused by alkaline treatment is a significant alteration that raises surface roughness. This process depolymerizes cellulose, exposes the short-length crystallites, and eliminates some of the lignin, wax, and oils covering the exterior of the fiber cell wall **[17]**.

First, to prepare the NaOH solution (Fig.s 10.2 and 11.2) for the different concentrations (2%, 3%, and 4%) we need the measurement of 1000 ml of distilled water corresponds to 20 g of NaOH (Fig. 9.2), and the same for the rest of concentration for 30 g and 40 g.

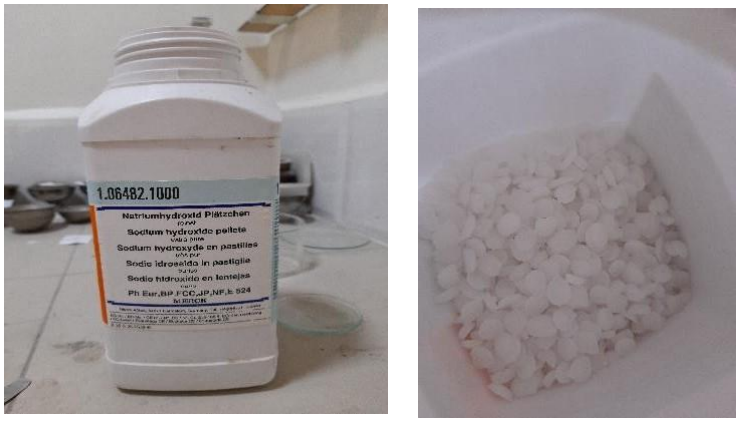


Fig. 2.9 NaOH product used

Fig. 2.10 Measurement of the quantity required

Fig. 2.11 NaOH solution preparation

The fibers pile weighed approximately 300 g (Fig. 2.12) and was divided into three equal parts (Fig. 2.13), then immersed in the previously prepared soda solution for 24 hours at room temperature (Fig. 2.14). this will allow the fibers to soften and release non-cellulosic substances such as waxes, pectin, lignin, and hemicelluloses that could reduce their adhesion to the cement.

Fig. 2.12 Pile of fibers

Fig. 2.14 Divided the fibers into three

Fig. 2.14 Fiber immersed

After the end of the immersion time, the fibers have been removed from the solution and rinsed thoroughly with clear water (Fig.15.2 and 16.2) to remove all impurities and residues that could affect the quality of the bond between the fibers and the cement.

Fig. 2.15 Washing the fiber

Fig. 2.16 Washed fibers

Finally, the washed fibers are placed in the oven for drying (Fig. 2.17). The drying process takes 6 hours at 60˚C.

Fig. 2.17 Fiber drying in the oven

After finishing the treatment, the fibers are cut to different lengths, the obtained fibers were kept in bags to avoid the absorption of water that exists in the ambient air.

2.4.2 Preparation of samples

With the objective of the elaboration of a bio-composite with cement matrix reinforced by a type of vegetal fibers and to study its behavior also to optimize this material, this work has been applied at the laboratory of quality control detective for the company of cement Tebessa for preparing mortars. This experiment based on three parameters that influence the behaviour of mortars (Table 2.3) which are:

- Length of the fibers in mm.
- Percentage concentration of the alkaline NaOH in %.
- the volume of fibers in the mortar in %.

Table 2.3 Factors of elaboration

Factors	Low	Middle	high
Length (mm)	1	3	5
Alkaline (%)	2	3	4
Volume (%)	1	1.5	2

This work aims to study the influence of the parameters mentioned above on the development of mortars. Furthermore, to extract the impact on the behavior of the material in compressive and bending strength so, a strategy of elaboration experiments has been developed by Design expert software (Table 2.4) to reduce the number of trials as much as feasible and enable the development of bio-mortars at the lowest possible experimental expenses.

Table 2.4 Plan of samples elaboration

Run	Length (mm)	NaOH (%)	Volume (%)
1	1	2	1
2	1	2	2
3	1	4	2
4	1	4	1
5	1	3	1.5
6	5	2	1
7	5	3	1.5
8	3	3	1.5
9	3	3	1.5
10	5	2	2
11	3	2	1.5
12	5	4	2
13	3	4	1.5
14	3	3	1
15	5	4	1
16	3	3	2
17	3	3	1.5

2.4.3 Determination of characteristics of bio-composites

In the determination of the characteristics of a composite material it is very important to know the factors essential to the constituents of the composite which is expressed in volume fraction or in mass fraction, to determine the mass fraction and used during the elaboration and theoretically pre-expressed in the elaboration plan (Table 4.2) in the form of volume fraction, we need to use the Law of mixtures as shown in the following equations **[18]**:

The fiber mass fraction

$$W_f = \frac{w_f}{w_c} \quad (1.2)$$

The matrix mass fraction:

$$W_m = \frac{w_m}{w_c} \quad (2.2)$$

Considering that:

$$v_m + v_f = v_c \quad (3.2)$$

So:

$$V_f + V_m = 1 \quad (4.2)$$

Where:

w_f: mass fraction.

w_m: fiber mass.

w_c: composite mass.

There is a relation between volume and mass the mass of this material per unit volume is the density, noted ρ, and is determined by the relation:

$$\rho = \frac{w}{v} \tag{5.2}$$

Where:

w : mass.

v: volume.

ρ: density.

From (5.2) the mass of fiber and matrix we have written:

$$w_f = \rho_f . v_f \tag{6.2}$$

$$w_m = \rho_m . v_m \tag{7.2}$$

$$w_c = \rho_c . v_c \tag{8.2}$$

By replacing in equation (1.2)

$$W_f = \frac{w_f}{w_c} = \frac{\rho_f . v_f}{\rho_c v_c} = \frac{\rho_f}{\rho_c} V_f \tag{9.2}$$

Finally

$$W_f = \frac{\rho_f}{\rho_c} V_f \tag{10.2}$$

We conclude from equation (1.2)

$$w_f = W_f . w_c \tag{11.2}$$

We have created a calculating program in Excel to make calculations easier and prevent mistakes caused by manual calculations and repetition. Through this program, we were able to calculate the fiber mass value for sample preparation. The results are reported in (Table 5.2)

Table 2.5 Fiber mass values

Volume fraction (%)	1%	1.5%	2%
Fiber mass in the sample (g)	2.944	4.416	5.888
Fiber mass in mold (g)	8.832	13.248	17.664

For this purpose, prepare samples as per the Algerian standard (NA 234) (equivalent to the European Standard EN 196-1) for the preparation of a standard mortar for mechanical testing. We need 450 ±2 g of cement, 1350 ±5 g of sand, and 225 ±1 g of water with different volumes of treated fibers diminishing from the weight of the sand, all of these constituents mixed with an automatic mixer (Fig. 2.18), the process is done by adding cement after the mixer add the water automatically then he mixes for 1 min at low speed, in the last 30s sand is added and mixed at high speed for 2 min for two times. Thus, the mixing process was completed in a total of 5 minutes, the mixture was then poured into prismatic molds of 40 x 40 x 160 mm^3. Then, to get rid of air bubbles we put the mold in a shock table subjects it to 60 blows, and brushed off the excess amount of mortar for a flat surface. In the end, samples are placed in a humid chamber at a temperature of 20°C and a humidity of 90,5%. The samples are removed from the mold after 24 hours and then immersed in water for 28 days before being tested.

Fig. 2.18 Automatic mixer

Fig. 2.19 1.5 % of fibers

Fig.2.20 2 % of fibers

Fig. 2.21 1 % of fibers

Fig.2.22 450 g of cement

Fig.2.23 1350 g of sand

Fig. 2.24 Mortar after mixing

Fig.2.25 Steel prismatic mold of 40 ×40 × 160 mm²

Fig. 2.26 Mold in shock table

Fig. 2.27 Final shape of mold

Fig. 2.28 Humid chamber

Fig.2.29 Molds after 24 hours

Fig. 2.30 Samples after de-molded

Fig.2.31 Samples immersed in water

2.5 Mechanical tests

2.5.1 Three-point bending test

The bending test is a mechanical test that is a member of the group of time-independent tests such as hardness and shock **[19]**. The breaking strength of a material is also assessed using the 3-point bending test. A bar made of the material to be tested is set up on two supports, and its center is subjected to increasing forces until it breaks (Fig. 2.32). Similar to the compression test, the flexion test typically does not allow for the rupture of ductile materials. The bending test is best suited for delicate materials. This experiment stands out for its easy assembly of the probe and its straightforward geometry (with or without machine usage). During the test, the upper section is in compression while the lower part is in traction **[20]**.

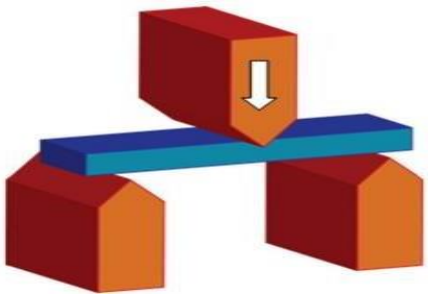


Fig. 2.32 Three-point bending **[19]**

2.5.2 Compressive test

The compression test involves applying two opposing axial stresses to a cylindrical specimen that is sandwiched between press plates. If the material being studied is ductile, this test cannot determine failure. To mill brittle materials (like ceramics) for a tensile test, the compression test is primarily utilized to estimate the fracture stress **[20]**. After 28 days of curing water, the samples were ready to be tested on compressive and bending.

Fig. 2.33 Samples for testing

The identification of compressive and bending strength is determined on a compression and bending test machine of type ToniNORM which is a combined test machine for the standard-

compliant testing EN 196 / ISO 679 of the compressive and flexural strength of cement and other binding materials **[21]**.

