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Sujet

Study and characterization of a cementitious eco-composite incorpring *Agave Americana* plant fibers

Présenté et soutenu publiquement, le **13/06/2022**, devant le jury composé de :

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Dedications

To my dear parents, who always supported me, my sister and my brothers thank you for your encouragement and your prayers

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Abstract

The study of the behavior of fiber-reinforced cement mortar in the fresh and hardened state requires a particular knowledge of the influence of the different elements that constitute it. The purpose of our work is to determine and compare the results of 3-point bending and compressive strength tests on cement mortar reinforced with plant fibers of the *Agave Americana* using field experiments and the design of experiments method, and a set of predictive models. A factorial design was therefore used to model the influence of two key parameters related to the cohesion of the mixture and its strength. The parameters considered in this study are: the percentage of fiber added to the mix and the curing period of the specimen. Our study includes two essential phases: A first phase whose objective is the extraction and the treatments of the fibers of the *Agave Americana* as well as the development of the mortar specimen cement/*Agave Americana*. The second phase whose objective is the establishment of mathematical models which allow to predict the workability of cement mortar as well as its resistance at 2,7 and 28 days while highlighting the correlations and the interactions which exist between the various components through the software Design Expert.

Keywords: *Agave Americana* fibers, extraction, mortar cement, Composite materials, modeling, experimental design, Design Expert, eco-friendly composite.

- **ملخص**

تتطلب دراسة سلوك الاسمنت المدعم بالألياف عبر مختلف مراحل جفافه معرفة خاصة بتأثير العناصر المختلفة التي تتكون منها .الغرض من عملنا هو تحديد نتائج اختبارات الانحناء بثلاث نقاط وقوة الانضغاط ومقارنتها على الخرسانة المسلحة بالألياف النباتية الصبار الامريكي باستخدام التجارب الميدانية وتصميم طريقة التجارب ومجموعة من النماذج التنبئية.لذلك تم استخدام تصميم عاملي لنمذجة تأثير معلمتين رئيسيتين تتعلقان بتماسك الخليط وقوته. المعلمات التي تم أخذها في الاعتبار في هذه الدراسة هي: النسبة المئوية للألياف المضافة إلى الخليط وفترة تجفيف العينة.تتضمن دراستنا مرحلتين أساسيتين :مرحلة أولى هدفها استخراج ومعالجة ألياف الصبار الامريكي بالإضافة إلى تطوير عينة الملاط الإسمنت/ الصبار الامريكي. المرحلة الثانية التي تهدف إلى إنشاء نماذج رياضية تسمح بالتنبؤ بإمكانية تشغيل الاسمنت بالإضافة إلى مقاومتها في2، 7 و28 يومًا مع إبراز الارتباطات والتفاعلات الموجودة بين المكونات المختلفة بفضل برنامج design expert

الكلمات المفتاحية: المواد المركبة ، ألياف الصبار الامريكي ، الإسمنت ، الاستخلاص ، النمذجة ، التصميم التجريبي، مركب صديق للبيئة.

• Résumé

L'étude du comportement du ciment fibrée à l'état frais et durci nécessite une connaissance particulière de l'influence des différents éléments qui le constituent. Notre travail a pour but de déterminer et comparer les résultats des essais de résistance à la flexion 3 points et à la compression sur le mortier de ciment chargé par des fibres végétales d'*Agave Americana* à l'aide d'expérimentations et par la méthodologie des plans d'expériences par un ensemble de modèles prédictifs. Un plan factoriel a été donc utilisé pour modéliser l'influence de deux paramètres clés liés à la cohésion du mélange et à sa résistance. Les paramètres considérés dans cette étude sont : le pourcentage en fibres ajoutée au mélange et de la période de séchage de l'éprouvette. Notre étude comporte deux phases essentielles : Une première phase dont l'objectif est l'extraction et les traitements des fibres de *l'Agave Americana* ainsi que l'élaboration des éprouvettes constituées de mortier ciment/*Agave Americana*. La deuxième phase a pour objectif l'établissement de modèles mathématiques qui permettent de prédire la maniabilité des composites élaborés ainsi que leur résistance à 2, 7 and 28 jours tout en mettant en évidence les corrélations et les interactions qui existent entre les différents constituants grâce au logiciel Design Expert.

Mots Clés : Extraction, fibres d'*Agave Americana,* mortier de ciment, matériaux composites, modélisation, plans d'expériences, Design Expert, composite ecologique.

Summary

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General Introduction

• Introduction

Composite materials are used in many fields such as the automotive industry, aerospace or medicine. One of the most successful technical applications in civil engineering is the reinforcement of concrete with steel. Actually, reinforced concrete is a composite material made of a cement matrix added to reinforcement by steel bars. However, the study of such materials must increasingly, because of the standards in force, take into account the environmental aspect in their development.

The composites plant fibers / cement matrix have been much thought in recent years because the plant fibers have many advantages compared to asbestos fibers: low cost, properties of elaboration healthier for the production of composites of different forms, renewal and recycling, while the use of asbestos has been challenged by the legislation on health.

Reinforcing cements with cellulosic fibers results in a composite material that can replace asbestos cement in many applications such as roofing sheets, piping, wall cladding and other applications. The choice of the reinforcement remains very important. It must have good intrinsic strength properties (modulus of elasticity, geometry, etc.), must be compatible with the mortar matrix with which it will be associated and must also have an acceptable cost.

In Algeria *Alfa*, *Disc*, *Cork* and *Agave Americana* are abundant, their harvests and their industrialization are a considerable source of income for entire populations. *Agave Americana* fiber is chosen for reasons of availability and economy, come from a renewable source and can be integrated in a rational way in the field of construction.

By this study, we involve ourselves in this field of research, in order to initiate and contribute to the understanding of eco-sourced composite materials.

Problematic

The main objective of this research is the study and characterization of eco-sourced composites based on natural *Agave Americana* fibers and a cement matrix. We are particularly interested in the realization and the characterization of some specimens for possible bending tests. Thereafter to see the influence of the various constituents of a fibered cement on its behavior and to put a set of simple mathematical models with predictive character of the essential mechanical properties of the cement to know the workability and the mechanical resistance using the Design Expert software.

In order to better understand the usefulness of these models, we have tried to present them graphically to identify major trends and predict the best direction for future studies.

Articulation of this research

After a general introduction, this research is composed of four chapters.

- The first chapter was devoted to a bibliographical research on the composite materials, their classification, advantages, as well as their fields of application.
- The second chapter was reserved for the state of the art on the different types of natural fibers, their morphologies, their chemical compositions as well as their characteristics and mechanical properties.
- The third chapter, which represents the practical part of this work, was dedicated to the elaboration of eco-composites with cementitious matrix and *Agave Americana* fiber reinforcement.
- The fourth chapter was reserved to a study the mathematical modeling and characterization of mechanical behavior of elaborated eco-composites.

The various results obtained are taken up finally in the conclusion which constitutes the closing of this work.

Chapter 1
Overview on composite materials

Chapter 1
Overview on composite materials

1.1 Introduction

The first uses of composites date back to the 1500s B.C. when early Egyptians and Mesopotamian settlers used a mixture of mud and straw to create strong and durable buildings. Straw continued to provide reinforcement to ancient composite products including pottery and boats. Later, in 1200 AD, the Mongols invented the first composite bow. Using a Combination of wood, bone, and "animal glue," bows were pressed and wrapped with birch bark. These bows were extremely powerful and extremely accurate **[1].** Today the growth of composites materials with metallic, elastomer, polymer or even ceramic matrices offer manufacturers and designers new possibilities of combining function, form and materials for the production of increasingly efficient systems. New processes of production, industrialization and structural design, which will extend technical possibilities and better, meet sometimes contradictory needs (function, strength, stiffness...) which the classic built materials does not provide easily. All thaws the competition between using the composite materials and classic ones is still hard **[2].**

1.2 Definition of the composite materials

In the general sense the word composite means a macroscopic combination of two or more distinct materials having a finite interface between them **[3];** with different physical and chemical properties. When they are combined they create a material which is specialized to do a certain job for instance to become stronger or more resistant then the components taken separately **[4].** A composite material also is defined as any alloy or raw material comprising reinforcement in the intimate association of at least two components: the reinforcement and the matrix, which must be compatible with each other and become united, which introduces the notion of a binding agent, the interface. Unlike conventional raw materials, the mechanical characteristics of which are known in advance, those of composites are only really known after manufacture, because the material and the product are produced at the same time. Currently, compounds with an organic matrix represent more than 99% of composite materials; however, there are also composites with an inorganic matrix (metallic or ceramic), the diffusion of which is still marginal **[5].**

1.3 Classification of composite materials

1.3.1 High-performance composites

Composites, usually used in aviation and aerospace, transportation, military defense, and other industries, are made from two or more constituent materials with significantly different physical or

chemical properties. High-performance composites (HPC) refer to composites reinforced with high-performance fibers, including carbon, glass, aramid, ultrahigh-molecular-weight polyethylene, ceramic, quartz, boron, and new fiber such as poly(p-phenylene benzobisthiazole) fibers. These fibers all have exceptional performance properties, such as high modulus, high tenacity, and high thermal resistance. Matrixes used in HPC are mainly polymers (epoxy resin, Bakelite resin, unsaturated polyester resin, etc.), metals (aluminum, magnesium, etc.), alloys (aluminum alloy, magnesium alloy, etc.), and ceramics (aluminum oxide, zirconia, silicon nitride, silicon carbide, etc.) **[6]**.

1.3.2 Large diffusion composites

"Large diffusion LD" composites are characterized by weak mechanical properties but cheaper to produce in large series compared to HP composites; they're used in the manufacture of parts of all kinds **[7].** Large diffusion composites represent 95% of the composites used. They are generally plastics reinforced by short fibers, the rate of reinforcement being around 30%. In more than 90% of cases, the main basic constituents are polyester resins, 95% of which are thermosets with glass fibers in 99% of the reinforcements used **[8]**.

1.4 Advantages and disadvantages of composite materials [9]

Composite materials present many advantages but also some disadvantages as displayed in the Table 1.1.

Table 1.1 Advantages and disadvantages of composite materials

Advantages of composite materials	Disadvantages of composite materials
Light weight and Excellent strength to weight ratio	Expensive material
Resistant to corrosion.	Specialized manufacturing process required
Ideal for external Shell structures	High-quality mold needed.
Wide range of sheet sizes, range of thickness and colors as pigments can be	Cannot be easily repaired as structure loses integrity
Better tensile strength than steel alloys	Cannot be easily recycled
Can be engineered to be anisotropic (fabric orientated in different directions	Requires appropriate finishes to seal surface fibers
Less expensive than natural timbers	Contains urea-formaldehyde which may cause eye and lung irritation when cutting
Isotropic (no grain), so not tendency to split	-

1.5 Fields of use of composite materials

Today composite materials are used in a wide range of industries including (**Fig.1.1**):

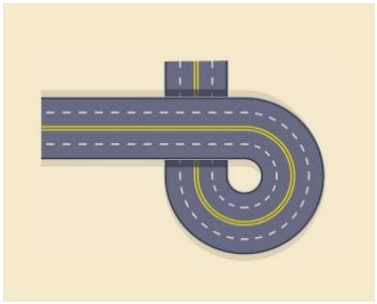
- Aerospace.
- Architecture
- Automotive
- Energy
- Infrastructure
- Marine
- Pipe & Tank
- Sports & Recreation
- Transportation

1.Aerospace

2. Architecture

3. Automotive

4. Energy



5. Infrastructure

6. Marine

7. Pipe and Tank

8. Sports and Recreation

9. Transportation

Fig. 1.1 Fields of use of composite materials

1.6 Basic constituents of composite materials

The basic constituents of composite materials are the reinforcement, the sizing and the matrix, which in general is called the resin system during processing and the matrix in the consolidated phase. The fibers carry the majority of the load and provide most of the stiffness of the material **[10].**

1.6.1 The matrix

The matrix is one of the two main basic constituents of composite materials. The main role of the matrix is to:

- Hold the reinforcements in place.
- To ensure their cohesion and protection.
- It also allows the transmission of mechanical forces to the reinforcements.
- It is generally homogeneous and isotropic.

There are ceramic matrices, metallic matrices, mineral matrices and organic matrices. Currently, thermosetting resins (TS) are mainly used in combination with long fibers, but the use of thermoplastic polymers (TP) reinforced with short fibers is strongly developing. The main thermosetting resins are the widely used polyesters, epoxy resins (or epoxides) which are the standard resin for HP composites and phenolic resins. The main thermoplastic resins used in composites are polypropylene (PP), polyamides (PA) **[11].**

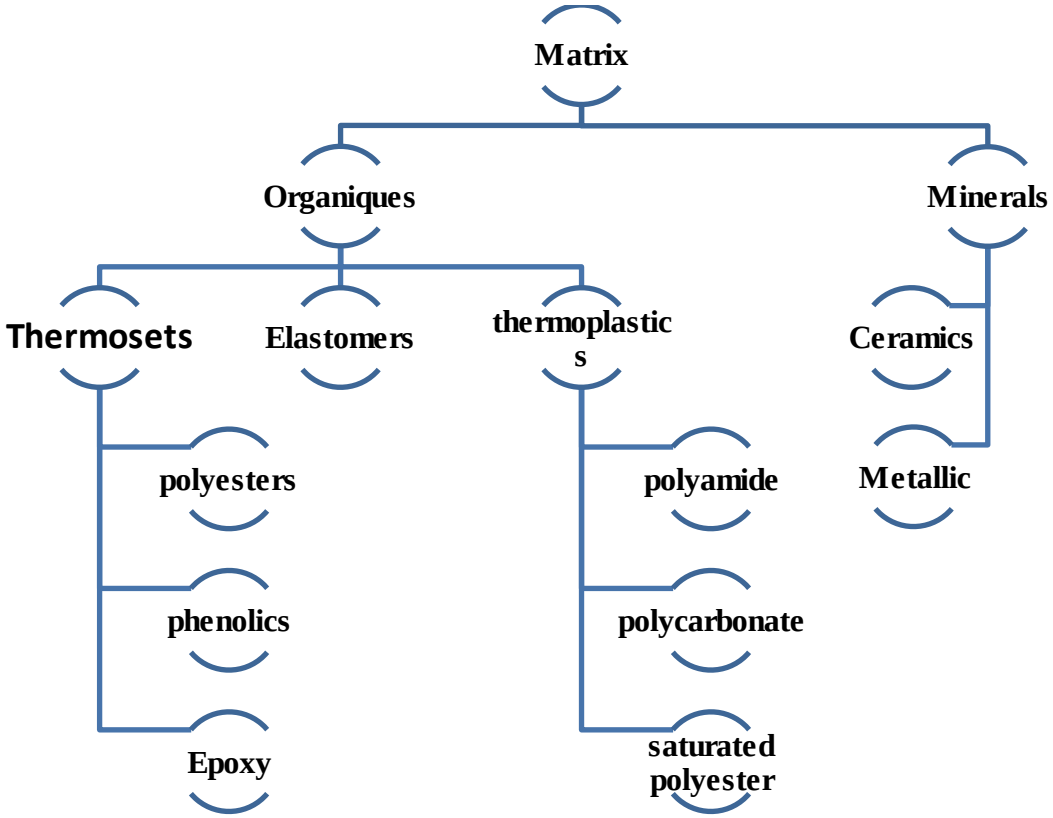


Fig. 1.2 Different matrixes families

1.6.1.1 Thermoset Matrixes

Thermosets, which are infusible and insoluble solids that form a three-dimensional network from molecular segments linked together by a high density of covalent bonds. Thermosets are irreversibly shaped by chemical reactions between the resin and the hardener, which ensures the creation of bridge bonds between the different molecular chains of the polymer. Given their low viscosity before curing, thermosets are widely used as a matrix in composite materials. We can mention unsaturated polyesters (vinyl ester, allyl derivatives, condensed polyesters, etc.), epoxy resins, condensation resins (phenolics, aminoplasts, furanics, etc.)**[12].**

Table 1.2 Mechanical characteristics of common thermosetting resins.

Thermoset Matrix	Density ($Kg.m^3$)	Longitudinal modulus of elasticity (MPa)	Shear modulus (MPa)	Coefficient of Poisson	Stress Of rupture (traction) MPa	Elongation at rupture %
	ρ	E	**G**	**υ**	**σ_r**	**A%**
Epoxides	1200	4500	1600	0.4	130	2
Phenolics	1300	3000	1100	0.4	70	2.5
Polyesters	1200	4000	1400	0.4	80	2.5
Vinylesters	1150	3300	_	_	75	4
Polycarbonates	1200	2400	_	0.35	60	_
Silicones	1100	2200	_	0.5	35	_
Urethanes	1100	700 à 7000	_	_	30	100
Polyamides	1400	4000 à 19000	1100	0.35	70	1

1.6.1.2 Thermoplastic Matrixes

Thermoplastics are derivatives of linear or slightly branched monomers that form the basic unit of a chain where they are repeated many times. Under the action of heat, the thermoplastic softens leading to the appearance of long-range motion in the molecular chains. Thermoplastics can then be solidified by cooling into the desired shape. This process of softening by thermal excitation and hardening by cooling can be repeated indefinitely because, unlike thermosetting resins, thermoplastics are virtually unaffected by chemical changes. Rather, it is a change in the intermolecular arrangement. This ability gives thermoplastic wastes very interesting recycling properties **[12].**

The main thermoplastic resins used in the manufacture of the manufacture of composites are:

- polyamides (PA)
- polyethylene terephthalates (PET) et butylenic (PBT)
- polyphenylene oxide (PPO or PPE)
- polyoxymethylene (POM)

- polypropylene (PP) is a semi-technical polymer, inexpensive, fairly stable in temperature, but combustible

Other Thermoplastics Matrix are beginning to be used for their thermo-stability properties (thermal resistance above 200°C) and good mechanical resistance:

- polyamide-imide (PAI)
- polyether imide (PEI)
- polyether sulfone (PES)
- polyether-ether-ketone (PEEK)

Table 1.3 Mechanical characteristics of common thermoplastic resins **[13]**

Thermoplastic Matrix	Density ($Kg.m^3$)	Longitudinal modulus of elasticity (MPa)	Shear modulus (MPa)	Coefficient of Poisson	Stress Of rupture (traction) (MPa)	Elongation At rupture %
	ρ	**E**	**G**	**υ**	**σ_r**	**A%**
PP	900	1200	_	0.4	30	20 to 400
PPS	1300	4000	_	_	65	100
PA	1100	2000	_	0.35	70	200
PES	1350	3000	_	_	85	60
PEI	1250	3500	_	_	105	60
PEEK	1300	4000	_	_	90	50

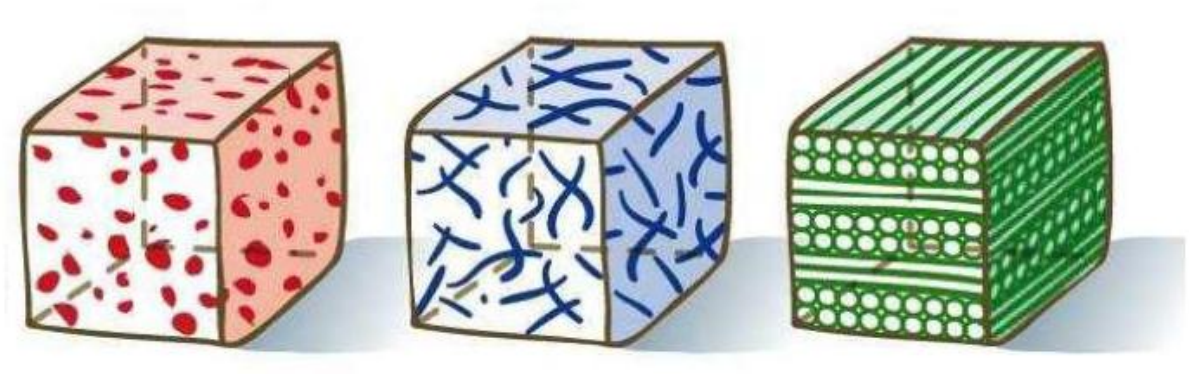
Table 1.4 Comparison of thermoplastic resins and thermosets **[13]**

	Thermoplastic	Thermoset
Basic state	Solid ready to use	Viscous liquid
Storage	Unlimited	Reduced (precautions to take)
Wettability of reinforcements	Difficult	Easy
Molding	Heating+cooling	Continuous heating
Cycle	Short	Long (× 2) (polymerization)
Term of impact	Fairly good	Limited
Thermal term	Reduced (<130°)	Better (>150°)
Scrap and waste	Recyclable	Lost or used as
Working conditions	Cleanliness	VOC emanation

1.6.2 The reinforcement

the reinforcement is generally composed of a material harder than the resin, its main role is to ensure a high resistance to the material, especially to traction, and which is generally in the form of fibers (diameters from 5 to 20 μm) (**Fig. 1.3**)

- unidirectional long fibers (carbon, glass)
- long woven fibers (3D and 2D fabrics)
- randomly distributed short fibers without preferred directions (mat)



1. Particle composite 2. Short fiber composite 3. Long fiber composite

Fig. 1.3 Geometric structures of long fiber, short fiber and particulate composite Reinforcements.