Fig. 2.34 Compression and bending test machine

Fig. 2.35 Bending test device

Fig. 2.36 Sample in bending

Fig. 2.37 Shape of mortar after bending test

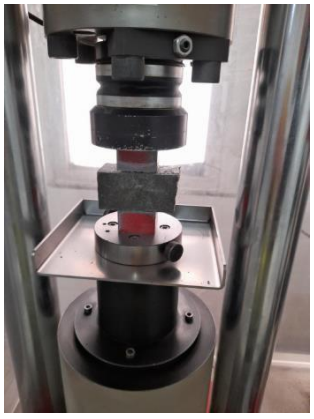


Fig.2.38 Compressive test device

Fig.2.39 Sample in compressive test

Fig.2.40 Shape of mortars after compressive test

Fig. 2.41 A magnified image of the mortar facet

2.6 Results

Table 2.6 Results obtained from bending and compressive test

N°	Bending (MPa)				Compression (MPa)				
	S1	S2	S3	Average	S1	S2	S3	Average	Gap
1	7.21	6.49	6.42	**6.71**	40.9	39.9	39.6	**40.13**	**$40.25^{\pm 0.65}$**
2	6.01	5.90	5.49	**5.8**	36.0	35.4	34.5	**35.4**	**$35.25^{\pm 0.75}$**
3	6.66	6.57	6.95	**6.73**	45.1	43.7	42.9	**43.9**	**$44^{\pm 1.1}$**
4	6.47	6.97	6.83	**6.76**	40.1	40.3	40.1	**40.17**	**$40.2^{\pm 0.1}$**
5	6.14	6.67	6.52	**6.44**	38.4	37.8	38.2	**38.13**	**$38.3^{\pm 0.1}$**
6	6.92	7.27	7.04	**7.08**	43.2	42.6	42.6	**42.8**	**$42.9^{\pm 0.3}$**
7	6.24	5.84	6.67	**6.25**	37.8	36.3	36.3	**36.77**	**$37.05^{\pm 0.75}$**
8	6.77	5.83	6.16	**6.30**	35.4	34.8	34.8	**36.08**	**$35.1^{\pm 0.3}$**
9	6.11	6.22	6.70		37.2	37.3	37.0		**$37.15^{\pm 0.15}$**
10	5.30	4.68	4.85	**4.94**	32.9	33.1	29.5	**31.83**	**$31.3^{\pm 1.8}$**
11	6.53	5.32	5.59	**5.81**	39.2	38.1	37.6	**38.3**	**$38.4^{\pm 0.8}$**
12	5.95	5.23	5.00	**5.39**	30.3	30.5	28.3	**29.9**	**$29.4^{\pm 1.1}$**
13	6.03	6.20	6.52	**6.25**	37.0	36.8	36.7	**36.83**	**$36.85^{\pm 0.15}$**
14	6.49	6.37	6.71	**6.52**	41.8	41.7	40.6	**41.37**	**$41.2^{\pm 0.6}$**
15	6.99	7.26	6.93	**7.06**	44.7	43.9	43.8	**44.13**	**$44.25^{\pm 0.45}$**
16	6.38	6.61	6.86	**6.62**	36.7	36.3	36.0	**36.33**	**$36.35^{\pm 0.35}$**
17	6.77	5.80	6.19	**6.30**	37.1	37.2	37.2	**36.08**	**$37.15^{\pm 0.15}$**

2.7 Conclusion

In conclusion, this is the experimental chapter in this research which focused on the elaboration of cementitious composites reinforced with hemp fibers. The use of vegetable fibers as reinforcement in cement-based materials has been extensively studied and has shown promising results in terms of improving the mechanical properties of cementitious matrices. The description of hemp fiber used in this work presented the most common uses, their properties, and their effect for use as a medicine since ancient times. The experimental study evaluated the effect of adding vegetable fibers to cementitious matrices on their mechanical performance, including bending and compressive. Overall, the results showed that the use of vegetable fibers as reinforcement in cementitious composites is a promising approach for developing sustainable building materials that can contribute to reducing environmental impact while improving the mechanical properties of construction materials.

Bibliography

1. Ringuette, B., *Matériaux composites à base de fibres de chanvre*, 2011, Université Laval.
2. Bouloc, P., *Le chanvre industriel: production et utilisations*2006: Editions France Agricole.
3. Manaia, J.P., A.T. Manaia, and L. Rodriges, *Industrial hemp fibers: An overview.* Fibers, 2019. **7**(12): p. 106.
4. Bonini, S.A., et al., *Cannabis sativa: A comprehensive ethnopharmacological review of a medicinal plant with a long history.* Journal of ethnopharmacology, 2018. **227**: p. 300-315.
5. Akbar, S., *Cannabis sativa L.(Cannabaceae) (Syn.: C. indica Lam.).* Handbook of 200 Medicinal Plants: A Comprehensive Review of Their Traditional Medical Uses and Scientific Justifications, 2020: p. 491-497.
6. Grégoire, M., *Extraction des fibres de chanvre pour des composites structuraux-Optimisation du potentiel mécanique des fibres pour des applications concernant des matériaux composites 100% bio-sourcés*, 2021.
7. Niyigena, C.s., *Variabilité des performances de bétons de chanvre en fonction des caractéristiques de la chènevotte produite en Auvergne*, 2016, Université Blaise Pascal-Clermont-Ferrand II.
8. Barbière, R., *Comportement en fatigue et optimisation de l'interface d'un composite tissé chanvre/époxy: effet de l'humidité*, 2020, ISAE-ENSMA Ecole Nationale Supérieure de Mécanique et d'Aérotechique-Poitiers.
9. Ilczyszyn, F., *Caractérisation expérimentale et numérique du comportement mécanique des agro-composites renforcés par des fibres de chanvre*, 2013, Université de Technologie de Troyes.
10. Ghnia, J.B., et al., *Hemp: A detailed ethnopharmacological review of a medicinal plant of great importance.*
11. Ali, E.M.M., et al., *Antimicrobial Activity of <i>Cannabis sativa</i> L.* Chinese Medicine, 2012. **Vol.03No.01**: p. 4.
12. Touw, M., *The religious and medicinal uses of Cannabis in China, India and Tibet.* Journal of psychoactive drugs, 1981. **13**(1): p. 23-34.
13. McPartland, J.M., *Cannabis as repellent and pesticide.* Journal of the International Hemp Association, 1997. **4**(2): p. 87-92.
14. Grotenhermen, F. and K. Müller-Vahl, *The therapeutic potential of cannabis and cannabinoids.* Deutsches Ärzteblatt International, 2012. **109**(29-30): p. 495.
15. Brenneisen, R., *Chemistry and analysis of phytocannabinoids and other Cannabis constituents.* Marijuana and the Cannabinoids, 2007: p. 17-49.
16. *http://www.sct.dz/ciments.html*. [cited 2023 19 march].
17. Li, X., L.G. Tabil, and S. Panigrahi, *Chemical treatments of natural fiber for use in natural fiber-reinforced composites: a review.* Journal of Polymers and the Environment, 2007. **15**: p. 25-33.
18. Samir, D., *Matériaux Composites.*
19. Hacini, K. and A. Souahi, *Etude Du Comportement Mécanique En Flexion 3 Points D'un Matériau Composite Par Les Eléments Finis.* Diplôme de magister, Université Badji Mokhtare Annaba, 2009.
20. Jacquot, B., *Propriétés mécaniques des Biomatériaux utilisés en Odontologie.* Société Francophone des Biomatériaux Dentaires (SFBD), 2010.
21. *https://tonitechnik.com/product/compression-and-bending-test-plant-toniprax/*. [cited 2023 16 april].

3

Overview of experimental design and fundamental statistical concepts

3.1 Introduction

Optimization methods and data analysis are crucial in various fields, including engineering, science, and business. The design of experiment (DOE) and response surface methodology (RSM) are two widely used techniques for optimizing processes and analyzing data. DOE allows researchers to systematically vary input variables to identify the optimal combination that yields the desired output. RSM, on the other hand, is a statistical technique that models the relationship between input variables and output responses. By combining these two techniques, researchers can effectively optimize processes and analyze data to gain insights and make informed decisions. In this chapter, we will explore the application of DOE and RSM in optimizing processes and analyzing data, and evaluating their effectiveness in various scenarios.

3.2 Experimental design

Design of experiments (DOE) is a useful method for organizing experiments to examine the data gathered and come to reliable, unbiased findings. The phrase "Design of experiment" refers to a systematic, ordered strategy for establishing the link between the elements influencing a process and its result. In experiments, we purposefully alter one or more process variables (or factors) to evaluate the impacts on one or more response variables. The (Statistical) design of experiments (DOE) is a useful method for organizing experiments in a way that allows for the analysis of the data acquired and the production of reliable and impartial findings. Establishing an experiment's goal and choosing the study's procedural parameters are the first steps in DOE. A complete experiment plan is laid out before an experiment is conducted using a selection of experimental designs. Obtain the most "Information" possible for a given quantity of experimental effect **[1]**.

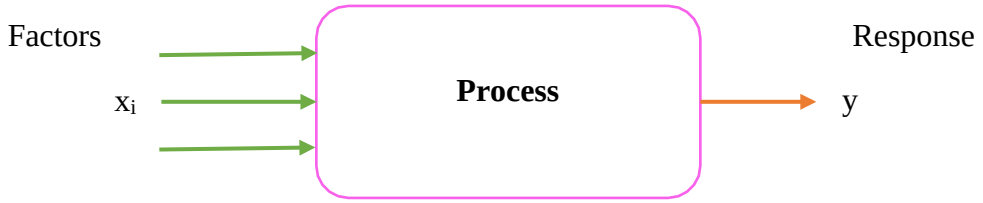


Fig. 3.1 Inputs and outputs of an experiment **[2]**

3.4 Common terms and concepts of experimental designs

3.4.1 Factors

Every variable that has the potential to affect the observed response is a factor or input. A factor may be qualitative, quantitative, continuous, discontinuous, and/or within the control of the researcher. Uncontrollable variables are unsettling elements that are hard to manage. Due to experimental research methodology, it is feasible to lessen or completely prevent their consequences. The range of a component under investigation is always between its lower limit and upper bound, or "low level" and "high level," respectively this is called level (Fig.3.2). This range of permitted variation for the factor must be defined by experts in the topic under study. Each of the components involved in the event under study will have its range of variance. The low level is denoted by -1 and the high level by +1 by convention to have a similar representation for all the components. The term "coded," "standardized," or "centered-reduced" refers to these variables **[3]**.

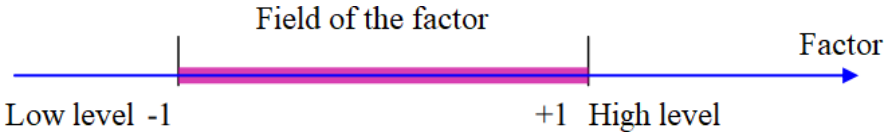


Fig.3.2 Field and levels of the factor **[4]**

3.4.2 Response

We call response the quantity of interest observed in the study of a phenomenon. This study can result in several responses. Only indirectly can the value of a response be changed by changing the factors **[3]**.

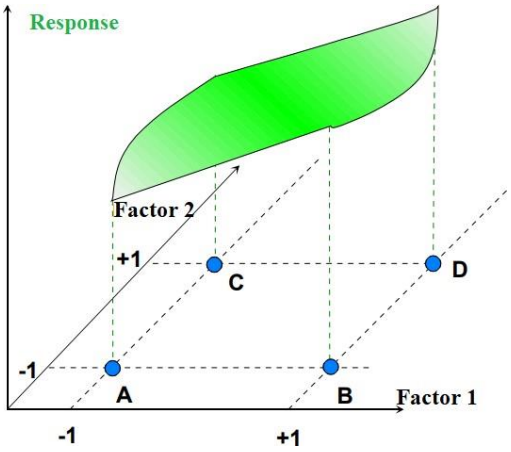


Fig.3.3 Response surface **[5]**

3.4.3 Experimental space

We will employ a two-dimensional space to describe the experimental space, which will make graphical representations easier. The notions provided are then easily extended to multidimensional spaces. A graded and oriented axis can be used to describe a continuous factor. A graded and directed axis is also used to indicate a second continuous factor. This second axis is perpendicular to the first. As a result, we get a Cartesian reference frame that defines a two-dimensional space. This area is known as the experimental space (Fig.3.4). All of the points in the plane "factor1*factor2" are included in the experimental space, and each one represents an experiment **[6]**.

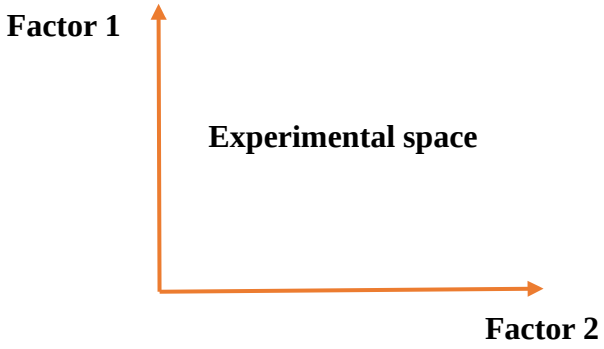


Fig. 3.4 Experimental space **[6]**

3.4.4 Experimental domain

A specific experiment is an experimental point (Fig.3.5), and the set of experimental points is the experimental domain (Study Domain). For two factors, the defined experimental region is typically a square (Fig.3.6), a cube for three factors, and so on **[4]**.

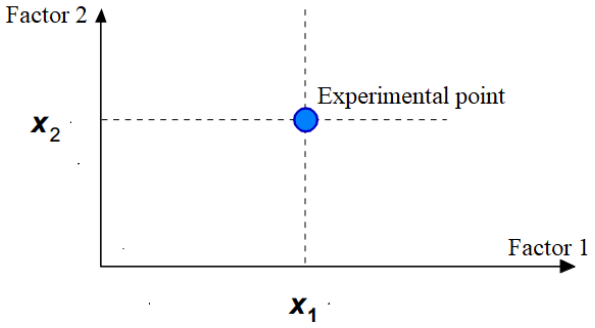


Fig.3.5 Experimental point **[5]**

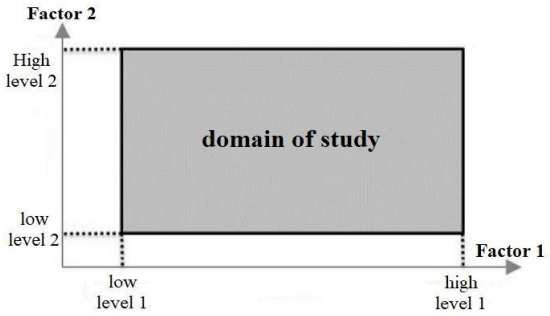


Fig.3.6 Experimental domain **[4]**

3.4.5 Reduced centered variables

Let A be the natural or real variable whose low-level A^- corresponds to the normalized variable -1 and the level A^+ to +1.

The middle value of the domain is:

$$A_0 = \frac{A^+ + A^-}{2} \tag{1.3}$$

The notion of steps is also introduced:

$$pas = \frac{A^+ - A^-}{2} \tag{2.3}$$

The passage from the original variables A to the coded variables noted X is given by:

$$X = \frac{A - A_0}{pas} \tag{3.3}$$

3.4.6 Effect of a factor

the influence of factor X relates to the variation of the response Y when X changes from a value at the level (-1) to another value at the level (+1), as illustrated in (Fig. 3.7). In terms of graphs, the larger the inclination, the bigger the effect, and this already offers indicators **[7]**.

We distinguish **[8]**:

The overall effect:

$$y_2\text{-}y_1 \tag{4.3}$$

The average effect:

$$\frac{y_2\text{-}y_1}{2} \tag{5.3}$$

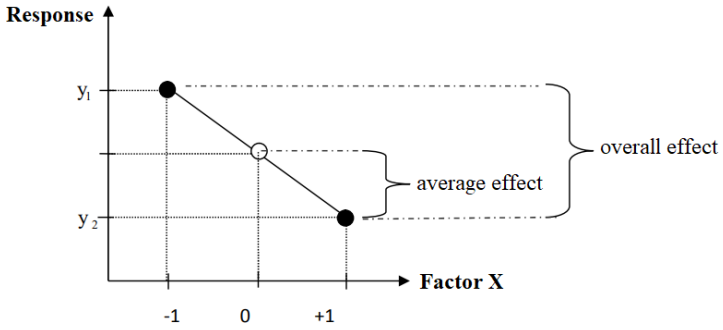
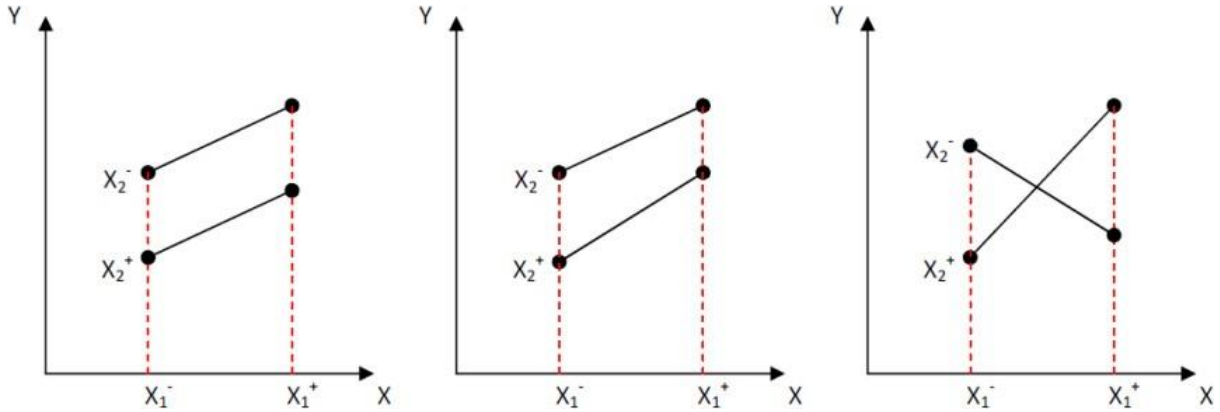


Fig.3.7 Illustration of the overall effect and the average effect **[8]**

3.4.7 Interaction

Interaction refers to the influence of one element on the value taken by another component. The fact that the two lines are not parallel depicts this interaction on a diagram (Fig. 3.8). The larger the deviation of the lines from parallel, the greater the degree of interaction **[7]**.



Interaction absence Faible interaction Forte interaction

Fig. 3.8 Interaction graphs **[7]**

3.4.8 Mathematical model

It is a mathematical connection that explains how a change in one or more factors causes a change in response. The goal of modeling the reaction with a mathematical model is to be able to determine all of the responses in the field of research without performing the tests. This is known as a hypothesized or a priori model **[7]**, in the form **[9]**:

$$y = a_0 + \sum a_i x_i + \sum a_{ij} x_i x_j + \Sigma a_{ii} x_i^2 + \ldots.. \quad (6.3)$$

Where:

y: the response.

x_i, x_j: represents the level of factor i and factor j.

a_0, a_i, a_{ij}, a_{ii}: are the coefficients of the polynomial.

3.5 Experimental design methodology (DOE)

Obtaining good results from a Design of experiment (DOE) involves these steps **[1]**:

- Set objective.
- Select process variables.
- Select an experimental design.
- Execute the design.
- Analyze and interpret the results.

The illustration below (Fig.3.9) shows the process of an experimental design:

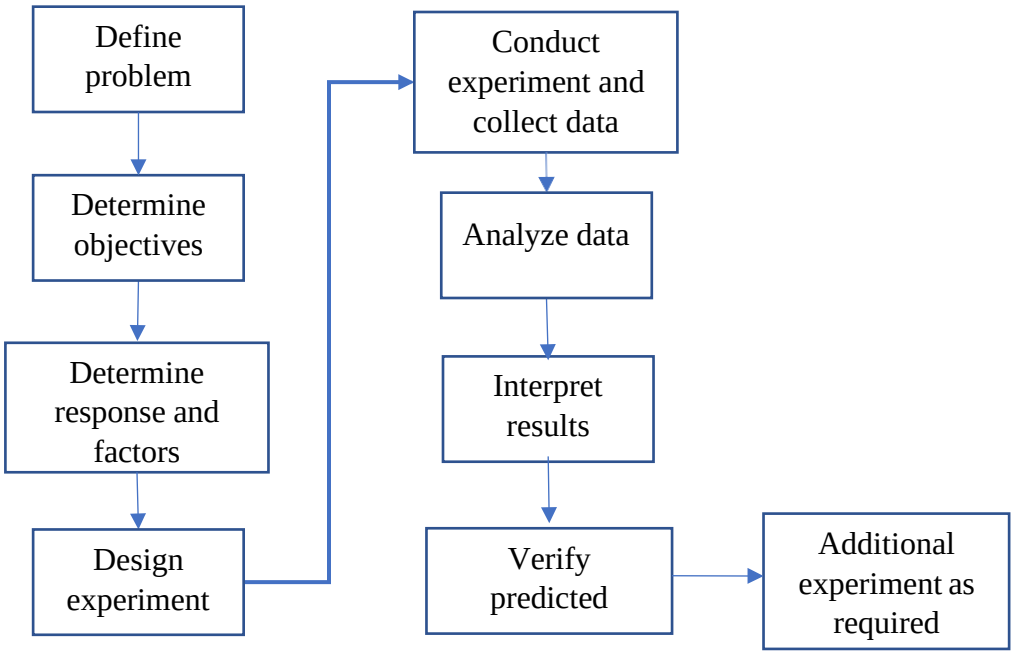


Fig.3.9 Experimental design process **[10]**

3.6 Design expert software

Design Expert software was developed by *State Ease*. This was originally made available in 1996 to assist with the execution of experimental designs, such as figuring out the best preparation recipe. In addition to optimization, this program can analyze the experimental variables. Depending on the experimental design to be used, there are three options for research directions in software. Options for screening, characterizing, and optimizing **[11]**.

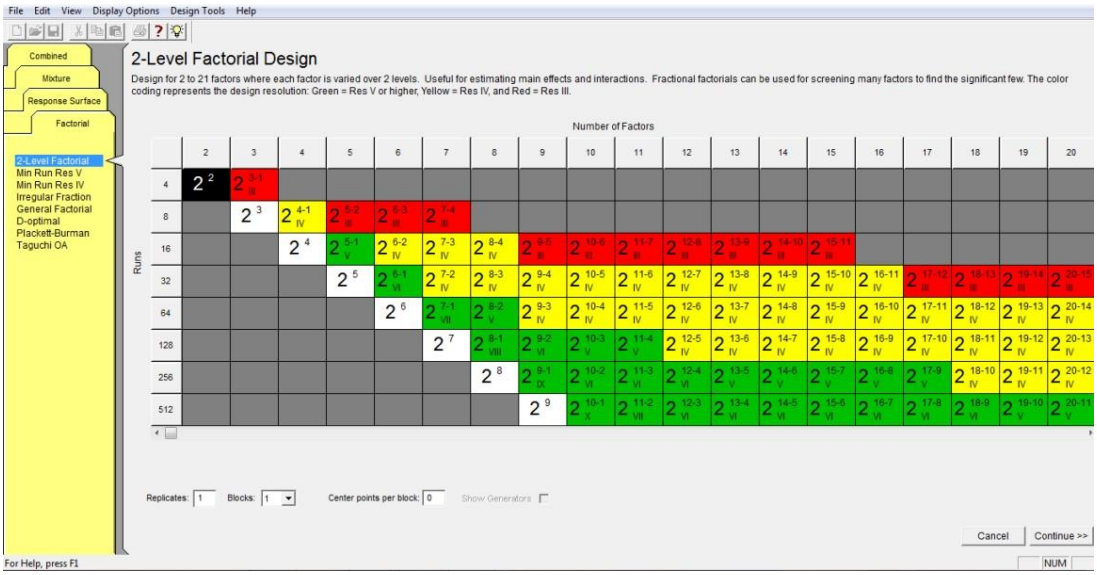


Fig.3.10 Interface of the design-expert program **[12]**

The program contains four design processes which are described as follows **[11]**:

- **Factorial design**

Factorial design is a method of modeling the connection between the response variable and one or more independent factors using regression equations. The most prevalent sort of design for process optimization is the factorial design. Factorials are used in research to examine the influence of numerous conditions on the study's outcomes, as well as the interactions between them.