The fibers are of organic type (polyamide fibers, polyester, polypropylene ...) vegetable and animal fibers. And inorganic (glass fibers, carbon, silica ....) or natural (cellulose). In general, they have excellent mechanical characteristics **[11].** Depending on the mechanical properties required, there are several reinforcement architectures for composites, unidimensional where the fibers are oriented in the same direction, bidimensional as in the case of woven or non-woven surfaces and three-dimensional with fibers oriented in several directions in space **[14].** The main reinforcing materials used for plastic composites are: glass, carbon and aramid fibers glass, carbon and aramid fibers (**Fig. 1.4**).

(1) (2) (3)

Fig.1.4 Most commonly used fibers: (1) glass fibers, (2) carbon fibers, (3) aramid fibers

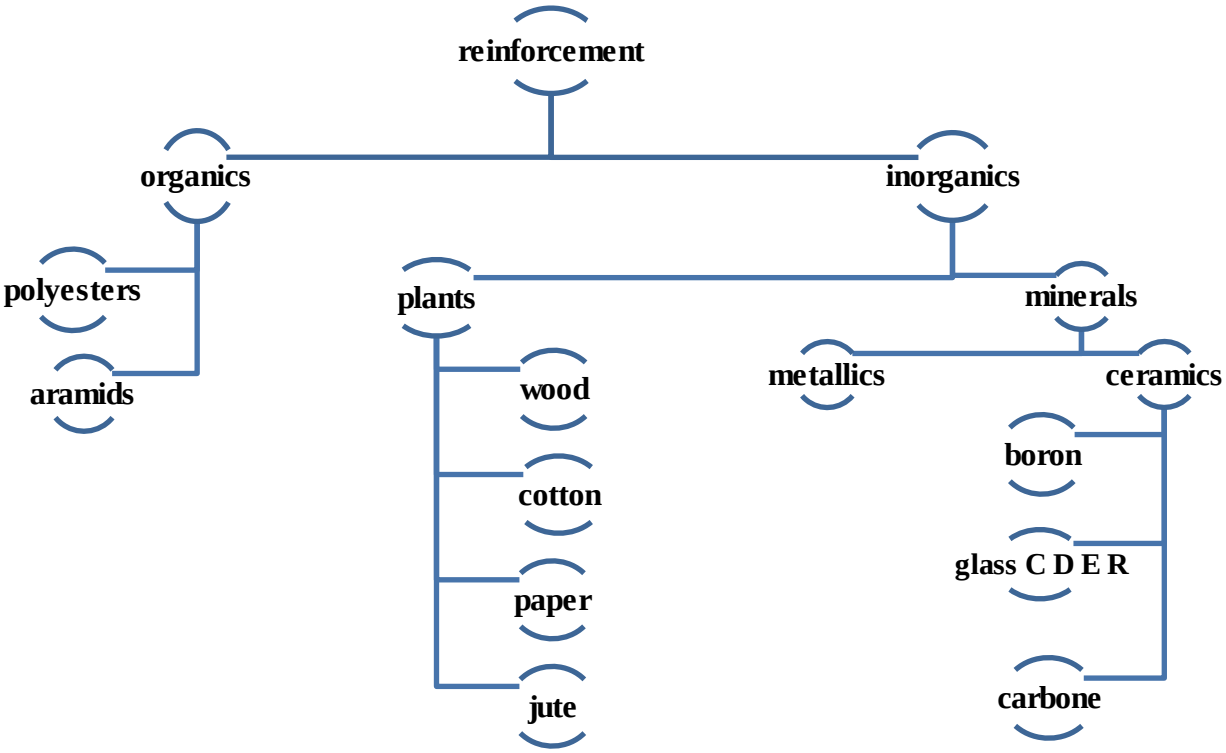


Fig. 1.5 Different types of reinforcement for composites

1.6.2.1 Glass fibers

Glass fibers (**Fig. 1.6**) In its massive form, glass is characterized by a great brittleness and a strong sensitivity to cracking. On the contrary, glass used in the form of small diameter fibers has very good mechanical characteristics. These glass fibers are obtained by extrusion of glass through a die with holes of 1 to 2 mm in diameter. The glass fibers are then drawn to diameters of 5 to 15 µm at the end of the process they are coated and wound (roving) (**Fig. 1.7**). The glass fibers are the essential reinforcement of large diffusion composites. They are obtained from sand (silica) and additives (alumina, carbonate of lime, magnesia, boron oxide). **[15].**

Fig 1.6 Glass fibers

They have an excellent performance-price ratio which places them in the first rank of reinforcements used in construction. We distinguish three types of fibers:

E: for high diffusion composites and common applications

R: for high performance composites

D: for the manufacture of printed circuits (dielectric properties).

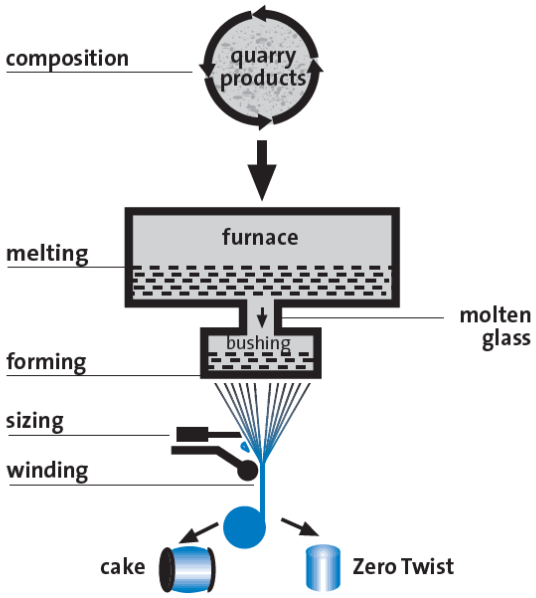


Fig. 1.7 Glass fiber manufacturing process

1.6.2.2 Carbone fibers

They are made from basic polymer fibers called precursor which is itself in the form of oriented fibers previously woven, and carbonized under tension in several steps, oxidation (100 to 200 C°), then pyrolysis (1500-2500 C°). Depending on the temperature and time of firing, the fibers have a "high strength" (HS) or a "high modulus" (HM). Generally, acrylic fibers are used as a precursor, made from poly acrylonitrile (PAN) **[11]** (**Fig. 1.8**). This is the most common fiber used in high performance applications. The fibers are produced from chemical precursors such as poly acrylonitrile ([CH2-CH-CH] n). They are used in particular to reinforce certain composite materials. Carbon fibers have certain advantages:

- They are rigid
- They resist well to traction, as well as to compression.
- They are nearly 70% lighter than steel.
- These characteristics are of great interest to the industry

Fig 1.8 Acrylic fibers made from poly acrylonitrile (PAN)

1.6.2.3 Aramid fibers (Kevlar)

Poly-Para-phenylene terephthalamide, known and marketed since the 1970s under the name of Kevlar, is made up of benzene rings separated by amide groups. The aramid fiber Kevlar has very interesting properties for the industry. It is characterized by an excellent resistance to traction, shock and wear. This type of fiber is mainly used in the manufacture of bulletproof vests and in the aeronautical industry. (**Fig. 1.9**) **[11].**

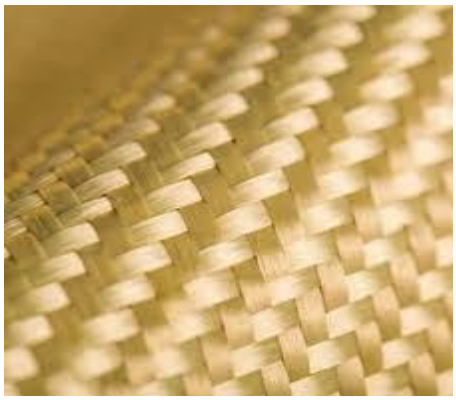


Fig 1.9 Aramid fibers (Kevlar)

1.6.2.4 Reinforcement architecture

Most reinforcements work well in tension, but offer less good performance in compression and shear. It is therefore imperative to play on the texture and geometry of the reinforcements to create an improved architecture. The reinforcements are assembled in order to facilitate their shaping. We can distinguish several categories of reinforcements:

- Mats.
- UD fabrics (unidirectional).
- Bidirectional fabrics.
- Multidirectional fabrics.

Mats

The mats (**Fig.1.10.1**) are layers of continuous or discontinuous yarns, randomly oriented and held together by a soluble binder in a plane. The absence of preferential orientation of the fibers leads to an isotropy of the mechanical properties of the mat in its plane. **[11, 15]**.

Unidirectional fabrics

In a UD mat (**Fig. 1.10.2**), the fibers are assembled parallel to each other using a very light weft. **[11]** Very high imbalance rate.

Fabrics

The fabric intended for the reinforcement of composites is a flexible surface (**Fig.1.10.3**), constituted by a regular assembly of woven or twisted yarns, obtained by interweaving the strands according to two directions perpendicular directions, warp and weft. They can have different weaves **[11]**. We distinguish mainly: canvas or taffeta, twill and satin (**Fig.1.11**).

Taffeta (canvas or cloth)

Each warp thread passes over and then under each weft thread, and vice versa (**Fig.1.11.1**). The fabric has a good flatness and a relative rigidity, but is not very deformable for the implementation. The numerous successive interweavings generate a significant fogging and reduce the mechanical properties **[16].**

Twill

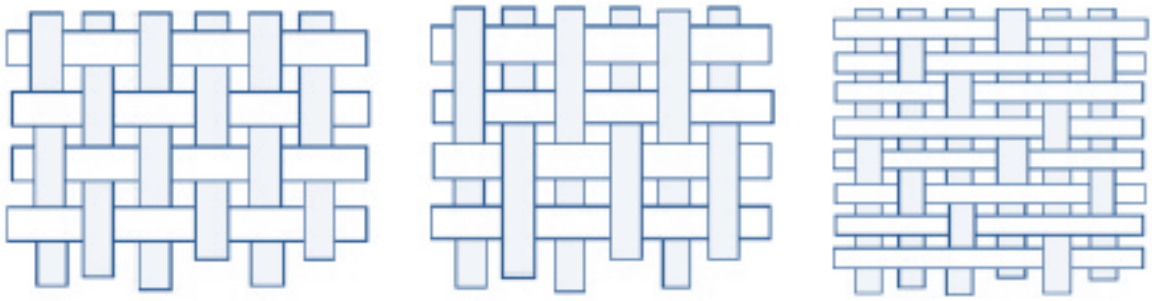
Each warp thread floats over several (n) weft threads and each weft thread floats over (m) warp threads (**Fig.1.11.2**). A weave that is more flexible than taffeta having a good density of threads **[16]**.

Satin

Each warp thread floats over several (n-1) weft threads and vice versa (**Fig.1.11.3**). These fabrics have different aspects on each side. These fabrics are quite flexible and suitable for shaping parts with complex surfaces. This type of fabric has a high specific weight **[16].**

(1) (2) (3)

Fig 1.10 Different architectures of composite reinforcements: 1) glass mats, 2) unidirectional (UD) fabric, 3) bidirectional fabric



(1) (2) (3)

Fig 1.11 Different forms of two-way fabrics for surface reinforcement: 1) Taffeta, 2) Twill, 3) Satin

1.7 Structural composite materials

The structures of composite materials can be classified into three types: monolayers, laminates and sandwiches.

1.7.1 Monolayer

Monolayers (**Fig.1.12**) represent the basic element of the composite structure. The different types of monolayers are characterized by the form of the reinforcement: long fiber (unidirectional UD, randomly distributed), woven fibers, short fibers **[17]**.

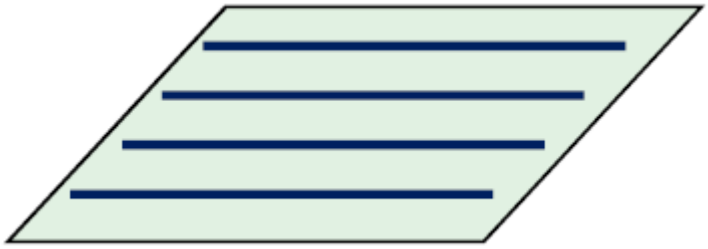


Fig 1.12 Monolayer UD (Unidirectional fold)

1.7.2 Laminates

Laminated composite (**Fig.1.13**) structures are made up of successive layers of resin-impregnated reinforcements. The layers are also called plies. Laminated structures made from composite materials are made up of stacks of unidirectional or bidirectional plies. These plies are made of long fiber reinforcements bonded with resin. The role of the reinforcement is to ensure the function of mechanical resistance **[17]**.

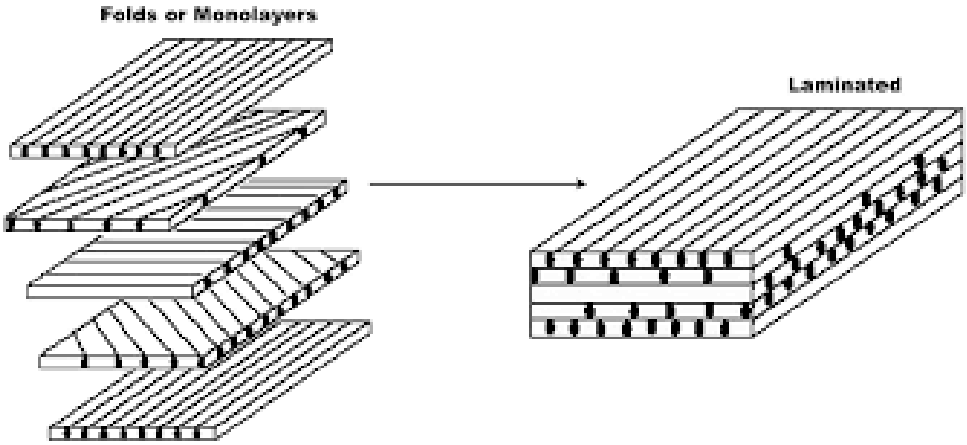


Fig 1.13 Laminate composed of several monolayers with different orientations

1.7.3 Sandwiches

These materials are composed of two very rigid and thin skins enveloping a very thick and weak core. The whole forms a very light structure. The assembly of the skins with the core is generally done by gluing, by welding or by polymerization in molds in the case of plastic composites. The cores can be solid (wood, foam, etc.) or hollow (light metal alloys, paper, etc.). The skins are generally laminates or sheets of light alloys (**Fig.1.14**). The most commonly used cores are honeycomb corrugated or foam **[18].** The most commonly used materials are:

- For solid cores

- balsa or cellular wood
- various cellular foams
- resins filled with hollow glass microspheres, called syntactic foams

- for hollow cores, mainly honeycomb and profiles

- light metal alloys
- Kraft paper (resin coated or not)
- polyamide paper, such as Nomex paper.

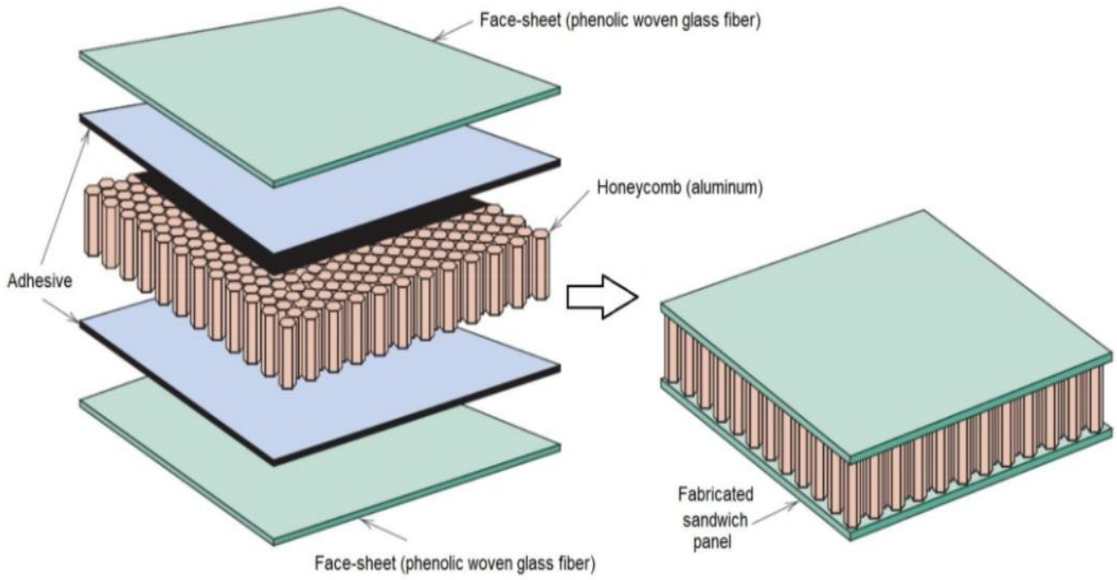


Fig 1.14 Sandwich Composite

1.8 Implementation of composite materials

The elaboration of a composite material generally takes place in three essential operations which are:

- Impregnation of the reinforcement by the resin
- Shaping to the geometry of the part in molds
- Curing of the system

1.8.1 Contact molding

It is a manual process for the realization of structures from thermosetting resins. Its principle consists in manually impregnating the reinforcements placed in a mold at room temperature and without pressure. After curing of the resin, the part is demolded. Contact molding (**Fig.1.15**) is an inexpensive process used to produce parts of any shape but at a very low rate **[11]**.

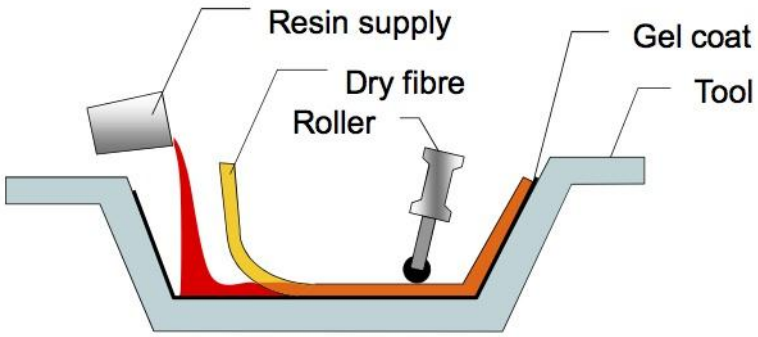


Fig 1.15 Simplified diagram of the contact molding process

1.8.2 Simultaneous spray molding

The molding is made by simultaneous projection (**Fig.1.16**) of the cut fibers and catalyzed resin on a mold. The equipment to project is constituted by a machine to cut the laminate and a gun projecting the resin and the cut fibers, the whole functioning by compressed air. The resin impregnated fiber layer is then compacted and bubbles are removed with a grooved roller **[19]**.