- **Combined design**

Combined is a design of experiment (DOE) combination of factorial/RSM with the mixture. Used to investigate the relationships between variable composition and process variables in a single DOE.

- **Mixture design**

mixture is used to describe components in a formulation that alter in proportion to one another. To obtain a stable total value, the percentage of each variable must always rise. Even though the quantity of variable components is extremely tiny, they can still be employed because this approach has a highly sensitive reaction to these substances.

- **Response surface methodology (RSM)**

The response surface methodology (RSM) is a technique for assessing how the reaction to key influencing variables varies. This approach allows you to find an approximate relationship between the input and output variables. Indeed, the output response is affected by changes in the

input parameters, which are referred to as stimuli. These can be represented by random variables allowing for the representation of physical models or the adjustment of a mathematical function. A response surface research might have a variety of objectives **[12]**:

- Optimize (maximize/minimize) one or more response variables.
- Find a satisfactory compromise between several response variables.
- Build a mapping of the variation of a response in a design.
- Find out in which proportions you can mix previously selected components.

3.7 Analysis technique

3.7.1 Analysis of variance (ANOVA)

ANOVA is an important method for assessing the significance of an effect or a mathematical model. The analysis of the variance principle is based on calculating the total difference between the various measurements of the experimental design and the average of these data **[7]**.

- **Probability P**

The value of P is the most crucial statistic in the analysis of the variance table. This number can only be between 0 and 1. If it is less than 0.05, the influence is considered significant, and if it is less than 0.01; the factor is considered extremely significant **[7]**.

- **Coefficients of determination (R^2)**

The coefficient of determination R^2 is defined as the proportion of response changes explained solely by the model. The R^2 is therefore a measure of the model's quality, with values ranging from 0 to 1. If it is near to 1, the model can calculate the values of the measured responses. If it is 0, the model does not explain anything **[7]**. it is defined by **[8]**:

$$R^2 = \frac{variation\ due\ to\ regression}{total\ variation} \tag{7.3}$$

$$R^2 = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \tag{8.3}$$

When the value of the coefficient of determination is closer to 1, the results are a more representative model.

3.8 Conclusion

In conclusion, the plan of experiment and experimental design using Design Expert software with response surface methodology (RSM) also the analysis of results with analysis of variance technic (ANOVA) is a powerful approach that enables the determination of the optimal experimental conditions that will lead to the desired outcomes. This approach allows for the systematic examination of multiple variables and their interactions, resulting in a better understanding of the underlying factors that affect the outcome. This also helps to minimize the number of experimental runs required, saving time and resources. Overall, this methodology provides an efficient and effective framework for scientific experiments, making it a valuable tool for research in many fields.

Bibliography

1. Ranga, S., et al., *A review on Design of Experiments (DOE).* Int. J. Pharm. Chem. Sci, 2014. **3**(1): p. 216-24.
2. DAAS, M.S., *Plans d'expériences.*
3. BOUDISSA, F., *Influence des radiations micro-onde sur l'extraction de l'huile de grignon d'olive imprégné de margines*, 2011, Université de Tizi Ouzou-Mouloud Mammeri.
4. اسماعلية and يمينة, *Optimisation des conditions d'immobilisation de la lipase candida rugosa (CRL) par la Méthode des Surfaces de Réponse (RSM).* 2016.
5. Goupy, J., *Plans d'expériences*2006: Ed. Techniques Ingénieur.
6. SAHEB, F. and O. MAHDJOUB, *modélisation de conception de mélange Pour évaluer l'effet de la nature de sable sur les propriété physico mécanique de béton*, 2017.
7. BENDAHMA, F., H. BENMEKKI, and B. ABSAR, *MEMOIRE DE FIN D'ETUDES DE MASTER ACADEMIQUE.*
8. Yahiaoui, I., *Les plans d'expériences Initiation à la construction et l'analyse des plans factoriels complets à deux niveaux et du plan composite centré.* Université A. MIRA de Bejaia, 2015.
9. Goupy, J. and L. Creighton, *Introduction aux plans d'expériences-5e éd.: Toutes les techniques nécessaires à la conduite d'une étude*2013: Dunod.
10. *https://www.moresteam.com/toolbox/design-of-experiments.cfm*. [cited 2023 6 may].
11. Sopyan, I., D. Gozali, and R. Guntina, *Design-expert software (DOE): An application tool for optimization in pharmaceutical preparations formulation.* Int. J. Appl. Pharm., 2022: p. 55-63.
12. LAMARAT, A. and L. TOUGGOURTI, *Prédiction de la température de coupe de l'acier AISI D3: Application au tournage en utilisant la méthode de surface de réponse (RSM)*, 2017.

4

Optimization of mechanical behavior in bending and in compression of *hemp* fiber reinforced cementitious elaborated bio-composites

4.1 Introduction

The most important objectives of this research are to create mathematical models that can predict mechanical behavior (bending and compression) and to understand the mechanisms that control this particular behavior. The Design-Expert software (version 10), which is a special software for the design and analysis of experiments, is employed for all planned experiments as well as the statistical analysis of the results. The mechanical behavior of composites made from processed bio-sourced materials was investigated and statistically analyzed using response surface methodology (RSM). This statistical technique, in particular, permits the development of mathematical models that incorporate various characteristics that impact the bending and compression responses of these composites. It is primarily based on the outcomes of experiments.

4.2 Implementation of experimental designs

4.2.1 Responses

In this work, the breaking stress in bending and the breaking stress in compression have been selected as studied responses.

4.2.2 Factors influencing the responses

Both the stress at breaking in bending and compression can be affected by a variety of factors. The previous analysis done and discussed in Chapter III has shown that the length of fibers (A), the concentration of the alkaline NaOH in % (B), and the volume of fibers in the mortar in % (C) had an impact on these two responses resulting obtained from 3-point bending and compression tests. The levels: minimum, average, and maximum, of each variable are designated respectively in the form coded by: -1, 0, and +1. The different factors that were taken into consideration as well as the range of variation tolerated for each of them (study area) are summarized in Table 4.1 below.

Table 4.1 Levels of the factors used by the central composite design

Factors	Decoded variables symbols	Low level (-1)	Average level (0)	High level (+1)
Length (mm)	A	1	3	5
Alkaline (%)	B	2	3	4
Volume (%)	C	1	1.5	2

4.2.3 Response Surface Methodology

A set of statistical as well as mathematical techniques that are referred to as "Response Surface Methodology" abbreviated as RSM are based on fitting empirical models to experimental data gathered through the experimental design [1-3]. It was initially proposed and developed by Box and Wilson [1, 4] and has been extremely successful in a wide range of scientific fields. RSM provides a variety of advantages, including the ability to predict the model for each response, the capacity to construct a strong model with minimal experimental data points, the capability to assess the interaction effect between factors, and the potential to accurately identify the optimal response [1]. Regression analysis and statistical approaches are employed together in this methodology to display the whole experimental data set and predict the relationships between a response of interest, y (the dependent variable), and several related input or control variables, denoted by X_1 through X_k (independent variables). A limited calculation interval is one of the characteristics of this experimental design. The levels that are being used, denoted by the codes (-1) and (+1), indicate the minimum and maximum level values assigned to the components that are centered around a middle value ((0), respectively). In this investigation, bending and compression tests, on a bio-composite consisting of cement mortar reinforced with *hump* fibers were carried out using the parameters listed in Table 4.1. Based on the methodology RSM, the parameters that were used to build the experimental design were fiber length (A), % of NaOH (B), and volumetric fiber fraction (C). The Central Composite Design was used in this study. Regression models can be rendered provided by the RSM technique and may be used to assess how different variables and their levels affect the mechanical properties (in this case, bending and compressive breaking stresses). In this work, a fitting analysis has suggested the implementation of a model involving a cubic function equation. The third-order polynomial equation used to fit the experimental data and determine the relevant model terms is expressed as the following:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \ldots + \beta_{kk} X_1^k + \beta_{kk+1} X_2^k + \ldots + \varepsilon \quad (1.4)$$

In this equation:

- Y represents the response variable.
- X_1, X_2, X_3, ..., X_k are the input variables or factors.
- β_0, β_1, β_2, β_3, ..., β_{kk}, β_{kk+1}, ... are the regression coefficients that need to be estimated.
- X_1^2, X_2^2, X_3^2, ..., X_1^k, X_2^k, ... represent the squared terms and higher-order terms of the input variables.
- X_1X_2, X_1X_3, X_2X_3, ..., $X_1X_2^{k-1}$, ... represent the interaction terms between the input variables.
- ε represents the error term.

The number of terms in the polynomial model depends on the desired degree of the polynomial and the number of input variables. The degree of the polynomial determines the maximum power to which the input variables are raised. For example, in a quadratic model, the maximum power is 2, resulting in squared terms and interaction terms. In a cubic model, the maximum power is 3, resulting in squared terms, interaction terms, and cubic terms. The coefficients β_0, β_1, β_2, β_3, ..., β_{kk}, β_{kk+1}, ... can be estimated using various regression techniques such as least squares regression or maximum likelihood estimation. The goal is to find the values of these coefficients that best fit the observed data and provide an accurate representation of the relationship between the input variables and the response variable. Instead of performing a test campaign consisting of 27 experiments for the bending test and a further 27 for the compression test, this number can be reduced to just 17 for each of these two tests, by selecting and implementing an experimental design with RSM and Composite Central Design and using Design expert software. Table 4.2 illustrates the levels and factors involved in this experimental design.

4.2.4 Design of experiments

In this study, two cubic polynomial models have been considered. To realize this part of the work, we have opted to implement the central composite design, which is a second-degree design (Fig 4.1).

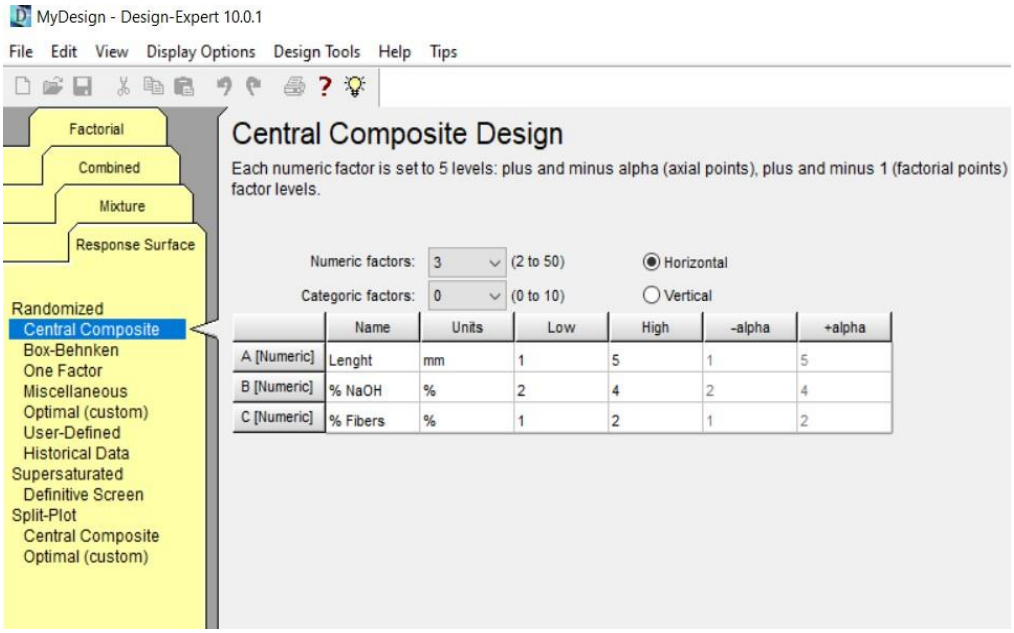


Fig. 4.1 Central composite design considered in this study

4.2.5 Central composite design

A central composite design is a type of experimental design used in response surface methods to create polynomial models for a response variable without having to do a full three-level factorial experiment. In this work, we have implemented the central composite design (Fig.1.4). A set of 17 experiments were performed (three points in the center of the model). The analysis was performed to evaluate the response functions. The cubic model shown in the previous Equation (1.4) can predict the stresses at failure in bending and compression. The tests have been numbered from 1 to 17, according to the following relationship:

$$N = 2^f + 2f + C_p \quad (2.4)$$

Where:

N: the overall number of points required in the plan

f: is the number of factors

Cp: the number of central points

The planning matrix lists several combinations of the basic factors (A), (B), and (C). The planning matrix based on the coded factors is displayed in Table 4.2. Table 4.3 depicts the one based on those that have been decoded. As shown in Fig.4.2, the data in this matrix will be statistically processed to estimate the coefficients of the mathematical models.

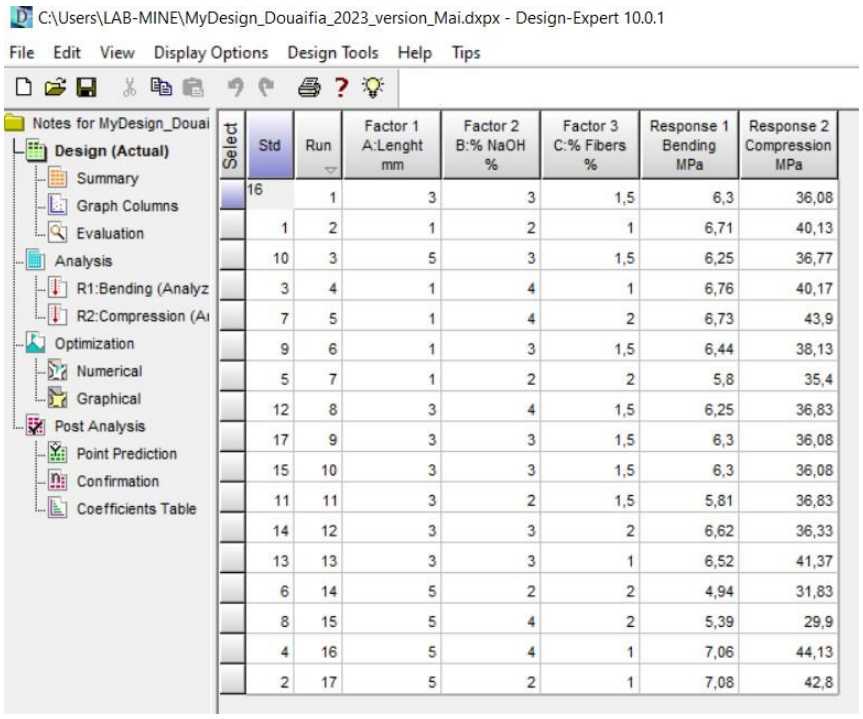


Fig.4.2 Central composite matrix used in this investigation

Table 4.2 Composite central coded matrix: factors (X_1 ,X_2 and X_3), responses (Y_1 and Y_2) obtained experimentally

N°	Factor 1 (X_1) Fibers length	Factor 2 (X_2) NaOH **(%)**	Factor 3 (X_3) Fibers (%)	Response 1 (Y_1) Bending break stress (MPa)	Response 2 (Y_2) Compression break stress (MPa)
1*	0	0	0	6.30	36.08
2	-1	-1	-1	6.71	40.13
3	1	0	0	6.25	36.77
4	-1	1	-1	6.76	40.17
5	-1	1	1	6.73	43.90
6	-1	0	0	6.44	38.13
7	-1	-1	1	5.8	35.40
8	0	1	0	6.25	36.83
9*	0	0	0	6.30	36.08
10*	0	0	0	6.30	36.08
11	0	-1	0	5.81	36.83
12	0	0	1	6.62	36.33
13	0	0	-1	6.52	41.37
14	1	-1	1	4.94	31.83
15	1	1	1	5.39	29.9
16	1	1	-1	7.06	44.13
17	1	-1	-1	7.08	42.80

*Three points in the center of the model

Table 4.3 Composite central decoded matrix: factors (A, B and C), responses (Y_1 and Y_2)

N°	Factor 1 (A) Fibers length	Factor 2 (B) NaOH (%)	Factor 3 (C) Fibers (%)	Response 1 (Y_1) Bending break stress (MPa)	Response 2 (Y_2) Compression break stress (MPa)
1*	3	3	1.5	6.30	36.08
2	1	2	1	6.71	40.13
3	5	3	1.5	6.25	36.77
4	1	4	1	6.76	40.17
5	1	4	2	6.73	43.90
6	1	3	1.5	6.44	38.13
7	1	2	2	5.8	35.40
8	3	4	1.5	6.25	36.83
9*	3	3	1.5	6.30	36.08
10*	3	3	1.5	6.30	36.08
11	3	2	1.5	5.81	36.83
12	3	3	2	6.62	36.33
13	3	3	1	6.52	41.37
14	5	2	2	4.94	31.83
15	5	4	2	5.39	29.9
16	5	4	1	7.06	44.13
17	5	2	1	7.08	42.80

*Three points in the center of the model

4.3 Results and discussion

4.3.1 ANOVA and Regression Models

In compliance with the pre-established experimental design, the cementitious composite manufactured with *Hemp* fiber-reinforced mortar was exposed to experimental bending and compression tests. The averaged results obtained from these tests are reported in Table 4.3. Applying the polynomial regression models provided in Table 4.4, including linear, two-factor interaction (2FI), quadratic, and cubic, each of the responses (bending and compression stresses) was analyzed sequentially. These models were statistically evaluated by determining the most significant variables that affect the mechanical characteristics of the mortar reinforced with bio-fibers. In Tables 4.5 and 4.6, respectively, the initial ANOVA outcomes for the bending and compression strengths can be seen. The sum of squares and mean square values for each parameter are displayed in the ANOVA Tables, and the p-value and F-value are defined as the ratio of the respective mean square effect and mean square error. In this research, we used a Central Composite Design, which provides for the development of a test campaign made of a set of 17 tests, including three points corresponding to the model's center. The number of trials ranged from 1 to 17. The

planning matrix is shown in Tables 4.2 and 4.3 covers different combinations of the factors: fiber length (A), percentage of NaOH (B), and volumetric fiber fraction (C). Mathematical models that depict the responses of bending stresses (Y_1) and compression stresses (Y_2) as a function of the independent variables A, B, and C were fitted using the results from the experimental trials conducted on the samples. The predictive models were expressed in terms of coded and decoded variables in the following equations:

1- Bending

The final equation in terms of coded factors:

$$Y_1 = 6.31 - 0.095X_1 + 0.22X_2 + 0.05X_3 - 0.069X_1X_2 - 0.36X_1X_3 + 0.17X_2X_3 + 0.031X_1^2 - 0.28X_2^2 + 0.26X_3^2 - 0.051X_1X_2X_3 - 0.044X_1^2X_2 - 0.64X_1^2X_3 - 0.096X_1X_2^2 \quad (3.4)$$

The final equation in terms of decoded factors:

$$Y_1 = 11.6793 - 3.20981A + 0.32734B - 6.26283C + 0.39687AB + 1.72625AC + 0.49125BC + 0.5233A^2 - 0.13992B^2 + 1.02282C^2 - 0.05125ABC - 0.010934A^2B - 0.32187A^2C - 0.048125AB^2 \quad (4.4)$$

2-Compression

The final equation in terms of coded factors:

$$Y_1 = 36.69 - 1.23X_1 + 0.79X_2 - 3.12X_3 - 1.14X_1X_2 - 3.03X_1X_3 + 0.65X_2X_3 + 1.91X_3^2 - 1.46X_1X_2X_3 \quad (5.4)$$

The final equation in terms of decoded factors:

$$Y_1 = 69.58704 - 0.95625A - 6.034754B - 37.18143C + 1.62625AB + 1.37AC + 5.695BC + 7.64114C^2 - 1.465ABC \quad (6.4)$$

Table 4.4 Fit summary of the two statistical models (bending stress and compressive stress)

Test type	Source	SD	R^2	Adjusted	Predicted	PRESS	Observation
Bending							
	Linear	0.42	0.5499	0.4461	0.0732	4.72	
	2FI	0.32	0.8043	0.6868	0.3313	3.41	
	Quadratic	0.32	0.8621	0.6847	-0.1886	6.05	
	Cubic	**0.012**	**0.9999**	**0.9995**	**0.8616**	**0.70**	**Suggested**
Compression							
	Linear	3.10	0.4879	0.3697	-0.1688	285.14	
	2FI	1.95	0.8446	0.7514	-0.3546	330.47	
	Quadratic	1.79	0.9081	0.7899	-0.8101	441.60	
	Cubic	**0.82**	**0.9918**	**0.9561**	**-12.059**	**3185.95**	**Suggested**

The suitability of the regression models to explain the experimental data at the 95% confidence level was explored from the ANOVA results. The significance of the principal effects and interaction effects in the predicting models was considered based on their probability values (p-values). P-values less than 0.05 require rejection of the null hypothesis denoting that the particular term significantly affects the measured response of the system [5-7].