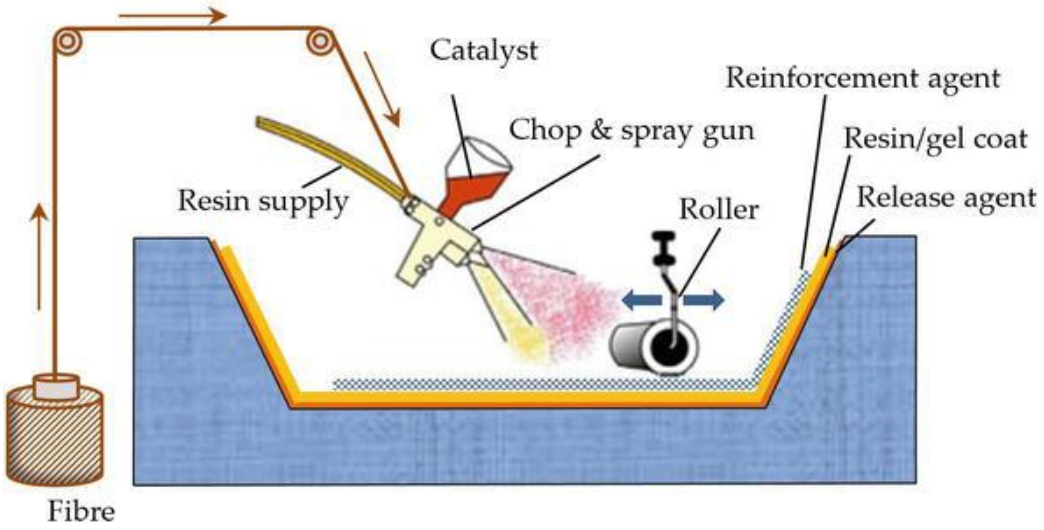


Fig 1.16 Simplified diagram of the Simultaneous Spray Molding process

1.8.3 Injection molding

The injection molding method (**Fig.1.17**) is the most common method of processing reinforced thermoplastics (other methods being extrusion, blow molding, thermoforming, etc.). Injection molding is performed on conventional presses used for the injection of thermoplastic resins. Pellets containing resin and reinforcement (short fibers, beads, etc.) or pre-impregnated mats are extruded by an Archimedean screw. The matrix is fluidized by heating and injected under high pressure into a heated mold, where polymerization takes place. The type of materials obtained is more generally called "reinforced plastics" than composite materials. Indeed, due to the nature of the reinforcements (short fibers, spheres, etc.), the stress at break and the Young's modulus of the resins are multiplied by a factor of about 2 to 4. This technique is suitable for the production of parts in very large series **[19]**.

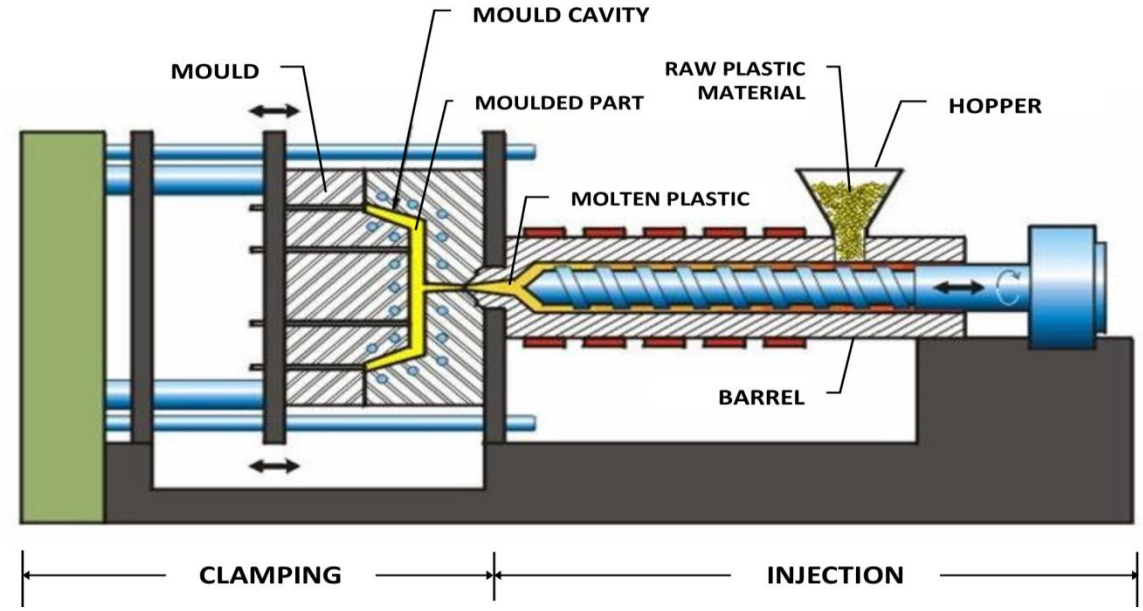


Fig 1.17 Simplified diagram of the Injection molding process.

1.8.4 Compression molding

The molding of composite materials with the technique of low pressure compression is implemented with the help of a compression press, a mold and a rigid counter mold. First, the mat (reinforcement) is placed on the lower mold. The resin is then poured onto the mat. The press is then closed and pressurized. The mold and counter mold press the resin and mat together, forcing it to form a composite material and spread evenly, discharging excess material through the holes provided. When the material is hard, the molds can be separated for demolding. In some cases, a deburring step will be necessary **[20]**.

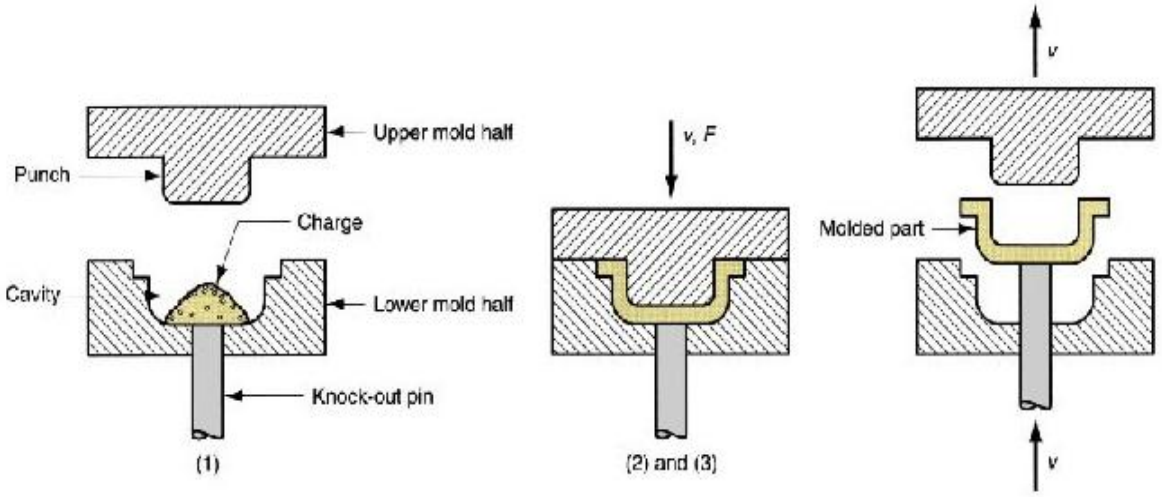


Fig 1.18 Simplified diagram of the compression molding process

1.8.5 Filament winding

The reinforcement (continuous wire, ribbon, etc.) impregnated with catalyzed resin is wound with a slight tension on a rotating cylindrical or rotation mandrel (**Fig.1.19**) .This type of molding is well suited to cylindrical and spherical surfaces, and allows for advanced part design. The resulting laminates can have high proportions of reinforcement (up to 80% by volume), thus enabling high mechanical characteristics to be obtained. The investment in equipment is very important. Depending on the relative movements of the mandrel and the reinforcement supply system, various types of windings (and consequently of stratifications) are obtained. We distinguish between: circumferential winding, helical winding, polar winding **[18]**.

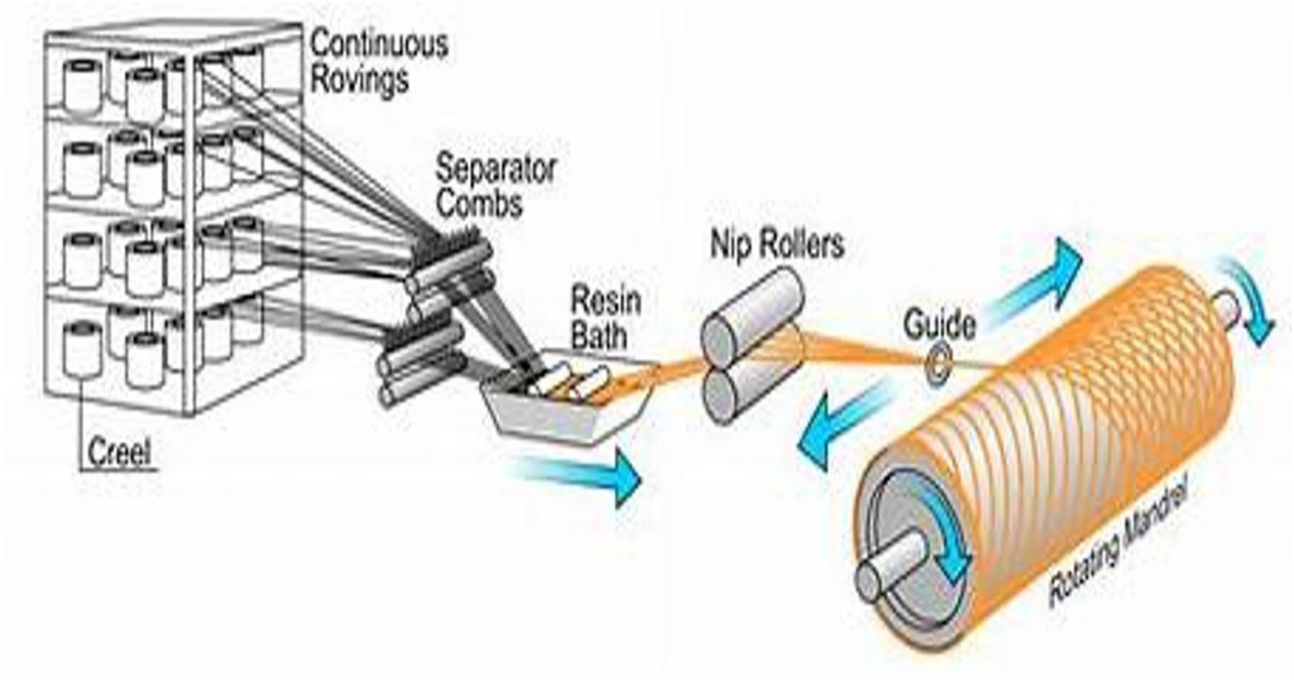


Fig 1.19 Simplified diagram of the filament winding process.

1.8.6 Pultrusion molding

The process is intended for the continuous production of profiles with constant cross-sections. Continuous reinforcements, various rovings, mats and fabrics in strips of appropriate widths, pulled by a traction bench located at the end of the production line, are successively predisposed in a precise way, impregnated with resin and shaped into the desired form by passing through a heated die in which the resin is hardened (**Fig.1.20**) **[11]**.

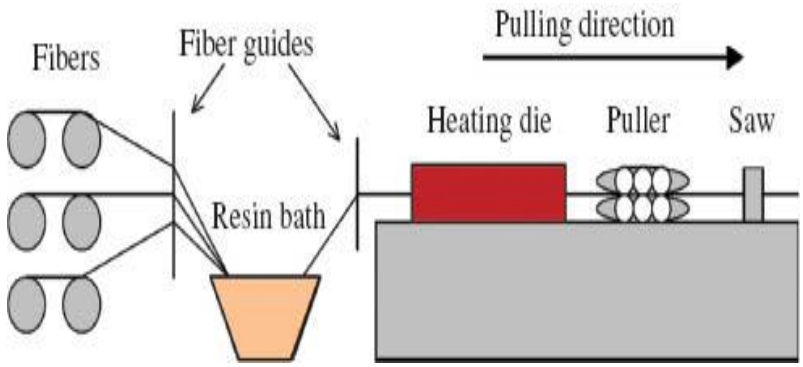


Fig 1.20 Simplified diagram of the pultrusion molding process.

Bibliographical references

[1] Rahul Reddy Nagavally. Composite materials - history, types, fabrication techniques, advantages, and applications. Affiliation: Kakatiya Institute of Technology & Science, Warangal, Telangana, India,2018.

[2] Pascal Celle, Marc Marzano, Les matériaux composites, Meudon : ANFA (Association nationale pour la formation automobile), 2014.

[3] Karbhari. Durability of Composites for Civil Structural Applications. The Institute of Materials, Minerals & Mining. 2007

[4] https://www.toppr.com/guides/geography/mineral-and-energy-resources/composite-materials-definition-and-its-types/ accessed on February 2022

[5] Louis Berreur, Bertrand de Maillard, Stanislas Nösperger, Etude prospective stratégique sur les matériaux composites, Rapport final, Paris: la Direction Générale de l'Industrie et des Technologies, 2001-2002.

[6] Yiki Yang, Jianyong Yu, Helan Xu, Baozhong Sun (editors). Porous Lightweight Composites Reinforced with Fiberous Structures .2017

[7] Bahlouli Nadia, Cours Matériaux Composites, DESS Mécanique et Stratégie industrielle. 2004. avancée

[8] https://www.recy.net/outils/plastiques/plast04_02.php accessed on February 2022

[9] JONES 97. Composite materials. Get revising part of the student room Articale. 2016.

[10] Ogin, Brøndsted, and Zangenberg, Composite materials: constituents, architecture, and generic damage, Editor(s): Ramesh Talreja, Janis Varna, 2016.

[11] Deghboudj Samir. Matériaux Composites. Universite l'Arbi Tebessi de Tebessa , faculte de Science et de la Technologie. 2020-2021.

[12] Herroudj Noura, Étude de L'introduction des fibres d'alfa de tailles différentes dans une matrice polychlorure de vinyle en vue d'améliorer ses propriétés physico- mécanique, Thèse de Magister Université Abderrahmane Mira Bejaïa Faculté de la Technologie, 2010.

[13] https://www.mecastyle.com/calcul-de-structure-materiau-composite accessed on February 2022

[14] Marianne Le Troedec, Caractérisation des interactions physico-chimiques dans un matériau composite à base de phyllosilicates, de chaux et de fibres cellulosiques, Thèse de doctorat, Limoges: Université De Limoges, Ecole Doctorale Sciences-Technologies-Santé, 2009

[15] Abdellah Houideg, Transfert de charge et frottement Fibre/ Matrice d'un Matériau Composite, Université des Sciences et de la Technologie d'Oran, Mémoire de Magister,2012.

[16] https://aeromodelisme.forumactif.org/t113-les-differentes-fibres-utilise-pour-le-composite accessed on February 2022

[17] Tahar Masri, Contribution à la modélisation des phénomènes de rupture des matériaux composites, Université Mohamed Khider, Biskra, Mémoire de Magister 2006.

[18] Jean-Marie Berthelot, Mécanique des Matériaux et Structures Composites, ISMANS Institut Supérieur des Matériaux et Mécanique Avancés, Le Mans ,2010.

[19] Jean.M.Berthelot, Matériaux composites : Comportement mécanique et analyse des structures ,5eme Ed, Technique Et Documentation, Lavoisier, (2012) .

[20] https://www.rotteleur-composites.com/ accessed on February 2022

Chapter 2
Natural fibers

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2.1 Introduction

The importance of natural fibers, which are used for hundreds of years in order to meet human needs such as clothing and sheltering, has considerably reduced through the use of synthetic fibers toward the end of the 1900s. The increasing environmental concerns and depletion of petroleum resources have increased the importance of natural fibers once again and have stimulated researchers and industries to use sustainable fibers instead of conventional synthetic fibers. Besides mechanical and physical properties such as good specific modulus values, low density, considerable toughness properties of natural fibers, low cost, recyclability, nontoxicity, and easy accessibility properties are also attractive aspects of natural fibers and these properties give an opportunity to use natural fiber reinforced composite products in various industries such as automotive, building, and furniture. This chapter gives information about structure and properties of common natural fibers, which are used as fiber reinforcement sources **[1]**.

2.2 Natural fibers

Natural fibers are biological structures composed mainly of cellulose, hemicelluloses and lignin. They also contain extractives, proteins and some inorganic compounds in small proportions. Vegetable fibers find many applications in the textile industry (clothing, furniture). Given their specific mechanical properties and their renewable character, they are beginning to find outlets in other industrial sectors (packaging, automotive). Natural fibers are classified according to their origins in:

2.2.1 Mineral fibers

They are of limited length, except for particular types such as asbestos, which are now known to be toxic and harmful to human health (carcinogenicity). Today it is strictly forbidden to use it in any way.

2.2.2 Animal fibers

These are the most used in the manufacture of textiles by hand spinners, such as sheep's wool and silk. Not all animal fibers have the same properties, and even within the same species, the fiber is not homogeneous. The most famous animal fibers are silk, wool and animal hair (**Fig.2.1**).

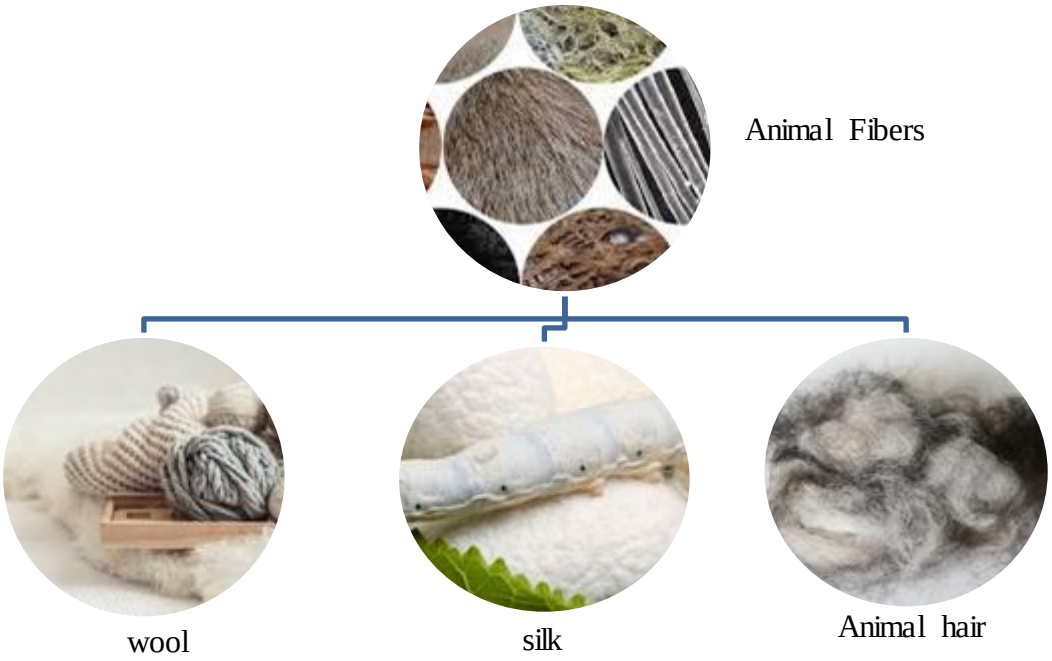


Fig.2.1 Natural fibers of animal origins.

2.2.3 Vegetable fibers

Vegetable fibers or Plant fibers are filamentary materials derived from biomass. They can be extracted from the fruit, stem or leaf of a cultivated plant, shrubs and grasses. They can be spun to make yarns and ropes. They are woven, knitted or braided to make textiles essential to society. The vegetable fiber presents a very important variability according to their origin. Indeed, the dimensions of the vegetable filaments depend on the state of maturity and also on the environmental conditions of growth of the plant. The variability of the diameter can be very important along the same plant fiber **[2]**. Plant fibers are also characterized by the diversity in thickness of their cell wall thickness resulting from the fibrous porosity **[3]**.The morphological shape of the plant fiber may also be an important factor in understanding stress transfer at fiber-matrix interfaces **[4]**.