From the ANOVA Tables, 4.5 and 4.6, "F-value" of the models is 2645.36 for bending and 41,68 for compression respectively, which implies that the two models are significant. There is only a 0.01% chance that the model could occur due to noise [8, 9]. Probability values less than 0.05 indicate that the model terms are significant [10, 11]. In the case of the bending stresses, the factors A, B, and C, the interactions AB, BC, and AC, the quadratic effects A^2, $B^{2,}$ and C^2 and the cubic effects ABC, A^2B, A^2C, and AB^2 are significant terms in the model. As well as the compression, the factors A, B, and C, the interactions AB, AC, and BC, the quadratic effect $C^{2,}$ and the cubic effects ABC are significant terms in the model. The R^2 adjusted R^2 and predicted R^2 determination coefficients were assessed to accurately analyze the regression analysis. R^2 represents the percentage of overall response variation that the models have predicted. The models' suitability and the precision of the derived values are evidenced by correlation coefficients that are close to 1 [5, 12]. Regression analysis uses the predicted R^2 to illustrate how well the model predicts responses for new observations. Because it is derived from observations not included in the model estimation, the predicted R^2 could be more useful for comparing models than the adjusted R^2. The coefficients of determination R^2 and adjusted R^2 are indicative of the adequacy of the polynomial fit and must be approximately 0.20 of one another, to be in reasonable agreement [6]. The two models have high coefficients of determination (R^2=0.9999 for bending stresses and R^2=**0.9766** for compression stresses). When the R^2 and adjusted R^2 are significantly different, there is a strong probability that non-significant terms are included in the model [13]. According to this study, for the first response (Y_1), the predicted and adjusted R^2 values are close to 1.00, **0.8616**, and **0.9995**, respectively, suggesting that the predicted and experimental bending stresses are in excellent agreement. This model can reveal 99.99% of the variability, according to the excellent agreement between the R^2 and the experimental values, which is 0.9999. Likewise, the values of R^2, predicted R^2, and adjusted R^2 for the second response (Y_2) are, respectively, **0.9766**, **0.6872,** and **0.9531**, indicating a good correlation between the predicted and observed values. A model is generally regarded as credible if its coefficient of variation (CV) is not higher than 15% [14]. The (CV) is calculated as the ratio

of the standard error of the estimate to the mean value of the observed response. Because of this, the obtained coefficient of variation values in this work of 0.19 % for bending stresses and 2.24 % for compression stresses indicate that the experiments were highly precise and reliable.

Table 4.5. ANOVA results and statistical parameters for bending stress

Source	*Sum of squares*	*df*	*Mean square*	*F-Value*	*P-Value Prob > F*	*Observation*
Model	*5.09*	*13*	*0.39*	*2645.36*	*< 0.0001*	***Significant***
A-Lenght	*0.018*	*1*	*0.018*	*121.91*	*0.0016*	
B-% NaOH	*0.097*	*1*	*0.097*	*653.77*	*0.0001*	
C-% Fibers	*5.000E-003*	*1*	*5.000E-003*	*33.77*	*0.0101*	
AB	*0.038*	*1*	*0.038*	*255.38*	*0.0005*	
AC	*1.03*	*1*	*1.03*	*6953.86*	*< 0.0001*	
BC	*0.23*	*1*	*0.23*	*1538.61*	*< 0.0001*	
A^2	*2.526E-003*	*1*	*2.526E-003*	*17.06*	*0.0257*	
B^2	*0.22*	*1*	*0.22*	*1462.53*	*< 0.0001*	
C^2	*0.18*	*1*	*0.18*	*1183.15*	*< 0.0001*	
ABC	*0.021*	*1*	*0.021*	*141.92*	*0.0013*	
A^2B	*3.062E-003*	*1*	*3.062E-003*	*20.68*	*0.0199*	
A^2C	*0.66*	*1*	*0.66*	*4478.23*	*< 0.0001*	
AB^2	*0.015*	*1*	*0.015*	*100.11*	*0.0021*	
Residual	*4.442E-004*	*3*	*1.481E-004*			
Lack of Fit	*4.442E-004*	*1*	*4.442E-004*			
Pure Error	*0.000*	*2*	*0.000*			
Cor Total	*5.09*	*16*				
Fit Statistics	*Std Dev =0.012*		R^2= ***0.9999***			
	Mean =6.31		*Adjusted* R^2 = ***0.9995***			
	C.V. % 0.19		*Predicted* R^2 = ***0.8616***			
	Adeq Precision = 193.798					

Table 4.6. Initial ANOVA results and statistical parameters for compression stress

Source	Sum of squares	df	Mean square	F-Value	P-Value Prob > F	Observation
Model	238.25	8	29.78	41,68	< 0.0001	**Significant**
A-Lenght	*15.13*	*1*	*15.13*	*21,17*	*0,0018*	
B-% NaOH	*6.30*	*1*	*6.30*	*8,82*	*0,0179*	
C-% Fibers	*97.59*	*1*	*97.59*	*136,58*	*< 0.0001*	
AB	*10.44*	*1*	*10.44*	*14,61*	*0,0051*	
AC	*73.21*	*1*	*73.21*	*102,45*	*< 0.0001*	
BC	*3.38*	*1*	*3.38*	*4,73*	*0,0413*	
C^2	*15.03*	*1*	*15.03*	*21,03*	*0,0018*	
ABC	*17.17*	*1*	*17.17*	*24,03*	*0,0012*	
Residual	5.72	8	0.71			
Lack of Fit	*5.72*	*6*	*0.95*			
Pure Error	*0.000*	*2*	*0.000*			
Cor Total	1050.59	18				
Fit Statistics	Std Dev =0.85		R^2 = **0.9766**			
	Mean =23.81		Adjusted R^2 = **0.9531**			
	C.V. % 2.24		Predicted R^2 = **0.6872**			
	Adeq Precision = 2.24					

4.3.2 Models validation

One of the most crucial steps in the design of experiments is model validation. It involves comparing the theoretical test results predicted by the model with the real experiment outcome. The model is validated if the predicted responses closely resemble the experimental ones. The model is rejected otherwise. Primary validation of the model consists of checking that the calculated and measured responses are correlated which allows us to judge more precisely the quality of the adjustment carried out. As can be seen in Tables 4.7 and 4.8, the comparison between the columns Y_{exp} (measured responses) and Y_{cal} (responses predicted by the model) and the relative error calculated from the relationship confirm that the fit is of very high quality. This correlation can also be illustrated by plotting the measured responses against the calculated responses [4, 5], as illustrated in Figs 4.3 and 4.4, which demonstrates a strong correlation between them, with a correlation coefficient of R^2=0.9999 for bending response and R^2=0.9766 for compression response.

Table 4.7 Comparison between the experimental bending responses with those computed using the model

N°	Factor 1 (A) Fibers length	Factor 2 (B) NaOH (%)	Factor 3 (C) Fibers (%)	Response 1 (Y_1) Experimental	Respons e 1 (Y_1) Predicted	Error % Response 1 Bending
1	3	3	1.5	6.300	6.308	0.127
2	1	2	1	6.710	6.712	0.030
3	5	3	1.5	6.250	6.244	0.096
4	1	4	1	6.760	6.762	0.030
5	1	4	2	6.730	6.732	0.030
6	1	3	1.5	6.440	6.434	0.093
7	1	2	2	5.800	5.802	0.0345
8	3	4	1.5	6.250	6.244	0.096
9	3	3	1.5	6.300	6.308	0.127
10	3	3	1.5	6.300	6.308	0.127
11	3	2	1.5	5.810	5.804	0.103
12	3	3	2	6.620	6.614	0.091
13	3	3	1	6.520	6.514	0.092
14	5	2	2	4.940	4.942	0.040
15	5	4	2	5.390	5.392	0.037
16	5	4	1	7.060	7.062	0.028
17	5	2	1	7.080	7.082	0.028

Table 4.8 Comparison between the experimental compression responses with those computed using the model

N°	Factor 1 (A) Fibers length	Factor 2 (B) NaOH (%)	Factor 3 (C) Fibers (%)	Response 1 (Y_1) Experimental	Response1 (Y_1) Predicted	Error % Response 1
1	3	3	1.5	36.080	36.686	1.680
2	1	2	1	40.130	40.104	0.065
3	5	3	1.5	36.770	35.456	3.574
4	1	4	1	40.170	39.747	1.053
5	1	4	2	43.900	43.778	0.278
6	1	3	1.5	38.130	37.916	0.561
7	1	2	2	35.400	35.675	0.777
8	3	4	1.5	36.830	37.480	1.765
9	3	3	1.5	36.080	36.686	1.680
10	3	3	1.5	36.080	36.686	1.680
11	3	2	1.5	38.300	35.892	2.547
12	3	3	2	36.330	35.472	2.362
13	3	3	1	41.370	41.720	0.846
14	5	2	2	31.830	32.380	1.728
15	5	4	2	29.900	30.053	0.512
16	5	4	1	44.130	43.982	0.336
17	5	2	1	42.800	43.049	0.582