2.2.4 Introduction to vegetable fibers

There is a wide variety of plant fibers that can be used for reinforcement or as fillers in reinforcement or as fillers for composite materials. They are classified according to their origin and their shape (**Fig. 2.2**).

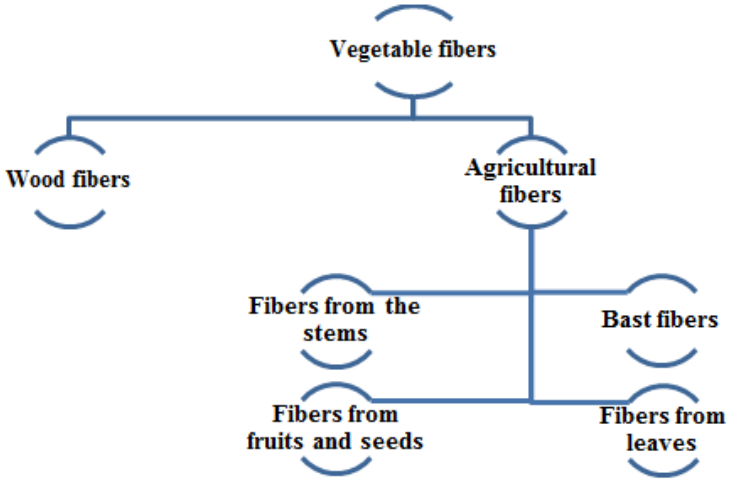


Fig. 2.2 Classification of vegetable fibers

2.2.4.1 Usefulness of the use of fibers

Plant fibers have many advantages in the formulation of materials, apart from their ability to store carbon dioxide and their **[6]**:

- Renewable character due to its annual production
- Low cost
- Biodegradability
- Renewable resource
- Requires little energy to produce
- Good thermal and electrical insulation
- Important specific mechanical properties (strength and rigidity)

Sisal fibers : Sisal is the name of the fiber extracted from the leaves of a plant called Agave Americana consisting of a rosette of large leaves with a triangular section up to 2m long (**Fig.2.3**). Originally from eastern Mexico, this fiber is used for the manufacture of rope, coarse fabrics, and carpets **[5]**.

Fig. 2.3 Agave Americana.

Abaca: Abaca (**Fig.2.4.1**) or banana fiber, derived from the banana plant (**Fig.2.4.2**), is durable and resistant to saltwater. Abaca, the strongest commercially available cellulose fiber, is indigenous to the Philippines and is currently produced there and in Ecuador. It was once the preferred rope fiber for marine applications **[6]**.

(1)

(2)

Fig. 2.4 **A**baca: 1. Abaca fiber, 2. Banana plant

Ramie: Ramie (**Fig.2.5**) belongs to the family Urticaceae (Boehmeria), which includes about 100 species. The popularity of Ramie as a textile fiber has been limited largely by the regions of production and a chemical composition that has required more pretreatment than is required of other commercially important liberal fibers. Ramie fiber reinforced composites have been manufactured using a hybrid method of fusion and injection molding. Different ramie fibers were fabricated by varying the fiber length, fiber content and fiber pretreatment of the fibers. The result showed an increase in fiber length and fiber content also shows an increase in tensile strength, bending and compressive strength in turn. However, they also have negative influences on the impact strength and elongation behavior of the composite **[6]**.

Fig. 2.5 Ramie.

Alfa: Alfa (from Arabic Halfa) (**Fig.2.6**) is a perennial herbaceous plant of the family Graminaceae, native to the arid regions of the western Mediterranean basin. The major foci are on the large Algerian-Moroccan plateaus, and beyond that it extends to western Morocco, southern Portugal, eastern and southern Spain. Alfa is found in the driest areas of the Mediterranean region. In the south its natural limit is determined by the drought. In the north and west it is the increasing humidity of the climate that eliminates the flora **[7]**. Alfa was used to make mats, curtains, carpets, baskets, trays, shoes, string and various ties. At the end of the 19th century and the beginning of the 20th century, Alfa paper appeared a quality paper that would give great economic importance to this plant **[8]**. The Alfa plant is composed of approximately 50 to 54% cellulose, 27 to 32% hemicellulose, 17 to 19% lignin, 0.5% wax and 2% ash. The plant is cylindrical, approximately 1 m long. Its structure is formed by several bundles of filaments, approximately circular, aligned along the length of the plant **[9]**.

Fig. 2.6 Alfa plant

Hemp fibers: Hemp is a variety of cultivated plant. It is an annual plant of the family Cannabis family (**Fig.2.7**). The main producers are France and Italy. Hemp fibers are highly sought after as reinforcement in thermoplastic matrix composites because of its high stiffness and economic interest. They are applied in internal structures, automotive parts and building materials **[5]**.

Fig. 2.7 Hemp fibers

Jute: Jute (**Fig.2.8**) is produced from plants of the genus Corchorus, which includes about 100 species. It is one of the cheapest natural fibers and is currently the Liberian fiber with the highest production volume. Bangladesh, India and China provide the best condition for growing jute **[6]**.

Fig. 2.8 Jute.

2.3 Morphologies of vegetal fibers

2.3.1 Structure of a vegetal fiber

The plant fiber is a composite in itself. The reinforcement is constituted by the layers of cellulosic micro fibrils in part crystalline. The latter is coated with an amorphous polysaccharide matrix (hemicellulose and pectin) which is associated by hydrogen bonding and covalent to the lignin. The plant fiber is composed of several walls parallel to the axis of the fiber and arranged in layers superimposed in the radial direction. These different layers form the middle lamella, the primary wall and the secondary wall. The latter border a lumen of variable diameter depending on the species. The secondary wall is composed of three layers of micro fibrils (S1, S2, and S3) (**Fig.2.9**) **[10]**.

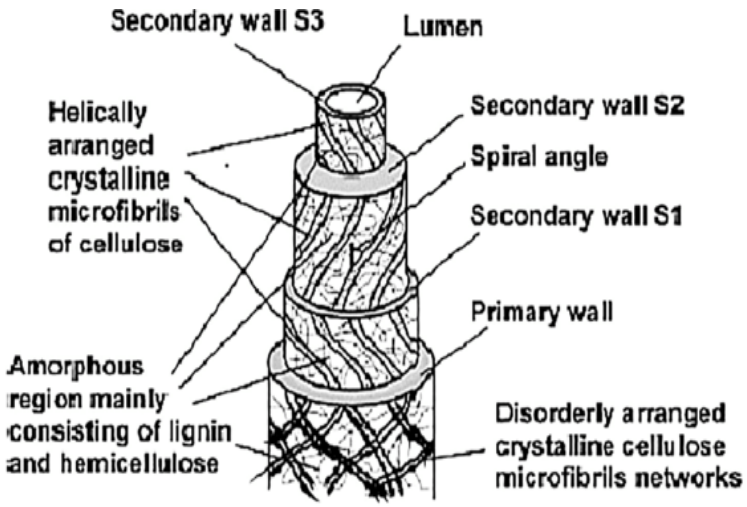


Fig. 2.9 Structure of the cell wall of vegetable fibers

2.3.2 Chemical composition of vegetal fibers

Plant biomass is made up of several macromolecules that are closely linked together within the plant wall. As we have seen previously, the most important compounds are cellulose, hemicellulose, pectins and lignins. These different constituents are arranged in a very complex way. In wood, cellulose is concentrated inside the fiber (**Fig.2.10**). The outer walls of the fiber are composed mainly of lignins and hemicelluloses and the inter-fiber junction lamellae are composed almost entirely of lignin. An additional network of pectins (acidic polysaccharide polymers) increases the complexity of the matrix. The polysaccharide network can also be solidified by a secondary network of HRGP (Hydroxyproline Rich Glyco Proteins) **[11]**.

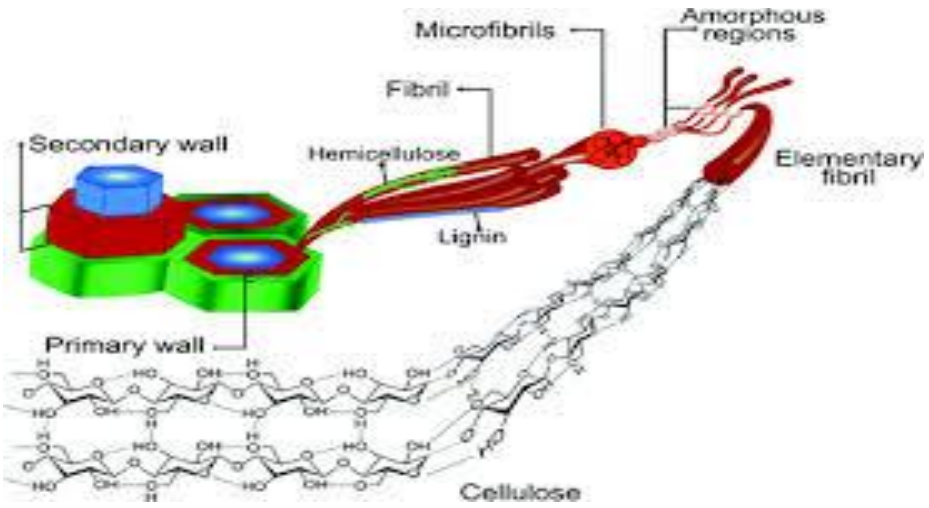


Fig.2.10 Chemical composition of a vegetal fiber.

2.3.2.1 Cellulose

The existence of cellulose as a common material in the cell walls of plants was first discovered by Anselm Payen in 1838 **[12]**. Cellulose represents the most abundant biological molecule on earth. From a chemical point of view, cellulose is a macromolecule consisting of a very long stereo-regular chain of C6H12O6 glucose bonds (**Fig.2.11**). Cellulose has a fibrillar and partially crystalline structure. Cellulose micro fibrils are made up of ordered crystalline zones and totally disordered amorphous zones. In the crystalline zone, the cellulose chains are arranged parallel to each other, linked to each other, bound by intra and intermolecular hydrogen bonds. All the properties of cellulose are closely linked to the high density of hydrogen bonds that develop between the chains. The molecular interactions are strong and ensure the necessary cohesion while preventing the penetration of reagents. Thanks to its cohesion, cellulose is insoluble in most solvents. Cellulose is very hydrophilic in nature .

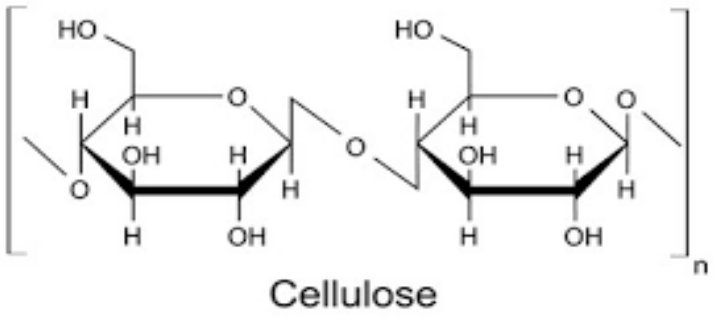


Fig.2.11 Chemical structure of cellulose

2.3.2.2 Hemicelluloses

In most natural fibers, cellulose is mixed with hemicelluloses which are also polysaccharides composed of a combination of 5- and 6-carbon rings (**Fig.2.12**). Hemicellulose forms the supporting matrix for cellulose micro fibrils. Hemicellulose differs from cellulose in three aspects. First, it is composed of neutral sugars: xylose, arabinose, galactose, glucose, mannose, and uronic acids whereas cellulose contains only 1, 4-βD-glucopyranose units. Second, it has a considerable degree of side-group-containing branching that accounts for its non-crystalline nature. Finally, in its natural state, it has a degree of polymerization between 50 and 300, while that of native cellulose is 10-100 times higher. Hemicellulose is very hydrophilic, soluble in alkaline medium, and easily hydrolyzed in acids **[13-14]**.

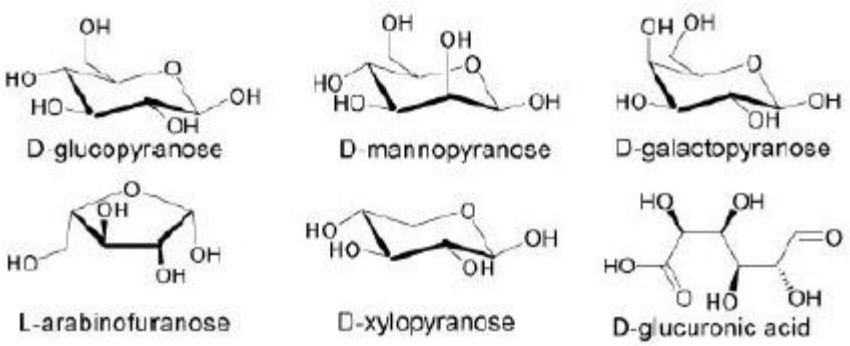


Fig. 2.12 Chemical structure of hemicelluloses

2.3.2.3 Lignin

Lignin is an extremely heterogeneous macromolecule composed of aromatic polymers. It represents, after polysaccharides, the most abundant natural polymer on earth. It participates in the structural rigidity of cell walls and protects plants against the attack of pathogenic organisms. Lignin is a three-dimensional molecule composed of phenyl propane units (**Fig.2.13**). The primary units (monolignols) constituting lignin are coniferyl alcohol, sinapyl alcohol, and p-coumaryl alcohol. During the

lignification process, plant phenoloxidases such as laccases intervene and allow the polymerization of the different elementary units. Once synthesized, lignin associates with the different polysaccharides to form a constitutive matrix of the plant wall **[14-15]**. Lignin is completely amorphous and hydrophobic. It is not hydrolyzed by acids, but soluble in soda when heated, easily oxidized and easily condensed with phenol **[15]**.

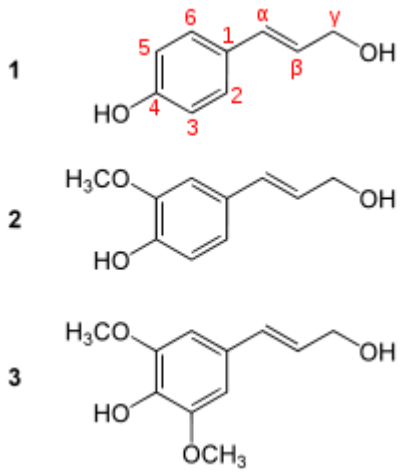


Fig. 2.13 Lignin structure

2.3.2.4 Pectins

Pectins (**Fig 2.14**) play a key role in the architecture of the plant wall. Structurally, pectins are a family of complex polysaccharides that contain a chain of α-D-galacturonic acid units linked together by α bonds (1-4), interrupted by the presence of L-rhamnopyranose units. Pectins also carry non-sugar substituents, mainly methanol, acetic acid, phenolic acid, and sometimes amide groups. The esterification of galacturonic acid residues with methanol or acetic acid is a characteristic that plays a very important role in the physical-chemical properties of pectins, particularly in gel formation **[5]**.

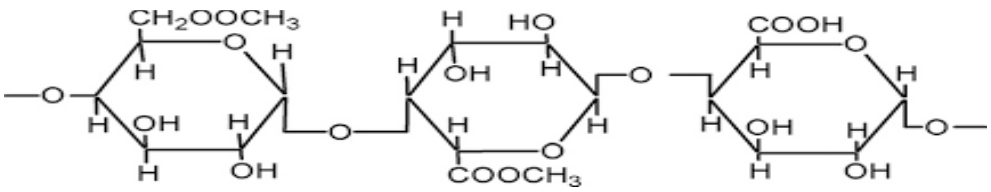


Fig. 2.14 Molecular structure of pectin

2.3.2.5 Waxes

Waxes are constituents of natural fibers that can be extracted with organic solutions. These materials consist of different types of water-insoluble alcohols and several acids such as palmitic acid, oleaginous acid and stearic acid **[16]**.

Table2.1 Chemical composition of some plant fibers **[5]**.

Fibers	Celluloses (Wt %)	Hemicelluloses (Wt %)	Lignins (Wt %)	Pectines (Wt %)
Linen	60-81	14-18.6	2-3	1.8-2.3
Jute	51-72	12-20.4	5-13	0.2
Sisal	43-88	10-12	4-12	0.8-2
Hemp	70-78	17.9-22	3.7-5	0.9
Kenaf	36	21	18	2
Ramie	68.6-76	13.1-15.0	0.6-1	1.9-2
Cotton	82.7-90	2-5.7	0.5-1	5.7
Wood	45-50	23	27	-
Date palm	41-45	6-10	30-40	-

2.4 Physical-mechanical properties of plant fibers

Vegetable fibers are generally good reinforcements for thermoplastic or thermoset matrices because of their relatively high strength and low density. Some characteristics of hemp and flax fibers reach values very close to those of "E" glass fibers (Table 2.1) **[5]**.

Table 2.2 Physical-mechanical properties of plant fibers compared to synthetic reinforcements [5].

Fibers	Density (g/cm3)	Resistance (MPa)	Young's modulus (GPa)	Elongation (%)
Flax	1.4	88-1500	60-80	1.2-1.6
Hemp	1.48	550-900	70	1.6
Jute	1.46	400-800	10-30	1.8
Ramie	1.5	500	44	2
Coir	1.25	220	6	15-25
Sisal	1.33	600-700	38	2-3
Abaca	1.5	980	-	-
Cotton	1.51	400	12	3-10
Kenaf (bast)	1.2	295	-	2.7-6.9
Banana	1.35	355	33.8	53
Coco	1.2	175	4.0-6.0	30.0
Galss – E	2.5	2000-3500	70.0	2.5
Glass – S	2.5	4570	86.0	2.8
Aramid	1.4	3000-3150	63.0-67.0	3.3.37

2.5 Fiber extraction methods

Usually, for a use of reinforcement of composite material, the vegetable fibers are taken in the chain of transformation of the textile industry. This sector has developed, for many years, techniques to separate the fibers from the rest of the plant. The techniques used to separate and present the fibers are mechanical, chemical and biological extraction **[17]**.

2.5.1 Mechanical extraction

2.5.1.1 Scutching

This method consists in separating the fibers (stalks) by mechanical action such as crushing and beating. This technique is rather used to extract flax or hemp fibers. It is carried out by automated machines which engage, maintain and release automatically the stems thanks to systems of grooved wheels with large teeth at the beginning then with finer teeth. Then, the fibers pass under the groove of the rollers with an angle close to 90° to make the crushing more efficient. The operation is carried out successively on the foot side and the head side. The short fibers, also called tow, which are less resistant, and the shives are recovered by suction and separated **[18]**.

2.5.1.2 Deflection

The extraction of the fibers is done by a combined action of scraping and beating. The machines called "raspadors" (scraper in Spanish language) grate the leaves of the plant and release the fibers. These machines mainly consist of a rotating shaft driven by a motor, on which supports holding steel beaters have been fixed. The stems inserted in front of the machine are caught between these beaters and a scissor table, grated and guided to the opposite side. The powder and the wood pass through screens. The distance between the blades is adjustable according to the batch **[18]**.

2.5.1.3 Rolling

The stalks are cut into pieces which are then crushed under a press or by rolling or by a combination of the 2 treatments. This is done several times in a row until the fibers are sufficiently separated **[18]**.

2.5.2 Chemical extraction

They allow avoiding the disadvantages of mechanical extraction, and especially a considerable saving of time and energy. In this section, we present the main methods for the chemical extraction of vegetable fibers.

2.5.2.1 Kraft process

This alkaline process aims at eliminating lignin, pectins and hemicelluloses under the action of a solution of Sodium Hydroxide (NaOH) and Sodium Sulfide (Na2S). The latter is a reducing agent; it protects the cellulose and avoids its oxidation. The cooking temperature is between 170° and 175°C for duration of 2 to 4 hours **[18]**.

2.5.2.2 Bisulfite process

This process separates lignin from cellulose fibers using various salts of sulfurous acid. The salts used in the reduction process are, depending on the pH: sulfites (SO32-) or bisulfites (HSO3-). It is based on the reaction on the lignin of calcium, sodium, and ammonium or magnesium hydrogen sulfite containing free sulfur dioxide. The pH is between 1.5 and 5 (sulfites or bisulfites), the duration is

between 4 and 14 hours and the temperature from 130° to 160°C which are also depending on the base used **[18]**.

2.5.2.3 Acid process

The non-cellulosic components are removed by the action of a preferably strong acid (sulfuric acid). The acid transforms the lignin into soluble lignosulfonic acid, or hydrochloric acid, which forms chlorolignins soluble in sodium hydroxide thanks to its chlorate ions.

2.5.2.4 Sodium hydroxyde process

This process uses only NaOH soda to dissolve the non-cellulosic subsistences such as lignin, pectin and hemicellulose, as well as the different constituents forming the reserve and the external wall of the plant stem. The temperature, pressure, concentration and duration of the treatment are to be defined according to the batch, the age and the type of the plant so as not to degrade the cellulose fibers. It is advisable to control the pH of the solution and to adjust it around 7. Reducers can be added to prevent the oxidation of the cellulose **[18]**.

2.5.3 Biological extraction

2.5.3.1 The retting process

The retting process used for the extraction of long fibers results from the separation of fiber clumps from other non-fibrous cell types through the digestion of living cells by microorganisms. The retting process consists of the degradation of water-soluble cell wall compounds **[19]**. There are many retting processes, namely:

2.5.3.2 The retting on land

The retting is a natural process intended to support the extraction of fibers. It consists in spreading the stems (of flax for example) in a field after its harvest, in order to benefit from the combined action of the sun and the rain supporting the development of microorganisms able to separate the non-cellulosic elements from the fibrous part of the plant. This operation can last from 6 to 8 weeks depending on the weather. In spite of the effectiveness of this method, it knows several disadvantages which reside in its whole dependence on the meteorological conditions (excess of humidity, very strong wind). Therefore, air retting is an efficient process if the weather is good but it remains very slow, therefore, it is a random process **[18]**.

2.5.3.3 Water retting

This type of retting is based on the same principle of development of micro organisms as the air retting; the difference is that the stalks (of hemp for example) are immersed in water during several days. The 5 to 7 kg bundles are subjected to the action of anaerobic bacteria. As soon as the fibers are detached along the whole length, the plant is plant is taken out of the water to be dried. This technique

gives less random results than the first one but it presents a major disadvantage: the pollution of water. Indeed, the retting of flax and hemp is very widespread in the north of Europe (France, Belgium, and Netherlands). The retting with water is carried out then in tank, in water (37°C) until the fibers are delignified and not sticky. This technique is in continuous regression, in favor of the retting on land **[18]**.

2.5.3.4 By microbial action

Three groups of microbial agents are capable of degrading the non-cellulosic components present in the stems or leaves of plants: bacteria, protozoa and fungi. In the first category of bacteria, there are three species, one with depolymerizing activity and another with glycosidase activity capable of hydrolyzing the main chain and cutting the side chains using the released oligosaccharides. The second one has only a depolymerize activity but is unable to use the hydrolysis products of hemicelluloses. Finally the third, which has glycoside activities but lacks depolymerize activity **[18]**.

Bibliographical references

[1] Ahmet Çağrı Kılınç, Özgür Seydibeyoğlu, in Fiber Technology for Fiber-Reinforced Composites, 2017

[2] Charlet, Morvan, Bréard, Jernot, and Gomina, Etude morphologique d'un composite naturel. La fibre de lin, Revue des Composites et des Matériaux Avancés, vol. 16, p. 11, 2006

[3] Ververis, Georghiou, Christodoulakis, Santas, , Fiber dimensions, lignin and cellulose content of various plant materials and their suitability for paper production, Industrial crops and products, vol. 19, pp. 245-254, 2004.

[4] Nardin, Interfaces fibre-matrice dans les materiaux composites. Application aux fibres vegetales, Revue des Composites et des Matériaux Avancés, vol. 16, p. 49, 2006.

[5] Djebloun Youcef. Contribution à la caractérisation des matériaux composites renforcés de fibres végétales, these de doctorat, Université Mohamed Khider – Biskra, 2018.

[6] Zabia Badr Eddine, Hidani Ibrahim, Mr. Issasfa Brahim. Extraction et caractérisation mécanique des bio-fibres, Universite Kasdi Merbah Ouargla , 2016/2017.

[7] Mme Herroudj Noura , Etude de l'introduction des fibres d'alfa de tailles differentes dans une matrice polychlorure de vinyle en vue d'ameliorer ses proprietes physico-mecanique, Université Abderrahmane Mira Bejaïa, 2010.

[8] Ben Brahim, Cheikh, influence of fibre orientation and volume fraction on the tensile propreties of unidirectional Alfa-polyester composite, Compos Sci Technol, 67,140-147, (2007).

[9] Composite material handbook, Materials science corporation, University of Delaware, army research Laboratory, july, (2000).

[10] Yahiaoui Lamia, « caractérisation d'un composite à la rupture à base des fibres végétales DISS», option mécanique applique, université de Sétif, 2011.

[11] Negoudi Abdelhakim Khinech Sayah, Etude de comportement mécanique des composites biosourcés, Universite Kasdi Merbah Ouargla, 2015.

[12] Biagiotti, Puglia, and Kenny, A review on natural fibre-based composites-part I: structure, processing and properties of vegetable fibres Journal of Natural Fibers, vol. 1, pp. 37-68, 2004.

[13] M. Ragoubi, Contribution à l'amélioration de la compatiblilité interfaciale fibres naturelles/matrice thermoplastique via un traitement sous décharge couronne, Thèse de doctorat, Nancy 1, 2010.

[14] H. Ville, Matériaux composites fibres naturelles/polymère biodégradables ou non, Thèse de doctorat, Ecole des Mines d'Alès, 2011.

[15] Alarcón-Gutiérrez, Influence de facteurs abiotiques sur la régulation des paramètres microbiens impliqués dans la dégradation de la matière organique d'une litière forestière méditerranéenne, Thèse de doctorat, Aix-Marseille 3, 2007.

[16] Thi, Vi Vi Do. Matériaux composites à fibres naturelles / polymère biodégradables ou non. Alimentation et Nutrition. thése de doctorat, Grenoble, L'université de Grenoble, 2011.

[17] BALEY, Fibres naturelles de renfort pour matériaux composites: Ed. Techniques Ingénieur. Matériaux fonctionnels, N° N2220., 2005.

[18] Dallel, Evaluation du potentiel textile des fibres d'Alfa (Stipa Tenacissima L.): caractérisation physico-chimique de la fibre au fil, Thèse de doctorat, Université de Haute Alsace-Mulhouse, 2012.

[19] Haddou Geoffrey. structure et propriétés physiques de composites à matrice biosourcée/fibres

naturelles continues pour application aéronautiques. thése de doctorat, Toulouse, L'université Toulouse 3 Paul Sabatier, 2017.

Chapter 03

Development of eco-composites with cement matrix and *Agave Americana* fiber reinforcement

Chapter 03
Development of eco-composites with cement matrix and *Agave Americana* fiber reinforcement

3.1 Introduction

Sustainability is defined by the World Commission on Environment and Development as the ability to meet current needs without compromising the ability of future generations to meet their own needs. One major problem facing mankind is the growing world population and the related pressure on construction environment. Fibers, mainly including metal fibers, synthetic fibers, and natural fibers, are widely used today for building reinforcement of cementitious composites. The existing literature is replete with studies showing that the incorporation of metal fibers or synthetic fibers can significantly enhance tensile strength, toughness, ductility, crack resistance, and impact resistance of cement composites. However, the production process of the aforementioned fibers consumes a huge amount of fossil energy and the contradiction between performance and price of these fibers cannot be effectively solved. Natural plant fibers represent a commendable solution to the above issues with their abundant supply and the characteristics of environmental protection, energy saving, and degradable regeneration **[1]**. Nowadays, the development of new bio composites based on *Agave Americana* fibers is widely required. *Agave Americana* is a natural fiber that a length of about 1 m or more and a diameter of 0.2-0.4 mm **[2]**, extracted from the leaves of the *Agave Americana* and has only been used since the nineteenth century. Its name comes from the first port of *Agave Americana* in Yucatan, Mexico. It is one of the most sought after natural fibers that are processed to make rugs, ropes, cords and yarns. Since short, thick fibers cannot be spun, bundles of fibers are used as raw material **[3].** Furthermore, it is easily available and possesses considerable tenacity and tensile strength, which contributes tremendously to its potential use in various engineering applications.

3.2 *Agave Americana* fibers

Agave Americana is a xerophyte plant that tolerates prolonged drought and high temperatures. The *Agave* gives fibers that can reach a length of 1m and characterized by their hardness. Nowadays and thanks to the advantages of natural fibers, it is increasingly used as reinforcement for composite materials, especially in automotive composites. *Agave Americana* fibers can be used as randomly distributed fibers (mats), long (unidirectional) or even (fabrics). The *Agave Americana* fibers that are the subject of this study are derived from the *Agave Americana* plant, native to Mexico (**Fig. 3.1**). This resistant plant grows all year round in hot and arid climates often unsuitable for other crops. It can grow in most types of soil except for clay, high salinity and wet soils. Its culture is relatively simple

because it resists well to the diseases. *Agave* can be harvested two years after being planted and its productive life can reach 30 years. Each plant can produce from 180 to 240 leaves depending on the geographical situation, the altitude, the rainfall and the variety considered **[4]**.

Fig. 3.1 *Agave Americana* plant

3.2.1 Identification

Agave Americana (century plant) is a vigorous, monocarpic, perennial herbaceous plant, 3-9 m high at flowering, with numerous leaves grouped in a dense rosette; fibrous roots, extending horizontally to (3-5) m, and vertically to 1,5 m, but concentrated in the first 30-40 cm of soil, base fleshy, bulbous, triangular in cross section, blade gradually widening toward the middle, then narrowing to a prickly terminal spine, dark brown, up to 3 cm long, concave above and convex below, margin usually without spines, surface dark green but covered with a white/greyish waxy layer (**Fig.3.1**). **[5], [6]** the botanical characteristics of this plant are summarized in the following table:

Table 3.1 Botanical characteristics of the *Agave Americana* plant

Common names	Century plant, sisal or American aloe
Latin name	*Agave Americana*
Family	Asparagaceae
Type	*Succulent*
Origin	Central America, Mexico
Color of the flowers	Yellow-green
Plantation	Spring
Flowering	Summer

3.2.2 World production

The main producers of *Agave Americana* fibers are: Brazil with an annual production of 120 000 t, including an export of nearly 100 000 t of raw fiber and manufactured products. Tanzania which

produces 30 000 t per year, half of which is dedicated to export. Kenya produces about 25 000 t/year and exports 20 000 t/year. Madagascar, which barely reaches 10 000 t/year **[7].**

3.2.3 Extraction of *Agave Americana* fibers

One of the main objectives of the researchers is to find out how to achieve the extraction of the fibers from natural resources and by economically profitable means and techniques. There are several methods and techniques for the extraction of *Agave Americana* fibers such as methods based on the immersion of the plant in water and those that use chemical solutions such as alkalis. The first technique consists in immersing the blades from the plant in a closed drum for 15 to 21 days at an ambient temperature of about 25°C, with the aim of accelerating biodegradation and making the extraction easier afterwards. Then the fibers are rinsed and washed with water and air-dried and brushed. The second method is based on the use of a mechanical means for the extraction of the fibers by a cutting machine or by the use of a scraper. Another relatively new technique is the burial of *Agave Americana* blades in the ground at a depth of 30 cm for 90 to 100 days, which promotes the biodegradation of the blade. The extraction of the fibers becomes easy, the fibers are then rinsed and washed with water and air-dried **[8]**. In our study we used *Agave Americana* blades (**Fig.3.2**) and (**Fig.3.3**). In order to separate the fibers from the rest of the blade and thus obtain their extraction we used a method that consists of immersing the blades in a closed drum full of water for 21 days, and then crushing the blades by a blunt knife to remove the outer envelope of the plant and release the fibers (**Fig.3.4**) and (**Fig.3.5**). Then the fibers were immersed in water for 2 days at an ambient temperature as shown in (**Fig.3.6**). Finally the fibers were left to dry at room temperature (of approximately 25 °C) for 12 hours (**Fig.3.7**).

Fig. 3.2 *Agave Americana* plant from The region of Tebessa.

Fig. 3.3 Blades used in *Agave Americana* Fiber Extraction.

Fig. 3.4 Crushing of *Agave Americana* fibres

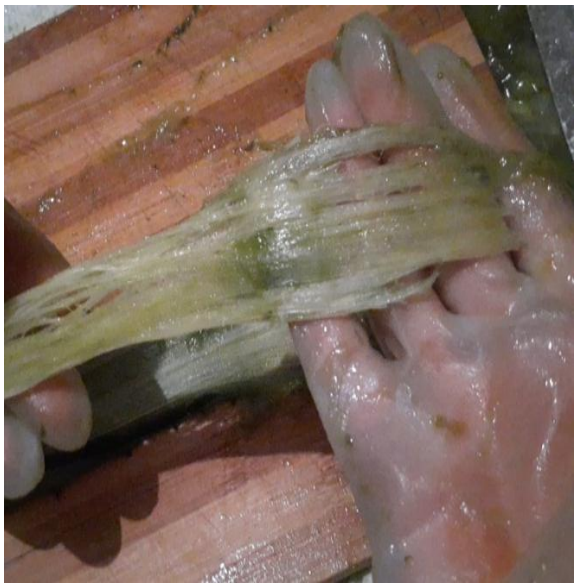


Fig. 3.5 Extraction of *Agave Americana* fibers

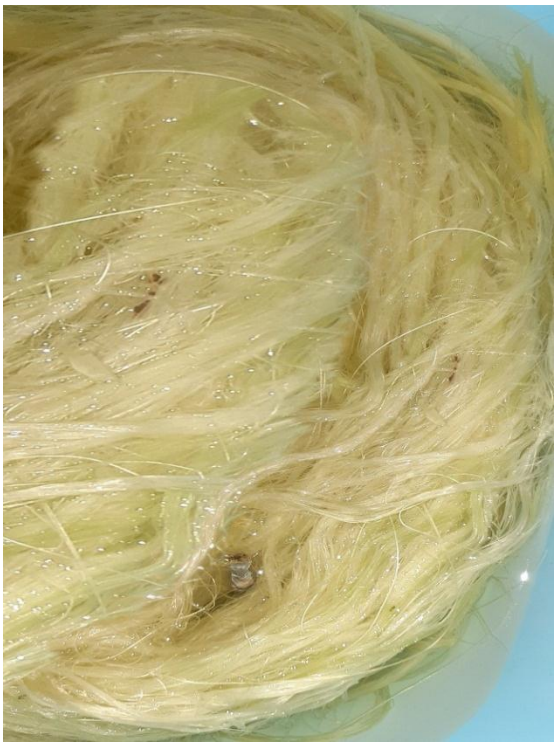


Fig. 3.6 ***A****gave Americana* fibres before drying.

Fig. 3.7 *Agave Americana* fibers after Drying.

3.2.4 The chemical composition of *Agave Americana* fibers

In general, the chemical composition of *Agave Americana* fibers is approximately as follows (70%) cellulose, (12%) hemicellulose, (10%) lignin and a small amount of pectin, waxes and fat (2%) **[9]**.

3.2.5 Fiber Surface Modification

In order to improve the physical and mechanical properties of composite systems made of *Agave Americana* fibers and the matrix, *Agave Americana* fibers must undergo several chemical and thermal treatments.

3.2.6 Mechanical Characterization of *Agave Americana* Fibers

Although there are many types of *Agave Americana*, not all of them can be used as reinforcement in polymers. The *Agave Americana* fibers chosen must have good mechanical properties (e.g., high Young's modulus).

3.3 Experimental program and test methods

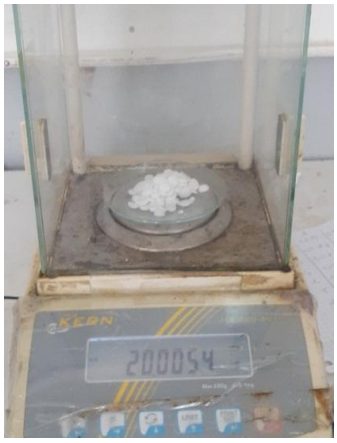
3.3.1 Alkaline treatment with low concentrations of Sodium hydroxide (NaOH) at room temperature

The process and the different steps followed for the treatment of the fibers the fibers were first mesured at 250g stacks (**Fig.3.8**).

- The dilution of NaOH was done by small grammage to avoid the heating of the solution. For 1 L of distilled water we have 20g of NaOH.(**Fig.3.9**) .
- The fibers were soaked in a 2% soda solution (NaOH) (**Fig.3.10**), for a period of 4 hours at room temperature.(**Fig.3.11**).
- After completion of the treatment time, the fibers were rinsed with tap water several times (**Fig.3.12**).
- The fibers were then immersed in a distilled water solution with 2% sulfuric acid (**Fig.3.13**) for 10 min. to neutralize the excess soda (**Fig.3.14**).
- They were then washed (**Fig.3.15**) several times with tap water, (**Fig.3.15.a**).
- and finally they were rinsed with distilled water for 15 minutes until neutral pH (**Fig.3.15.b**).
- At the end the fibers were dried at 60°C for 6 hours in an oven(Fig.3.16). Once dried, the fibers were brushed then (**Fig. 3.17**) cut to 15 mm lentgh (**Fig.3.18.a**) and stored in a bag (**Fig.3.18.b**).

Fig 3.8 250 g stack of *Agave Americana* fibers .

a.Sodium hydroxide.



b.Mesuring 20g of NaOH

c. 20g of NaOH.

d.1 liter of distilled water

e. adding the NaOH to the distilled water

f. 2% sodium hydroxide solution.

Fig 3.9 Preparation of sodium hydroxide solution

Fig 3.10 The fibers soaked in 2 % Soda Solution.

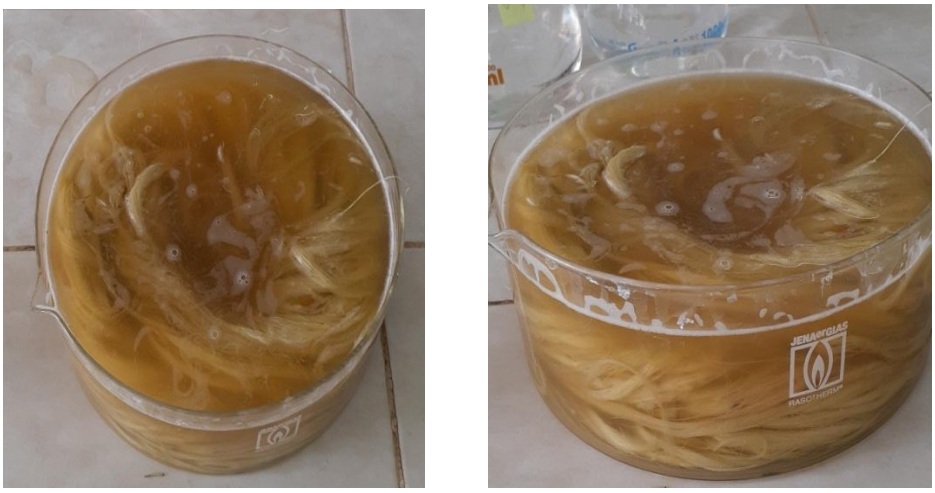
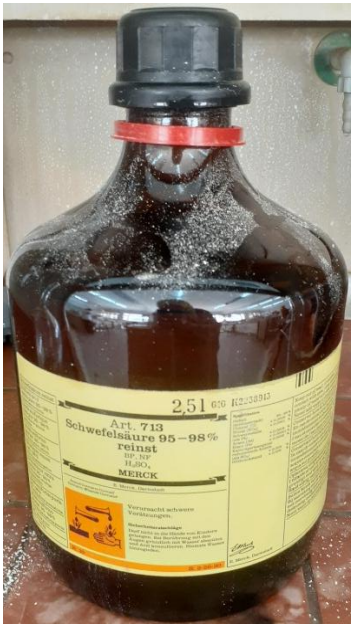


Fig 3.11 The fibers after soaking for 4 hours in NaOH solution.