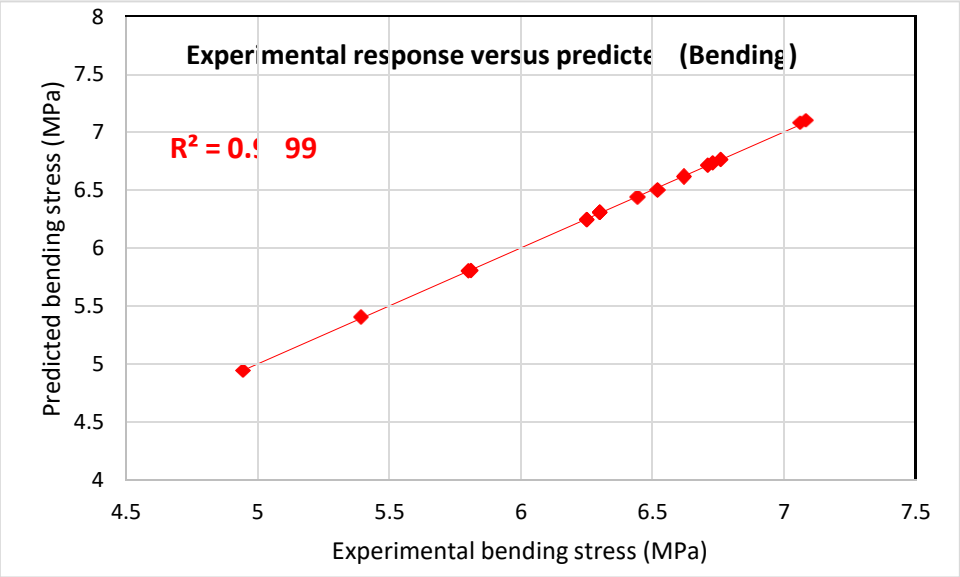


Fig.4.3 Measured-bending response versus predicted

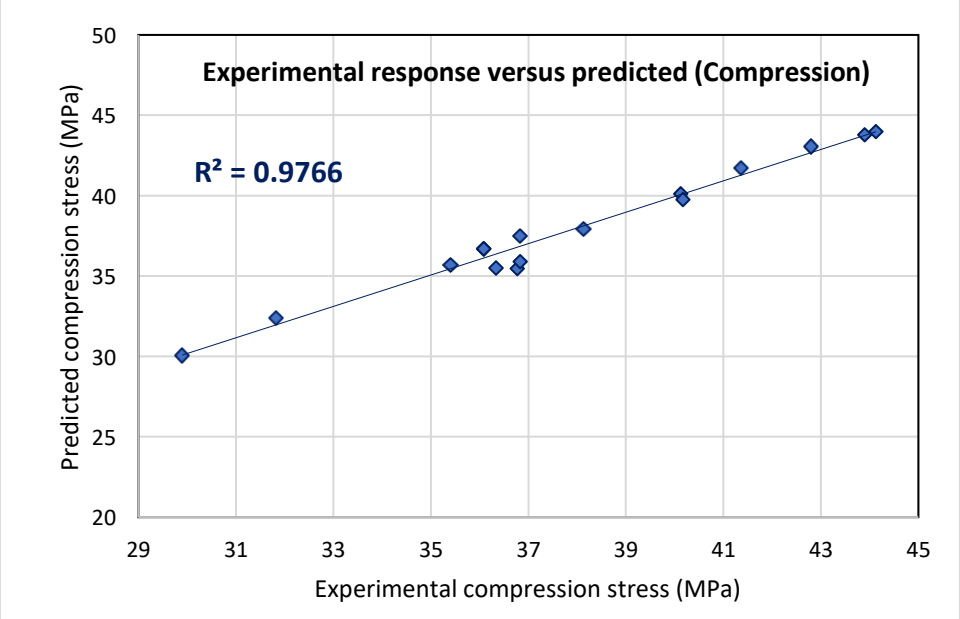


Fig.4.4 Measured-compression response versus predicted

Fig.4.5 shows the actual versus predicted plot of two responses (Y_1) and (Y_2). According to this plot we can notice that for both cases the distribution of the values of the responses Y1 and Y2 are uniformly scattered along a bisector at 45 degrees. This allows us to validate the both models [15, 16] . The relationship between the expected values of the response based on the model equation and the actual values acquired in the experiment were examined. With an R^2 value of 0.9999 and

0.9766 for bending and compression stresses, respectively, it is evident that the linear regression fit is appropriate and that the model accurately fits the experimental data.

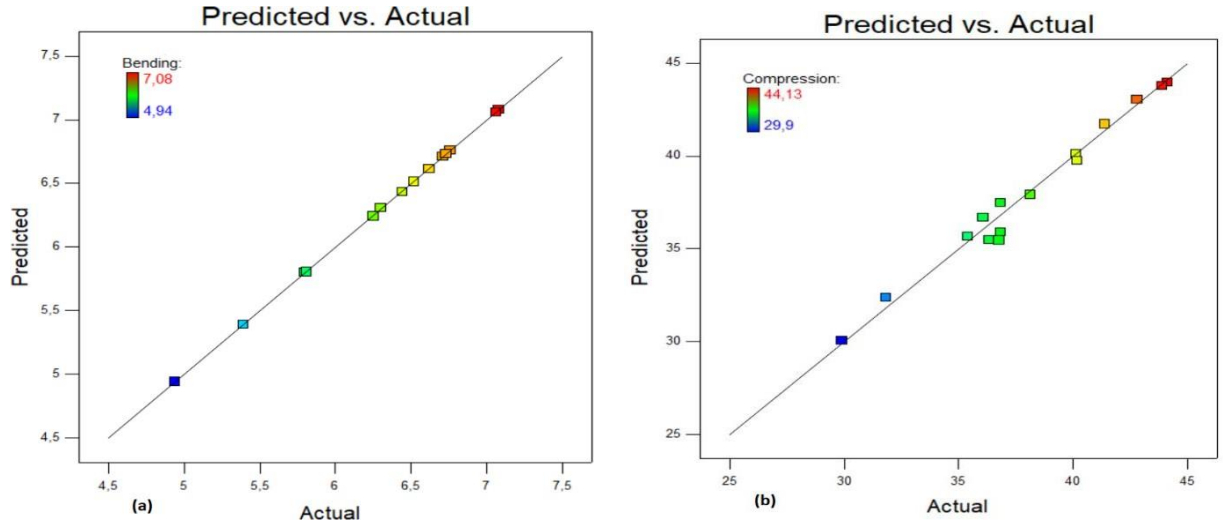


Fig.4.5 Plots of predicted versus experimental values for (a) bending (b) compression

4.3.3 Residual analysis

Residual analysis is used to validate and improve statistical models, to detect specification errors, to identify outliers and to evaluate the fit of the model to the observed data. It is an important tool to ensure the reliability and interpretability of the results of a statistical study. An analysis of the residuals (see Table **4.11**) can achieve the appreciation of the quality of the second order model.

$$Av_{res} = \frac{\sum_{i=1}^{N} e_i}{N} \qquad (7.4)$$

Where:
e_i: residuals
N : number of trials
The average value of the residuals according to Equation 4.10 is of the order of 0,02352 % and is close to 0 [5].

Table 4.9 Computation of the residues in case of bending response

N°	Response (Experimental)	Response (Computed)	Residue
1	6.300	6.308	0.008
2	6.710	6.712	0.002
3	6.250	6.244	-0.006
4	6.760	6.762	0.002
5	6.730	6.732	0.002
6	6.440	6.434	-0.006
7	5.800	5.802	0.002
8	6.250	6.244	-0.006
9	6.300	6.308	0.008
10	6.300	6.308	0.008
11	5.810	5.804	-0.006
12	6.620	6.614	-0.006
13	6.520	6.514	-0.006
14	4.940	4.942	0.002
15	5.390	5.392	0.002
16	7.060	7.062	0.002
17	7.080	7.082	0.002
		Sum	0.004
		Average	0.00023

Table 4.10 Computation of the residues in case of compression response

N°	Response (Experimental)	Response (Computed)	Residue
1	36.080	36.686	0.606
2	40.130	40.104	-0.026
3	36.770	35.456	-1.314
4	40.170	39.747	-0.423
5	43.900	43.778	-0.122
6	38.130	37.916	-0.214
7	35.400	35.675	0.275
8	36.830	37.480	0.65
9	36.080	36.686	0.606
10	36.080	36.686	0.606
11	38.300	35.892	-2.408
12	36.330	35.472	-0.858
13	41.370	41.720	0.35
14	31.830	32.380	0.55
15	29.900	30.053	0.153
16	44.130	43.982	-0.148
17	42.800	43.049	0.249
		Sum	-1.468
		Average	-

4.3.4 Graphic analysis of results

A major advantage of experimental designs is the presentation of results in graphical form. Several graphs are available to interpret the equation of the empirical model. In the case of designs with response surfaces, this restitution is essentially done with 3D response surfaces curves.

4.3.5 3D response surfaces

Figs.4.6 and 4.7 display the 3D response surfaces in the case of bending and compression stresses responses respectively. According to the cubic models given in Eqs. 4.4 and 6.4 and Tables 4.5 and 4.6, three interactions combination between the manufacturing parameters are significant for both bending and compression stresses :(A-B),(B-C) and (A-C).Considering a fixed value for parameter C=1.5 % (percentage of fibers), the maximum value for the bending test response was found to be around 6.25 MPa and to be depending on the combination of processing conditions. This value was achieved for a bio-composite with a fiber length of 3 mm and that had been treated with 4% NaOH. For the interaction. Considering a fixed value for parameter B=3 % (NaOH treatment), case of (A-

C) interaction, the maximum value for the bending test response was found to be around 6.62 MPa. This value was achieved for a bio-composite with a fiber length of 3 mm and percentage of fibers of 2 %. For a fixed value for parameter A=3 mm (fiber length), case of (B-C) interaction, the maximum value for the bending test response was found to be around 6.62 MPa. This value was achieved for a bio-composite with 3% NaOH and percentage of fibers of 2 %. The maximum value for the compression test response was determined to be around 38.16 MPa and to vary depending on the combination of processing conditions when parameter C=1.5% (percentage of fibers) was fixed. This result was obtained using a bio-composite with 1 mm long fibers that had undergone 3% NaOH treatment. The highest value for the compression test response had been found to be close to 41.37 MPa when parameter B=3% (NaOH treatment) was fixed. This result was obtained using a bio-composite with 1% fiber content and a 3 mm fiber length. In the event of (B-C) interaction, given a fixed value for parameter A=3 mm (fiber length), the same maximum value of 41.37 MPa of the response in the compression test was reached for a bio-composite that has endured 3% NaOH treatment and 1 % volumetric fiber reinforcement.

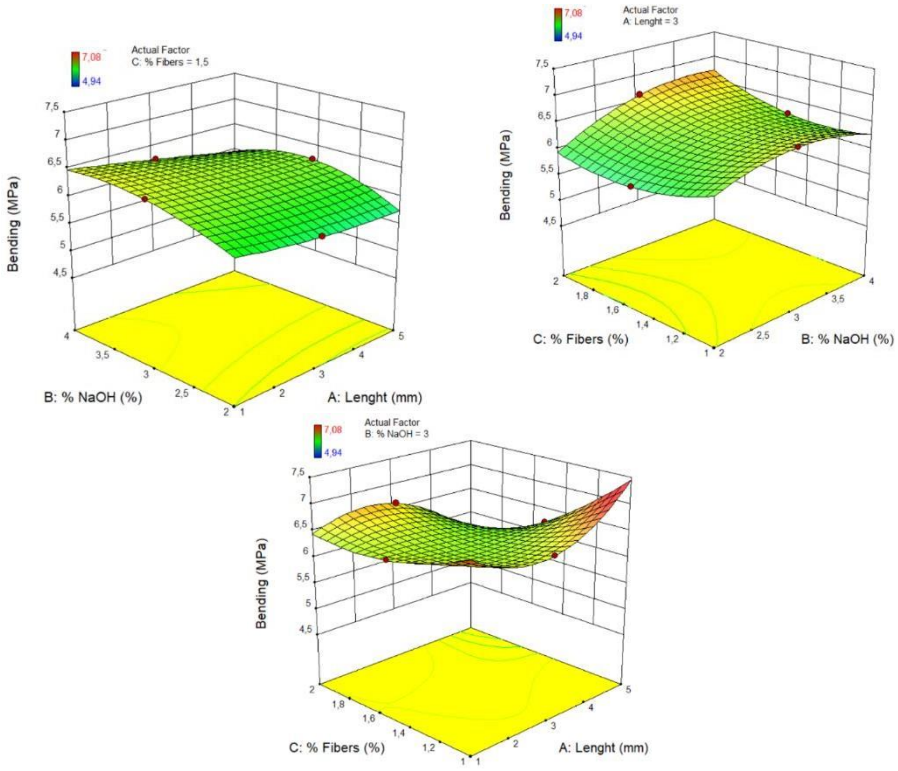


Fig.4.6 3D response surface plot for the binary interactions AB),(B-C) and (AC) effects of the bio-composite manufacturing parameters on the bending response

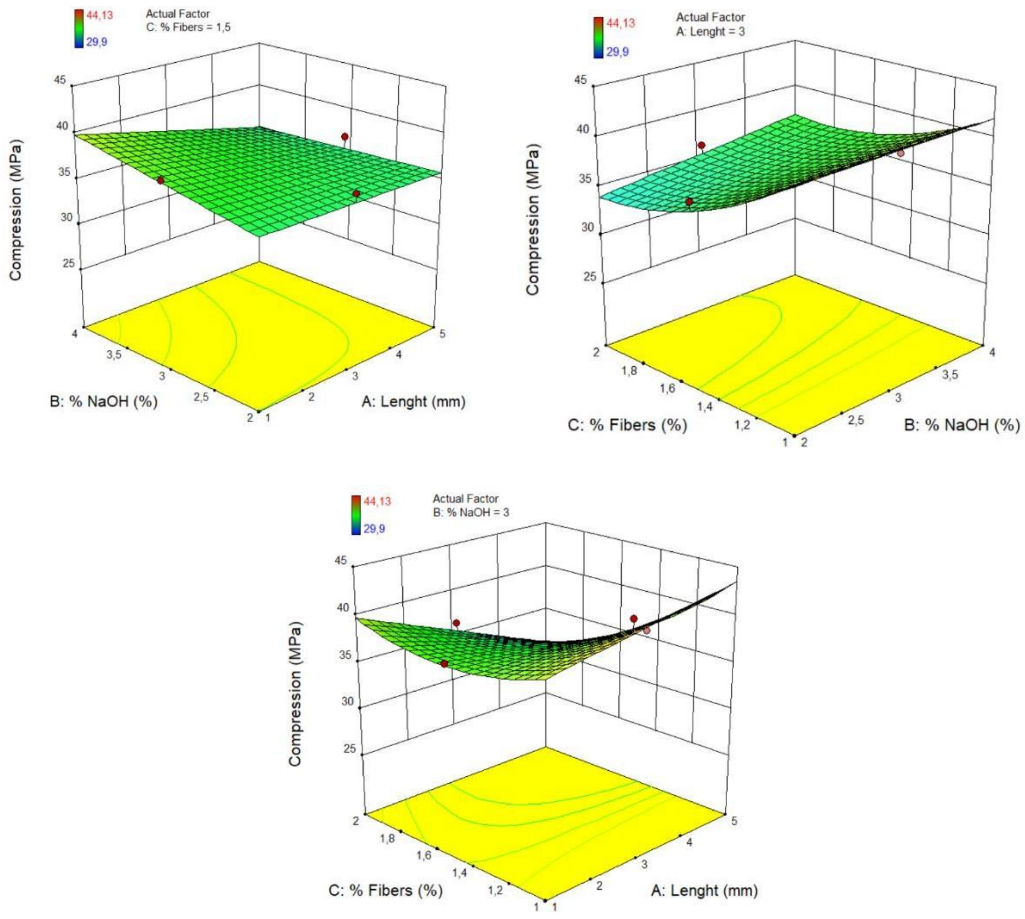


Fig.4.7 3D response surface plot for the binary interactions AB),(B-C) and (AC) effects of the bio-composite manufacturing parameters on the compression response

4.3.6 Optimization of responses

The final step is to find the combination of the factors that gives the optimal response. The response surface methodology (RSM) was used to select the processing settings that would result in the bio-composite's bending and compressive properties being optimized. Three factors were used to perform the optimization: fiber length (A), NaOH % (B) and volumetric fiber percentage (C). Based on the experimental findings confirmed in Fig.4.8, the optimal values of the responses, with target values of the objective function close to 1 (or 100%) for both tests (i.e. bending and compression), are selected as the most significant parameter values concerning the response factor. The RSM process tends to find, among a multitude of solutions, the 10 best cases. In this context, the most efficient response values recorded for bending and compression stresses are respectively equal to 7.08 MPa and 43.05 MPa. They were recorded for the bio-composite with 1% V fiber, a fiber length of 5 mm, and treated with 2 % NaOH as depicted in Fig.4.8.

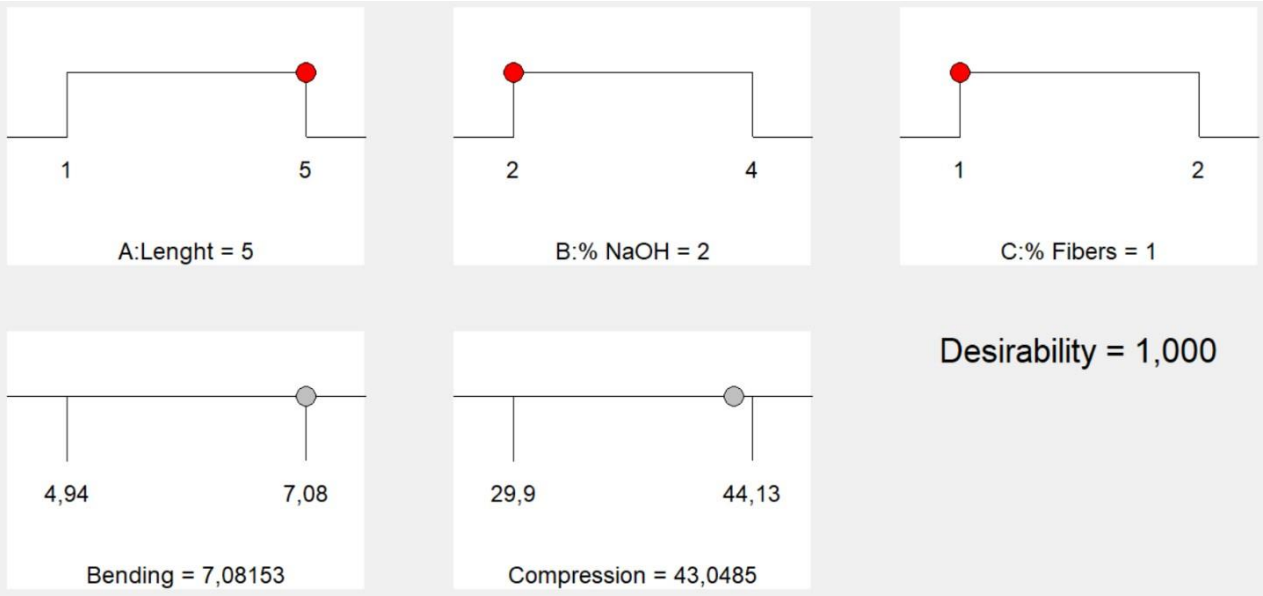


Fig.4.8 The optimum conditions for the bio-composite manufacturing parameters given the results of the bending and compression tests

4.5 Conclusion

This chapter examines the experimental findings of 3-point bending and compression mechanical testing of *hemp* fiber-reinforced bio-mortar specimens. These findings indicate that using *hemp* fibers into mortar can greatly enhance mechanical qualities in compression and bending. The ANOVA analysis shows that the cubic models developed in the two trials provide the best choice of elaboration parameters for *hemp* fiber-reinforced mortars, resulting in the maximization of their mechanical properties under flexural and compressive stresses. Finally, statistical analysis demonstrated that the experimental study's outcomes are efficient and dependable.

Bibliography

1. Lin, H.-J. and C.-I. Liao, *Compressive strength of reinforced concrete column confined by composite material.* Composite Structures, 2004. **65**(2): p. 239-250.
2. Ombres, L., *Flexural analysis of reinforced concrete beams strengthened with a cement based high strength composite material.* Composite Structures, 2011. **94**(1): p. 143-155.
3. Rimshin, V. and P. Truntov. *An integrated approach to the use of composite materials for the restoration of reinforced concrete structures*. in *E3S Web of Conferences*. 2019. EDP Sciences.
4. Zollo, R.F., *Fiber-reinforced concrete: an overview after 30 years of development.* Cement and concrete composites, 1997. **19**(2): p. 107-122.
5. Deghboudj, S., W. Boukhedena, and H. Satha, *Optimization of Bending and Compressive Strength Behavior of Agave americana Fiber Reinforced Cementitious Composite Using Response Surface Methodology.* Periodica Polytechnica Civil Engineering, 2023.
6. Labib, W.A., *Plant-based fibres in cement composites: A conceptual framework.* Journal of Engineered Fibers and Fabrics, 2022. **17**: p. 15589250221078922.
7. Khan, M., et al., *Basalt fibers in modified whisker reinforced cementitious composites.* Periodica Polytechnica Civil Engineering, 2022. **66**(2): p. 344-354.
8. Felekoğlu, B., *Effects of loading conditions and specimen thickness on the flexural behavior of fiber-reinforced cementitious composites.* Periodica Polytechnica Civil Engineering, 2014. **58**(3): p. 279-291.
9. Said, K.A.M. and M.A.M. Amin, *Overview on the response surface methodology (RSM) in extraction processes.* Journal of Applied Science & Process Engineering, 2015. **2**(1): p. 8-17.
10. Yazici, E. and H. Deveci, *Extraction of metals from waste printed circuit boards (WPCBs) in H2SO4–CuSO4–NaCl solutions.* Hydrometallurgy, 2013. **139**: p. 30-38.
11. Mongomery, D., *Montgomery: design and analysis of experiments.* John Willy & Sons, 2017.
12. Komuraiah, A., N.S. Kumar, and B.D. Prasad, *Chemical composition of natural fibers and its influence on their mechanical properties.* Mechanics of composite materials, 2014. **50**(3): p. 359-376.
13. Hill, W.J. and W.G. Hunter, *A review of response surface methodology: a literature survey.* Technometrics, 1966. **8**(4): p. 571-590.
14. Myers, R.H., A.I. Khuri, and W.H. Carter, *Response surface methodology: 1966–l988.* Technometrics, 1989. **31**(2): p. 137-157.
15. Mohammed, B.S., et al., *Deformation properties of rubberized engineered cementitious composites using response surface methodology.* Iranian Journal of Science and Technology, Transactions of Civil Engineering, 2021. **45**: p. 729-740.
16. Hassan, M.Z., et al., *Optimization of tensile behavior of banana pseudo-stem (Musa acuminate) fiber reinforced epoxy composites using response surface methodology.* Journal of Materials Research and Technology, 2019. **8**(4): p. 3517-3528.

General conclusion

The study carried out in this research had the objective of the development of new bio-sourced composites made of cement matrix reinforced by *hemp* fibers based on an experimental design built with central composite design (CCD) of Response Surface Methodology (RSM). To identify the changes in the mechanical behavior of bio-mortar samples under bending and compressive affected by the three influencing parameters which are (A): fiber length, (B): concentration of NaOH, and (C): volumetric fiber percentage.

The analysis of characterizations carried out in this work has led to many conclusions that allow us to consider various research perspectives on *hemp* fibers and their bio-composites:

- Chemical treatment with sodium hydroxide (NaOH) has been useful in enhancing the surface of fibers by removing undesirable elements (lignin, pectin, waxes, etc.), resulting in good adherence and improved interface quality between the fibers and the matrix.
- The analysis of variance (ANOVA) shows that the coefficients of determination are of high values (close to 1) which refers to excellent agreement between the predicted and the experimental values.
- The model was credible due to the coefficient of the variation value lower than 15%, the obtained results were 0,19% in bending and 2.24% in compression.
- The correlation was strong between measured responses and responses predicted by the model with a coefficient R^2=0.9999 for bending response and R^2=0.9766 for compression response which allows us to validate the models.
- The optimization results show the best mechanical properties were obtained for a fiber volume of 1%, with a length of 5 mm treated with 2% NaOH corresponding to 7.08 MPa in bending and 43.05 MPa in compression.