Fig 3.12 Rinsing the fibers with tap water several times.



a.sulfuric acid

b. mesuring 20ml of sulfuric acid

c. 1L of distilled water

d. 2% sulfuric acid solution

Fig 3.13 Preparation of 2% sulfuric acid .

Fig 3.14 The fibers soaking in 2% sulfuric acid somution for 10 min.

a. washing the fibers with tap water for several times

b. washing the fibers with distilled water for 15 minutesuntil neutral pH

Fig 3.15 Washing the fibers after treatments .

Fig 3.16 The treated fibers drying in the oven.

Fig 3.17 Brushing of treated *Agave Americana* fibers

a. Fibers cut to 15 mm length b. fibers stored in a bag

Fig 3.18 Cutting and storing the treated fibers

The fibers were washed with distilled water and then dried at 60 ◦C for 6 hours in an oven once dried they were cut to 15 mm length and stored at room temperature.

3.3.2 Mortars reinforced with plant fibres

3.3.2.1 Introduction

Several industrialists are trying to exploit the flora, rich in plant and marine fibers. For this reason, they have used plant fibers as reinforcement in cement matrix composites. Indeed, several works have been interested in cements reinforced by plant fibers in order to analyze its mechanical, thermal and also acoustic properties. Among the fibers used are hemp, bagasse , coir, *Agave Americana* etc..Agave Americana plant (sisal), is a plant biomass, renewable, light and low cost. It can be exploited to reinforce composite materials with cement matrix and open new industrial axes. The study of these

fibers will be done through an experimental campaign, on fibrous cementitious composites with different rates of fibers, based on 3-point bending tests, and compression tests.**[10]**

3.3.2.2 Characterization of used materials

3.3.2.2.1 Cement

The cement (**Fig.3.19**) used in all mixtures was a Portland Composite Cement (CEM IIA/M-(P-L) 42.5 N), produced by Cement Company of Tebessa conforming to Algerian standard NA 44 **[11]**.

Table 3.2 Technical data sheet - product CEM IIA/M-(P-L) 42.5 N) **[11]**

mechanical tests (NA 234)			
	deadline in days	**Guarantees NA 442**	**Measures**
bending resistance MPa	02 days		4.7
	07 days		7.2
	28 days		8.1
compressive strength MPa	02 days	≥ 20	23.3
	07 days		40.0
	28 days	≥ 42.5	51.0

Fig 3.19 Cement (CEM IIA/M-(P-L) 42.5 N).

3.3.2.2.2 Sand

The sand (**Fig.3.20**) used is standard sand certified (CEN, EN 196-1) from the quarries of the New Company of LITTORAL, originally in France. It is limestone sand with an apparent density of 1.42 g/cm^3 **[11]**.

Fig 3.20 Standard sand.

3.3.2.2.3 Fiber

Agave Americana fibers as described and mentionned previously. Note that this work was supported by the Cement Company of Tebessa Laboratory part of the Industrial Group of Algerian Cements.

3.3.4 Specimen preparation

The specimens are made according to a reference mortar (control) prepared in accordance With the European Standard EN 196-1. For the control mortar, the composition of cement, sand and water is 1: 3: 0.5 with 450 ± 2 g of cement, 1350 ± 5 g of sand and 225 ± 1 ml of water.

Concerning natural fibers reinforced cementitious mortars; different raw and treated fibers percentages (by decreasing the weight of the sand each time) are added to the mixture: 0.5, 1, and 1.5%. The process of adding the fibers to the mixture was performed by hand gradually adding and mixing the treated and raw fibers for five minutes, in order to promote and facilitate the fibers dispersion in the mixture. The samples were placed in steel prismatic molds of 40 ×40 × 160 mm^3 (**Fig.3.21**) and then using a shock table (**Fig.3.22**) the molds were subjected to 60 blows in two phases to get rid of air bubbles. The samples after that were placed for 24 h in a humid chamber with a temperature of 20°C and a humidity of 90,5% (**Fig.3.23**) . After demolding, the mortar specimens are left in water to cure until ready to be tested (2days, 7days and 28 days). The designations of the various mortar mixes are noted in Table 3.3.The stoppage at 1.5% is related to the problem of mixing, because above 1.5% the fibers agglomerate leading to poor dispersion in the matrix. In addition, the length of the fibers is chosen to be 15 mm in order to ensure a homogeneous distribution of these fibers in the mortar samples.

Table 3.3 The designations of the various mortar mixes

Fibers		Sand ± 5 (g)	Cement ± 2 (g)	Water ± 1 (ml)	Final samples
%	(g)				
0.5% T	8.5	1341.5	450	225	Fig 3.24
1% T	17.1	1332.9	450	225	Fig 3.25
1% R	17.1	1332.9	450	225	Fig 3.26
1.5% T	25.6	1324.4	450	225	Fig 3.27

T: treated fibers.
R: raw fibers.

Fig 3.21 Steel prismatic mold of 40×40×160 mm^3

Fig 3.22 Shock table

Fig 3.23 Humid chamber

a. measuring the fibers.

b. measuring the sand

c. measuring the cement

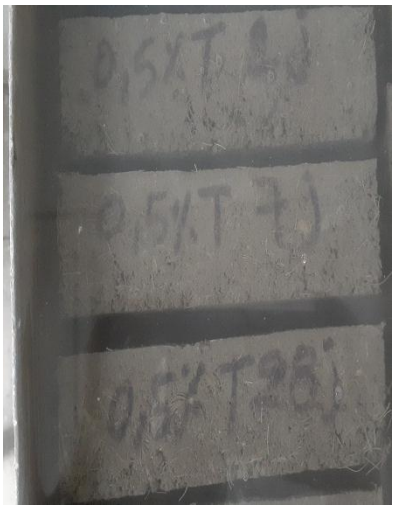
d. measuring the water

e. adding the fibers to the mixture

f. dispersion of the fibers in the mixture.

g. final samples

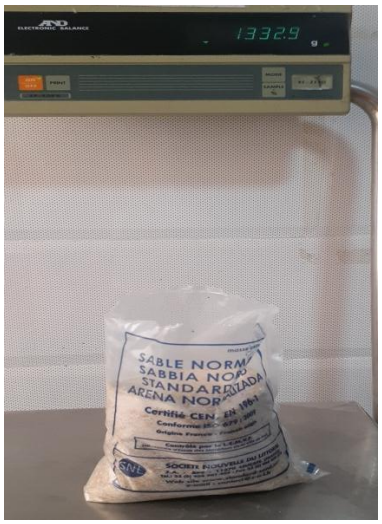
h. distribution of fibers on the sample surface



i. samples in the water

Fig 3.24 0.5% treated fibers mortar

a. measuring the fibers.



b. measuring the sand

c. measuring the cement

d. measuring the water

e. adding the fibers to the mixture

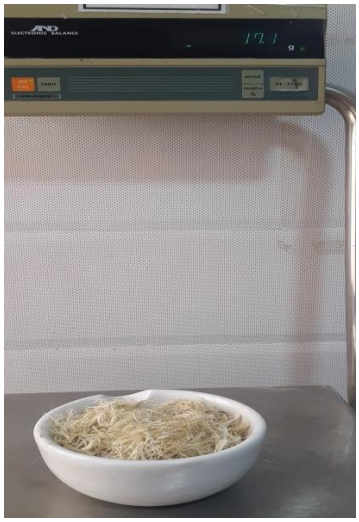
f. dispersion of the fibers in the mixture.

g. final samples

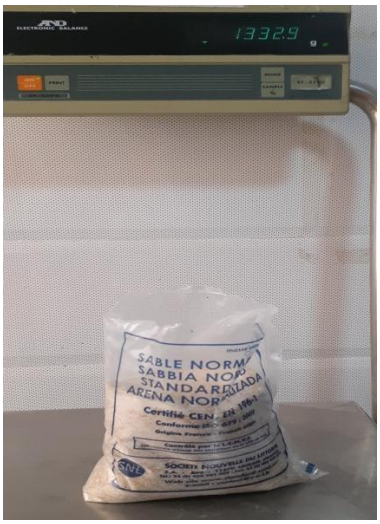
h. distribution of fibers on the sample surface

i. samples in the water

Fig 3.25 1% treated fibers mortar



a. measuring the fibers.



b. measuring the sand

c. measuring the cement

d. measuring the water

e. adding the fibers to the mixture

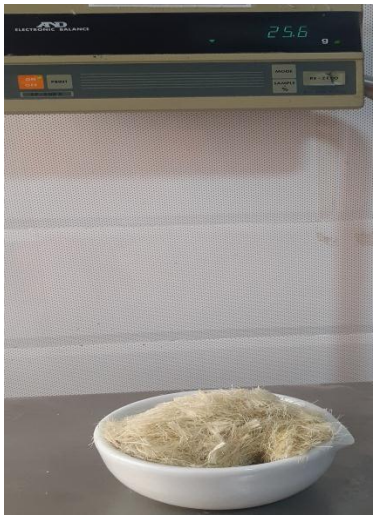
f. dispersion of the fibers in the mixture.

g. final samples

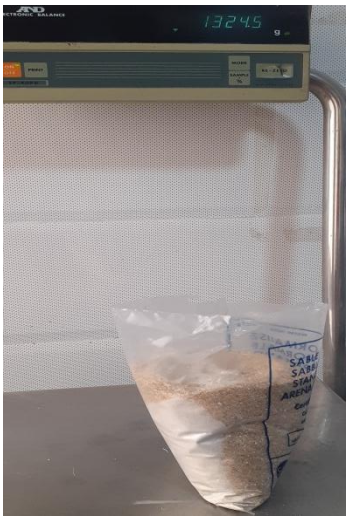
h. distribution of fibers on the sample surface

i. samples in the water

Fig 3.26 1% raw fibers mortar



a. measuring the fibers.



b. measuring the sand

c. measuring the cement

d. measuring the water

e. adding the fibers to the mixture

f. dispersion of the fibers in the mixture.

g. final samples

h. distribution of fibers on the sample surface

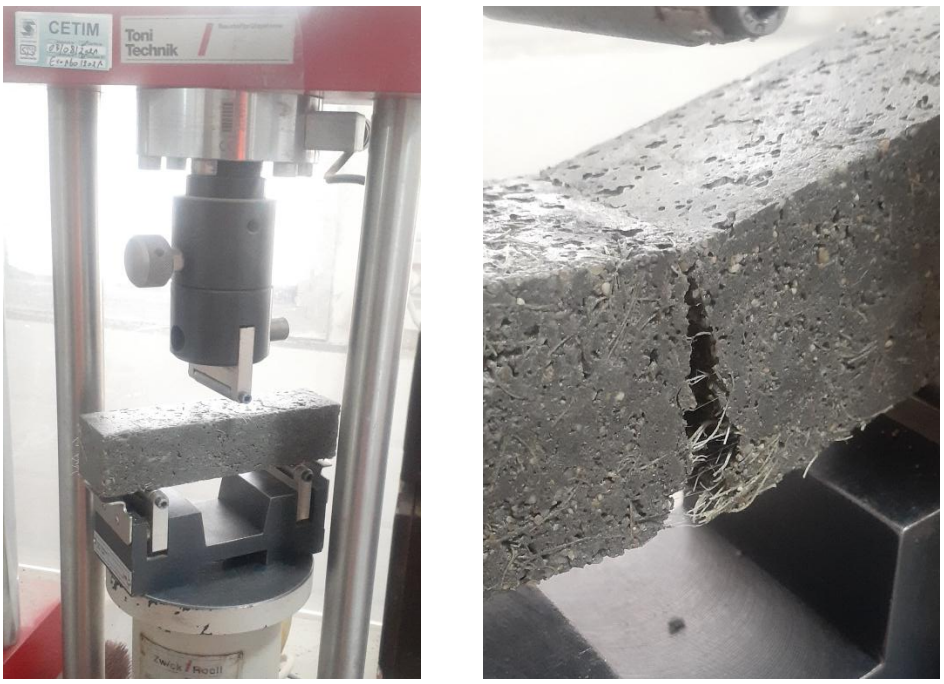
i. samples in the water

Fig 3.27 1. 5% treated fibers mortar

3.5 Mechanical strength Characteristics of cement-*Agave americana* fiber composites

3.5.1.Identification of flexural tensile strength

The three-point bending (**Fig.3.30**) test is carried out in accordance with European Standard EN 196-1. The 4. 4. 16 cm3 prismatic specimens were prepared and tested after 2,7and 28 days of water curing to study the evolution of flexural tensile strength results (**Fig.3.32**) as a function of time. For each mixture formulation studied.



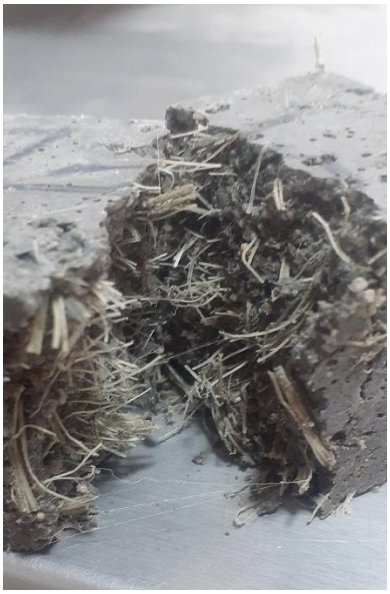
a. 3-point bending assembly b. specimen in bending test

Fig 3.28 Flexural tensile strength machine

3.5.2 Identification of compressive strength

The compressive strength (**Fig.3.31**) of the specimens were carried out following the flexural tests on two broken pieces left from the flexural test according to the European Standard EN 196-1. The mortar specimens, 4. 4. 16 cm3 in size, were divided into two specimens at each age, compressive strength testing of each specimen was performed, and the results (**Fig.3.33**) were averaged .Compressive strength of mortar cubes was determined after 2, 7 and 28 days of water curing.

a.Compressive strength assembley



b. spicemen after compressive srength test

Fig 3.29 Compressive strength machine

3. 6 Results and discussion

Table 3.4 Mechanical strength Characteristics of cement-*Agave americana* fiber composites results

Specimen ratio (%)	Curing period (days)	Flexural strength (N/mm²)	Compressive strength (kg/cm²)		
			Cube 1	Cube 2	Average results
0.5 T	2	3.8	15.2	16.4	15.8
	7	4.1	27.5	28.1	27.8
	28	6.4			38.2
1 T	2	2.6	12.3	12.7	12.5
	7	3.4	17.9	15.5	16.7
	28	3.9			20.4
1 R	2	2.2	10.8	10.7	10.7
	7	4.0	18.8	20.5	19.6
	28	4.9			28.0
1.5 T	2	1.5	5.8	5.8	5.8
	7	2.2	13.7	10.8	12.2
	28	3.0			18.6
Z	2	4.1			19.6
	7	6.0			37.2
	28	7.2			48.2

T : treated fiber specimen

R : raw fibers specimen

Z : witness specimen

3.6.1 Effect of fibers addition on the flexural strength

Figure **3.32** shows the variations in flexural strength of composite specimens as a function of the fiber ratio content in the specimen after 2, 7 and 28 days of curing. As it can be seen in this Figure, the best flexural strength is obtained for the 0.5% fibers ratio for 28 days curing period .As it can be seen in same Figure the flexural strength of the mortar samples decreased gradually with increasing fiber ratio in the mixture content. Previous researches shows that the treated fiber reinforced mortar samples have more strength than the raw fiber reinforced mortar samples This behavior is related to the effect of the chemical treatment of the fiber which removes the amorphous materials on the outer surface of the fiber producing a rough surface which improves the interlocking and interfacial adhesion between the fiber and the cementitious matrix, resulting in an increase in the tensile strength of treated fiber reinforced cementitious composites**.** **[12] [13]**

Unlike what has been previously mentioned in our trial we found that the raw fiber reinforced mortar samples have more strength than the treated fiber reinforced mortar samples (**Fig.3.33**). Unfortunately we cannot explain this behavior exactly due to the lack of conditions necessary to carry out experiments to explain it.

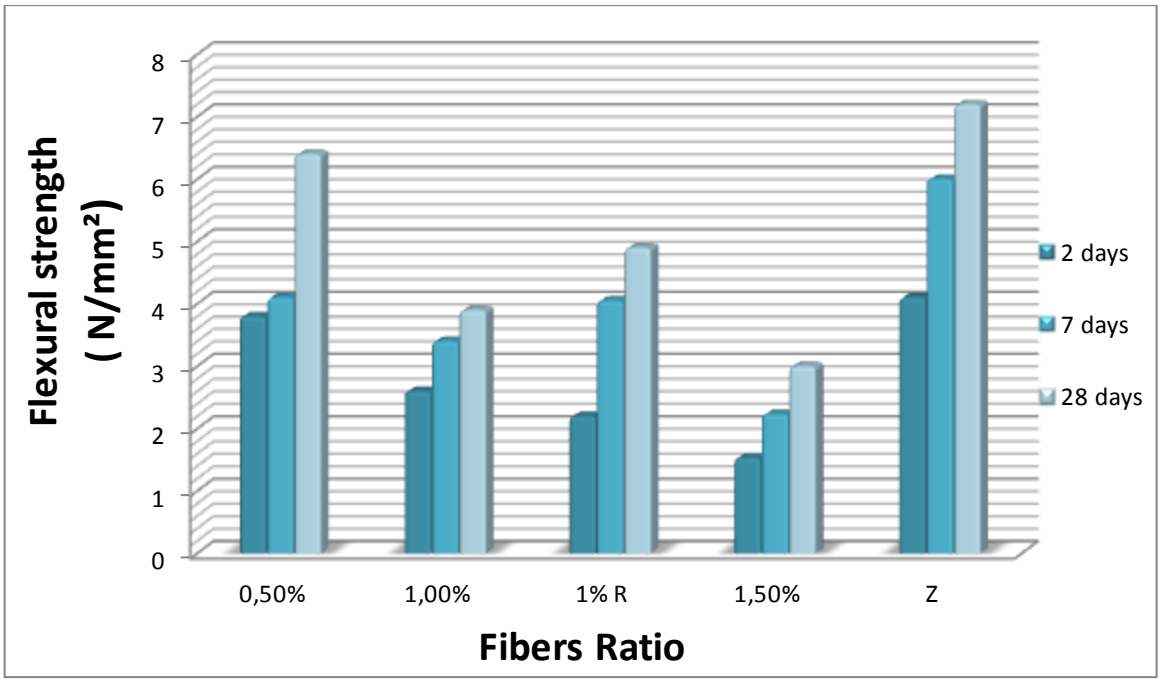


Fig 3.30 Influence of *Agave Americana* treated and raw fiber ratio on the flexural strength of mortars

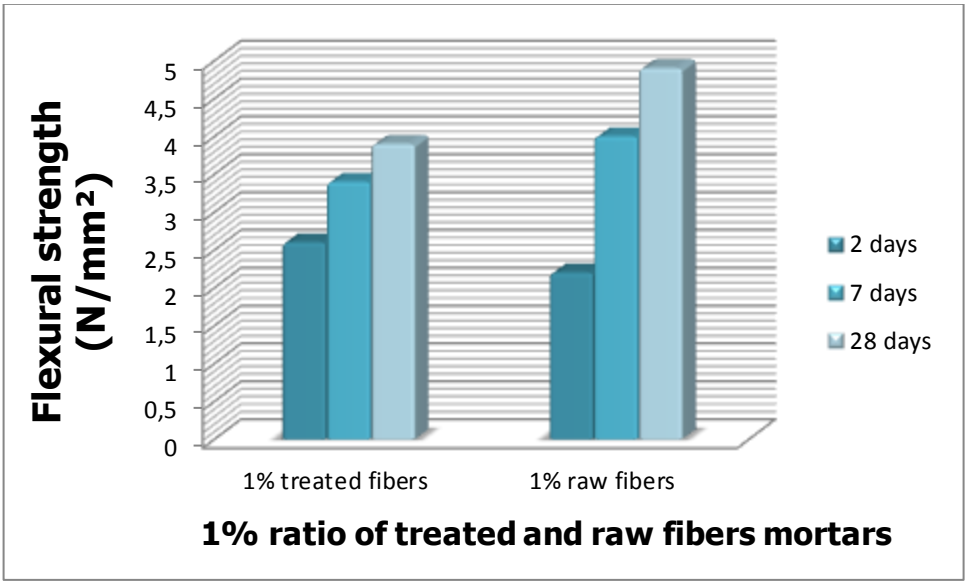


Fig 3.31 Influence of 1 % *Agave Americana* treated and raw fiber ratio on the flexural strength of mortars

3.6.2 Effect of fibers addition on compressive strength

The variations in compressive strength of composites as a function of the fiber ratio content at 2, 7 and 28 days of curing are illustrated in Figure **3.34**. The results indicated that there is a decrease in strength with increasing fiber ratio content for all composite specimens. Whereas the raw fiber reinforced mortar samples have more compressive strength than the treated fiber reinforced mortar samples (**Fig.3.35**).

This decrease of strength of fiber reinforced mortar is due to the creation of large number of pores or voids might be due to the bad distribution of porous fibers in cement matrix. When the fiber ratio content increases, the cohesion of the composites decreases and the porosity increases, subsequently leading to a considerable drop in their compressive strength.

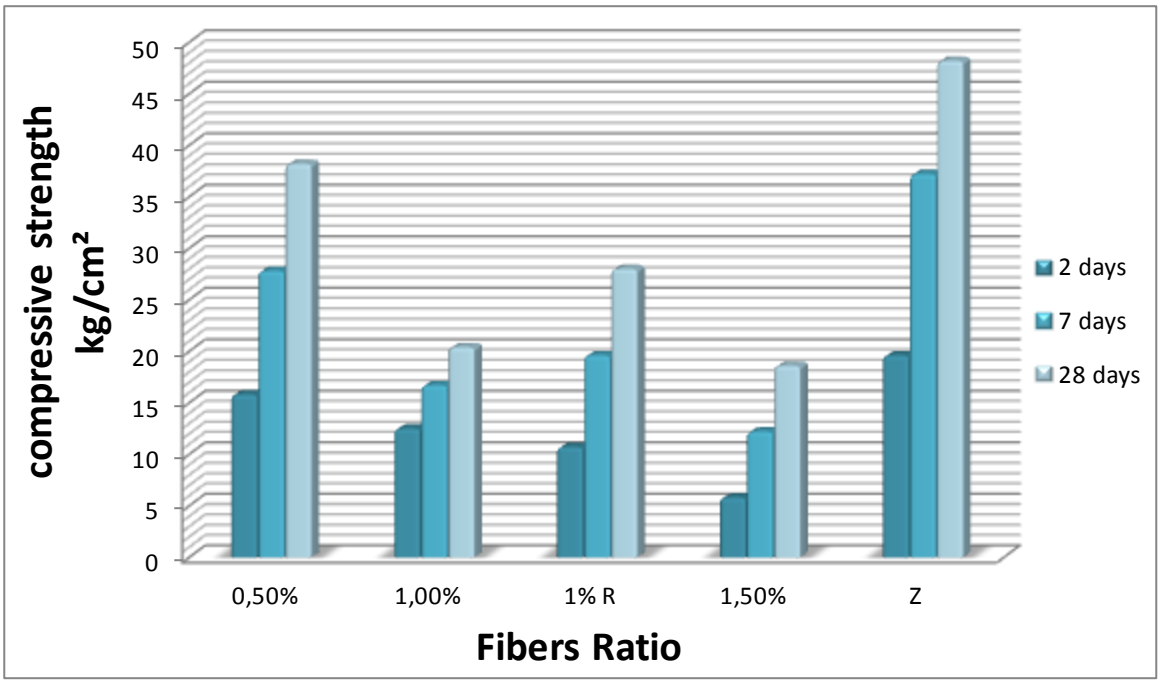


Fig 3.32 Influence of *Agave Americana* treated and raw fiber ratio on the compressive strength of mortars

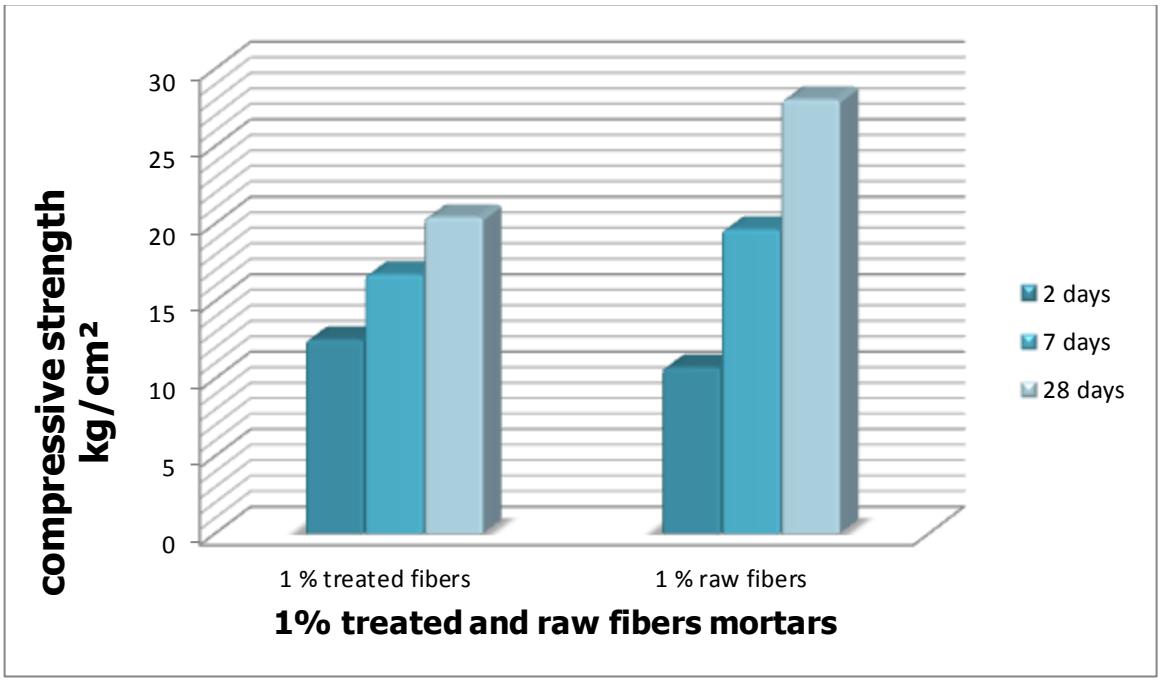


Fig 3.33 Influence of 1 % *Agave Americana* treated and raw fiber ratio on the compressive strength of mortars

3.7 Advantages of fibered cement

The advantages of using fibers in concrete are presented according to the technical and economic aspects.

3.7.1 Technical aspect

The use of fiber-reinforced concrete is advantageous mainly in terms of crack control and load bearing capacity, depending on the type of fiber and the amount used.

The main technical advantages are:

- three-dimensional reinforcement uniformly distributed throughout the concrete;
- an increase in toughness due to post-cracking behavior (residual strength);
- High absorption energy;
- High impact resistance;
- High fatigue resistance;
- Increased shear strength.

3.7.2 Economic aspect

The main economic advantages of fiber-reinforced concrete are:

- a reduction in the number of people working on the site, which implies a reduction in the cost of installation and construction time;
- An optimization of the dimensioning.

Bibliographical references

[1] Kaiyue Zhao, Shanbin Xue, Peng Zhang , Yupeng Tian and Peibing Li, Application of Natural Plant Fibers in Cement-Based Composites and the Influence on Mechanical Properties and Mass Transport, Article, Center for Durability & Sustainability Studies of Shandong Province, Qingdao University of Technology, Qingdao 266033, China, 25 October 2019

[2] https://jardinage.lemonde.fr/dossier-496-agave-americana-plante-tequila-mezcal.html accessed on may 2022

[3] https://www.complements-alimentaires.co/sisal/ accessed on may 2022

[4] http://www.fao.org/economic/futurefibres/fibres/sisal/en/ accessed on may 2022

[5] https://www.wgc.de/fr/produkte/sisal accessed on may 2022

[6] https://www.benuta.fr/blog/materiau-autres-fibres-naturelles-sisal-jute-bambou coco.html accessed on may 2022

[7] https://sisal.ooreka.fr/comprendre/fibre-sisal accessed on may 2022

[8] Belaadi Ahmed, Elaboration et caractérisation des matériaux composites à renfort en fibres naturelles (fibre de sisal), Mémoire de Magister, Université 08 Mai 1945- Guelma, (2011).

[9] Djebloun Youcef, Contribution à la caractérisation des matériaux composites renforcés de fibres végétales , Thèse de Doctorat en Génie mécanique de l'université de Biskra (2018).

[10] Zidi and Sghaier Propriétés physiques et mécaniques des ciments renforcés par des fibres de PosidonieL. Allègue, Laboratoire de Génie mécanique(LGM), Ecole Nationale d'Ingénieur de Monastir-5000Monastir- Tunisie

[11] http://www.sct.dz/ciments.html accessed on may 2022

[12] Cao, Shibata , Fukumoto . Mechanical properties of biodegradable composites reinforced with bagasse fibre before and after alkali treatments. Compos Part A: Appl Sci Manuf. 2006;37:423–429.

[13] Sedan , Pagnoux , Smith , et al. Mechanical properties of hemp fibre reinforced cement:influence of the fibre/matrix interaction. Eur Ceram Soc. 2008;28:183–192.

Chapter 4

Mathematical and statically modeling and characterization of mechanical behavior of elaborated eco-composites

Chapter 4

Mathematical and statically modeling and characterization of mechanical behavior of elaborated eco-composites

Part 1: Overview of experimental designs and basic concepts of statistical laws

4.1.1 Introduction

The purpose of this investigation is to develop predictive mathematical models of the mechanical behavior in bending and compression of bio-composites with cement matrix reinforced with plant fibers and to understand the mechanisms that govern this behavior. The analysis of the mechanical behavior (bending tensile test and compression test) of these bio-composites was carried out through an experimental study as well as a statistical method using the design of experiments and the Design Expert software.

4.1.2 Main advantages of the experimental design

The most important advantages of this method are **[1]**:

- Reduction in the number of tests;
- Possibility of studying a large number of factors;
- Detection of interactions between factors;
- Modeling of the studied responses;
- An optimum precision of the results;
- The design of experiments method enables a fast and accurate interpretation by providing a precise experimental model of the studied system;

4.1.3 Definition of experimental designs

Design of experiments (DOE) also known as experimental design is a method for planning or organizing scientific and industrial experiments in such a way as to obtain the information corresponding to the objective previously set. Their purpose is to achieve the maximum amount of information with the minimum number of experiments in relation to the objective set. Design of experiments are applied in different fields of science. The comprehension of the design of experiments method is based on two essential notions, that of experimental area and that of mathematical modeling of the studied quantities **[2-3]**. In general, an experimental design consists in emphasizing and quantifying the influence existing between two types of variables:

- Factors
- Response

4.1.3.1 Factors

Factors are the variables that we want to study and that are supposed to have an influence on the studied system. They are limited by two bounds: lower (low) and upper (high). The low level is designated by the sign (-1) and the high level by the sign (+1), (**Fig.4.1**) **[2]**.

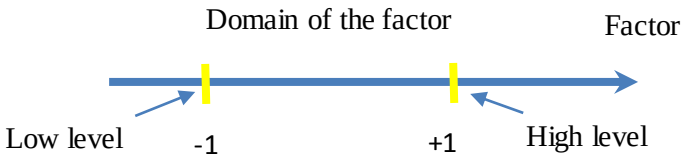


Fig.4.1 Domain and factor levels

4.1.3.2 Response

An experimental response is a measured phenomenon that is observed when the parameters being studied are varied. In other terms, it is the measured result of a study **[1]**. This quantity is often quantitative and measurable as the compressive strength, bending strength, it can as well be qualitative, for example a visual appreciation of the surface state of a metal (degradation, no degradation) **[1]**. Knowing that to each location of a studied domain corresponds a response. All these responses form the response surface **[4]** (**Fig.4.2**). A phenomenon can be described by many experimental responses **[2,3,5]**.

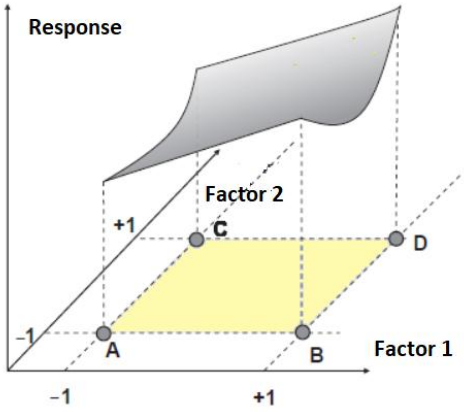


Fig.4.2 Response surface

4.1.3.3 Experimental area

An experimental area is represented by a two-dimensional (factor1, factor2) Cartesian reference system, as shown in (**Fig.4.3**) **[4]**.

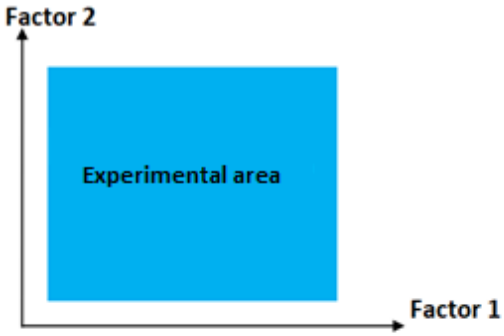


Fig.4.3 Representation of the experimental area

4.1.3.4 Experimental domain

It provides the space in which the factors can be varied. The information obtained from the experimental results will only be valid in this domain. The experimental domain is therefore usually a square for two factors, a cube for three factors, etc. (**Fig.4.4**).

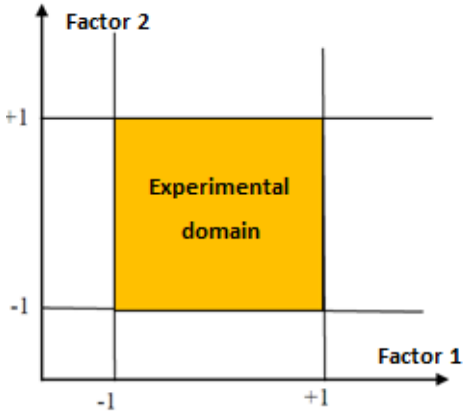


Fig. 4.4 Representation of a two-factor experimental domain

4.1.3.5 Experiment matrix

An experiment matrix is a mathematical object, which gathers the levels taken by the coded variables for the various experiments to be carried out within the limits of the chosen design. It consists of (N) rows corresponding to the number of experiments and (K) columns corresponding to the number of variables.

4.1.3.6 Experimental design

An experimental design corresponds to the conversion of the experiment matrix into a matrix that can be directly used by the experimental designer, since the variables will be expressed in natural variables. The experimental design should be carefully examined to ensure that all experiments are achievable and without risk **[6]**.

4.1.3.7 The effect of a factor

The effect of a factor (X_i) is the comparison between the values of the response (Y) when this last one passes from level (-1) to level (+1). It is marked by a constant noted (A_i) which would be:

- Positive if this variation of the factor is favorable to the increase of the response (**Fig.4.5**).
- Negative if it is favorable to the decrease of the value of the response (**Fig.4.5**).

This constant is involved in the mathematical model used to describe this response.

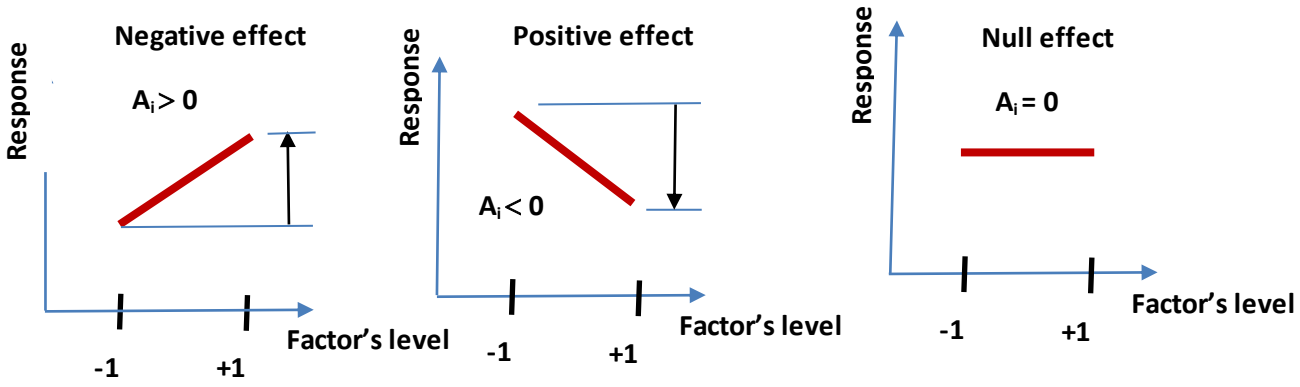


Fig.4.5 Effect of a factor

A linear model without interaction between its factors is given by the following relationship:

$$Y = A_0 + \sum_i^k A_i X_i \qquad (4.1)$$

Y: Response predicted by the model

A_0: Constant of the model it responds to the average of the measurement results

X_i: Factor i

A_i: Effect of factor i

k: Number of factors studied

4.1.3.8 Interaction

An interesting concept is the interaction between two factors. We are referring to an interaction between two factors (X_i) and (X_j) when the effect of the factor (X_i) on the response will depend on the value of the factor (X_j). Noted (A_{ij}) the interaction occurs in the model in the following manner:

$$Y = A_0 + \sum_{=1i}^K A_i X_i + \sum_{ij} A_{ij} X_i X_j \qquad (4.2)$$

A_{ij}: Interaction effect Xi Xj

4.1.4 Experimental methodology

In a study based on experimental designs, the approach to be respected is the follows:

- Definition of the objective ;
- Choice of the experimental answers ;
- Choice of the factors and the experimental domain of interest;
- Establish the experimental strategy;
- Construction of the matrix of experiments;
- Construction of the experimental design ;
- Experimentation;
- Calculation of the estimates of the information sought;
- Interpretation of the results;

4.1.5 Types of experimental designs

There are many classical experimental designs appropriate for all cases encountered by an experimenter, the most commonly used are **[4,7]** :

- Screening designs: to find the most influential factors on a response.
- Modeling designs or designs for response surfaces: 1^{st} or 2^{nd} degree models.
- Mixture designs: appropriate for dependent factors.
- Full factorial designs: all combinations of factor levels are present.
- Fractional factorial designs: all levels of each factor are present, but not all possible combinations of factors.

Factorial designs and response surfaces are useful for studies with independent factors. Mixture designs are widely used to study formulations. Combined designs are designs that combine process factors and fractions of mixture components. Among these different designs, the response surfaces are of particular interest, because they allow the study of the mode of action of the factors on the responses, and also allow the prediction and optimization of responses. It is this type of design that will be developed in this study.

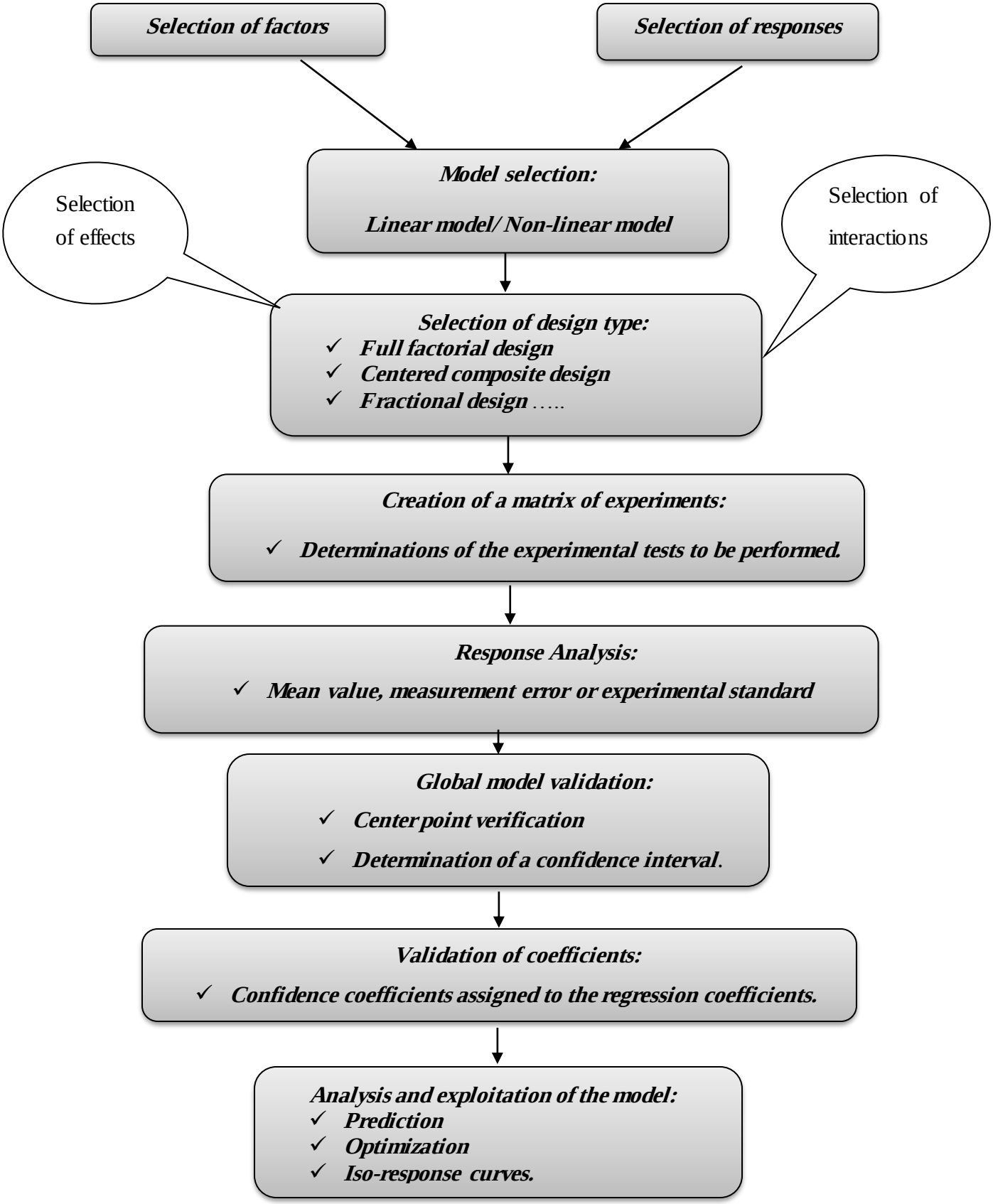


Fig.4.6 Organizational chart general treatment of plans of experiments **[5]**

4.1.6 Central composite plane

A central composite plane (Box and Wilson plane) is a plane used to construct a response surface. They are used to build second order mathematical models. They are applied to continuous variables.

A composite design consists of three parts:

- A factorial design with factors taking two levels;
- At least one experimental point located in the center of the study domain;
- Axial points, these experimental points are located on the axes of each factor.

(**Fig.4.7**) shows a composite design of two factors. Points A, B, C and D are the experimental points of a factorial design 22. E is the central point (it can be repeated one or more times), F, G, H and I are the axial points; they form what is called the star design **[1]**.

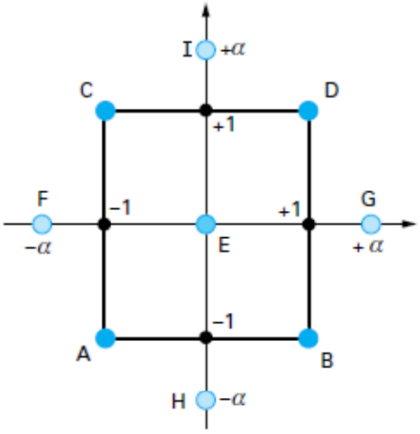


Fig.4.7 Composite design with two factors

4.1.7 Computation and refining of the model

4.1.7.1 Analysis of variance

The analysis of variance (ANOVA or Analysis of Variance) permits to compare the variances of the values calculated by the model and the residuals. This analysis consists of a statistical test (Fisher - Snedecor test).

4.1.7.2 Fisher (F-value)

F Fisher represents the ratio of the mean square of the model to the one of the residuals. This ratio is used to estimate the probability that these two squares are not equal.

4.1.7.3 Probability (P-value)

The most important statistical quantity in an analysis of variance is the P-value. If it is less than 0.05, it is concluded that the effect is significant, and if it is less than 0.01, it is assumed that the factor is highly significant **[1]**.

4.1.7.4 Coefficient of determination (R^2)

The coefficient of determination, (R^2) is a measure that assesses the ability of a model to predict or explain an outcome in the linear regression setting. It is a number between 0 and 1 that measures how well a statistical model predicts an outcome. In summary, as the coefficient of determination approaches 0, the scattering of the points around the regression line increases. On the opposite, the closer the R^2 is to 1, the more the scatter plot tightens around the regression line. This coefficient is given by the relation below:

$$R^2 = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \quad (4.3)$$

$\hat{y}_i$: Computed response,

y_i: Measured response

$\bar{y}$: Average response.

4.1.7.5 Adjusted coefficient of determination (R^2_{adj})

The Adjusted Coefficient of Determination (Adjusted R-squared) is an adjustment for the Coefficient of Determination that takes into account the number of variables in a data set. It also penalizes you for points that don't fit the model.

$$R^2_{adj} = \frac{\frac{\sum_{i=1}^{n}(\hat{y}_i - y_i)^2}{n - \upsilon_{model}}}{\frac{\sum_{i=1}^{n}(y_i - \bar{y})^2}{n-1}} \quad (4.4)$$

ϑ_model: number of degrees of freedom (ddl) of the model

Because of the consideration of degrees of freedom, we always have $R^2aju \leq R^2$.

4.1.7.6 Residues

It relates to the disparity between the measured and calculated response values. The following relationship is being used to compute the average of the residuals:

$$Aver_{residues} = \frac{\sum e_i}{N} \quad (4.5)$$

In this relationship, (e_i) represents residual and (N) the number of trials.

4.1.7.7 Graphical representation

In an analysis of a physical phenomenon, the graphical representation represents an essential tool for the interpretation of the results, but it also makes it possible to draw conclusions more quickly and to

orientate the continuation of the study **[1,8]**. One of the main advantages of experimental designs is the presentation of results in graphical form (**Fig.4.8**).

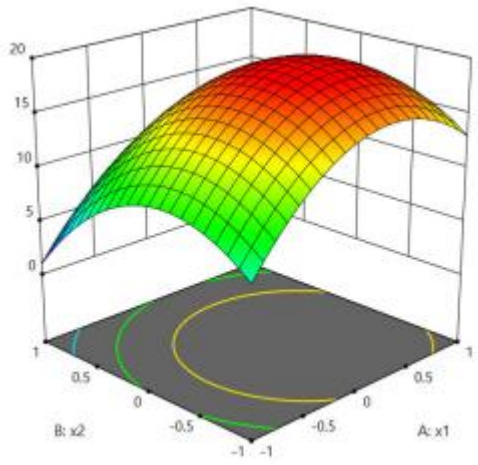


Fig.4.8 Example of a response surface curve **[1]**

Part II: Mathematical modeling of the mechanical behavior of processed composites

4.2.1 Bending behavior (uni-factorial models)

As indicated in chapter 3, we have conducted a campaign of 3-point bending tests on normalized specimens. The results obtained are being used to analyze the mechanical bending behavior of composites made of cement matrix reinforced with *Agava Americana* plant fibers with different percentages (0.5, 1 and 1.5 %). The tests were performed after a standard drying time (2 days, 7 days and 28 days). The results are shown in Table **4.1**. The percentage of fibers (factor P) is fixed and the evolution of the bending stress (σ_{flex}) is examined as a function of the drying time (T). **Fig.4.9** depicts the different (σ_{flex}) curves as a function of (T). The models are generated by interpolating the results of the experimental tests. We used the Excel spreadsheet to plot the curves (**Fig.4.9**) and to obtain logarithmic models with a very good correlation as illustrated in Table **4.1**. These models permit us to determine the values of the bending failure stresses (σ_{flex}) for any chosen value of the drying time in the interval [2 days to 28 days]. As an example, for a drying time of 15 days and for different fiber percentages (0.5, 1 and 1.5 %) the bending failure stresses values are represented in Table **4.1.**

Table 4.1 Bending stresses at a drying time of 15 days and for different percentages (P)

Fiber percentages (%)	Model	σ_{flex} (MPa)	Coefficient of correlation R^2
0.5	σ_{flex} = 1,0178×ln(T) + 3,0307	5.7869	0.9939
1	σ_{flex} = 0,4915×ln(T) + 2,2883	3.6193	0.9946
1.5	σ_{flex} = 0,5685×ln(T) + 1,1017	2.6412	0.9999

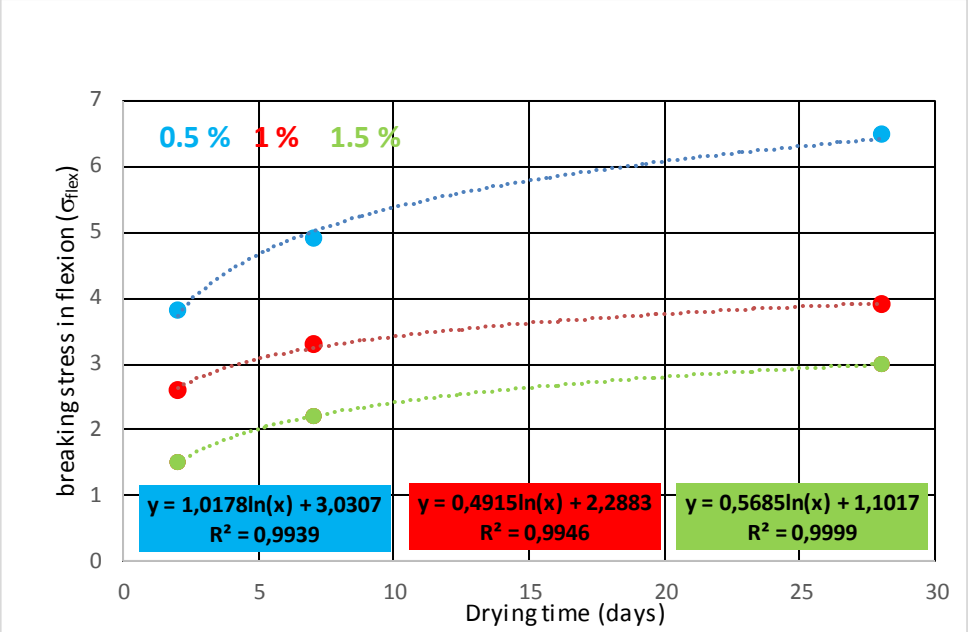


Fig.4.9 Bending failure stresses values for a drying time for different fiber percentages (0.5, 1 and 1.5 %)

Second, the drying time (factor T) is fixed, and the evolution of the bending stress (σ_{flex}) as a function of the percentage of fibers is investigated (P). The different (σ_{flex}) curves as a function of (P) are shown in (**Fig.4.10**). The models are generated by interpolating the outcomes of the experimental testing, as in the preceding case. The curves and polynomial models with a very good correlation are depicted in Table **4.2** were plotted using an Excel spreadsheet. These models enable us to predict the bending failure stresses (σ_{flex}) for any selected percentage of fibers (P) in the range of [0.5 days to 1.5 percent]. The bending stresses values for a percentage of fibers of 0.75 % and varied drying times (2 days, 7 days, and 28 days) are included in Table **4.2**.

Table 4.2 Bending stresses at a percentage of fibers of 0.75 % and for different drying times (2 days, 7 days, and 28 days)

Drying times (days)	Model	σ_{flex} (MPa)	Coefficient of correlation R^2
2	$\sigma_{flex} = 0.2\times P^2 - 2.7\times P + 5.1$	5.7869	1
7	$\sigma_{flex} = -P^2 + 0.1\times P + 4.3$	3.6193	1
28	$\sigma_{flex} = 3.36\times P^2 - 10.2\times P + 10.74$	2.6412	1

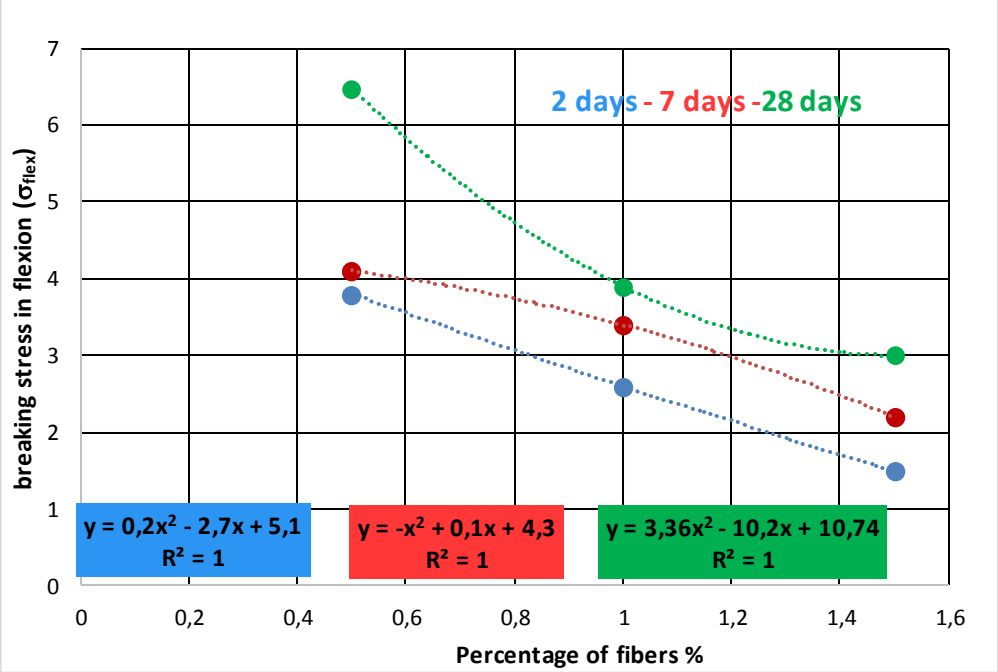


Fig. 4.10 Bending stresses values versus percentage of fibers for varying drying times (2 days, 7 days, and 28 days)

4.2.2 Compression behavior (uni-factorial models)

We conducted a campaign of compression tests on normalized specimens as for the bending case. The findings will be utilized to investigate the mechanical compression behavior of elaborated bio-composites. Table **4.3** displays the results. The percentage of fibers (factor P) is kept constant, and the evolution of compression breaking stresses (σ_{comp}) as a function of drying time (T) is investigated. The varied (σ_{comp}) curves as a function of (T) are included in (**Fig.4.11**). The models are created by approximating the experimental test data. We plotted the curves (**Fig.4.11**) and obtained logarithmic models with a high correlation, as shown in Table 4.3, using an Excel spreadsheet. These models enable us to calculate the compression failure stresses (σ_{flex}) for every value of drying time in the range of [2 days to 28 days]. The compression values for a 15-day drying time and different fiber percentages (0.5, 1, and 1.5 %) are seen in Table **4.3**.

Table 4.3 Compression failure stresses at a drying time of 15 days and for different percentages (P)

Fiber percentages (%)	Model	σ_{comp} (MPa)	Coefficient of correlation R^2
0.5	$\sigma_{comp} = 8.4704 \times \ln(T) + 10.407$	33.3452	0.9950
1	$\sigma_{comp} = 2.9877 \times \ln(T) + 10.586$	18.6768	0.9957
1.5	$\sigma_{comp} = 4.8461 \times \ln(T) + 2.5583$	15.6775	0.9991

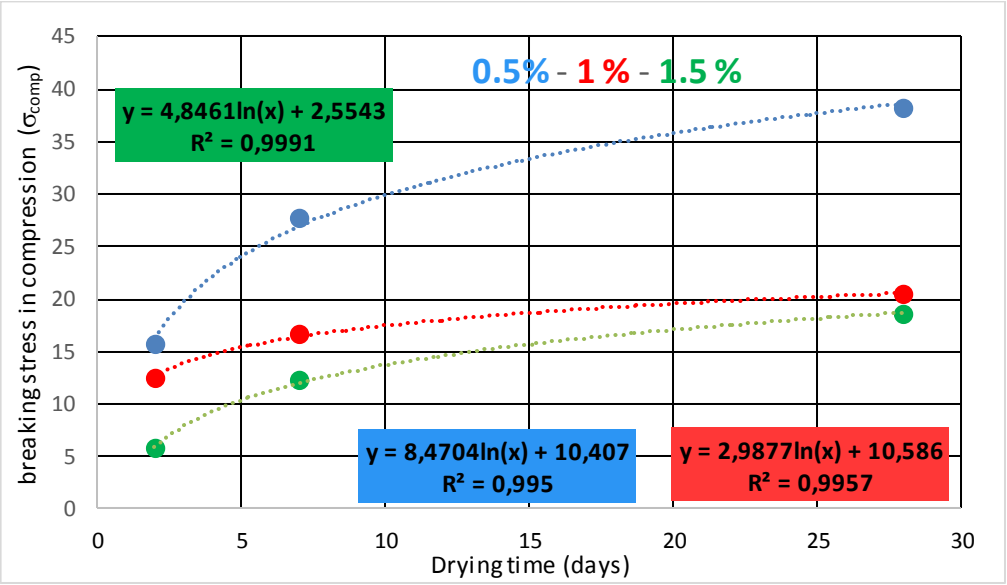


Fig. 4.11 Compression failure stresses values for a drying time for different fiber percentages (0.5, 1 and 1.5 %)

In the second step, the drying time (factor T) is fixed, and the evolution of the compression stresses (σ_{comp}) as a function of the percentage of fibers is investigated (P). The different (σ_{comp}) curves as a function of (P) are shown in (**Fig.4.12**). The models are created by interpolating the outcomes of the experimental testing. As in the preceding case, the polynomial models are presented in Table 4.4. The curves are plotted using an Excel spreadsheet as shown in (**Fig.4.12**). These models enable us to predict the compression failure stresses (σ_{comp}) for any selected percentage of fibers (P) in the range of [2 to 28 days]. As an example, the compression failure stresses values for a percentage of fibers of 0.75 % and varied drying times (2 days, 7 days, and 28 days) are computed and included in Table **4.4.**

Table 4.4 Compression failure stresses at a percentage of fibers of 0.75 % and for different drying times (2 days, 7 days, and 28 days)

Drying times (days)	Model	σ_{flex} (MPa)	Coefficient of correlation R^2
2	$\sigma_{comp} = y = -6.8\times P^2 + 3.6\times P + 15.7$	14.575	1
7	$\sigma_{comp} = 13.2\times P^2 - 42\times P + 45.5$	21.1775	1
28	$\sigma_{comp} = 32\times P^2 - 83.\times P + 72$	27.3	1

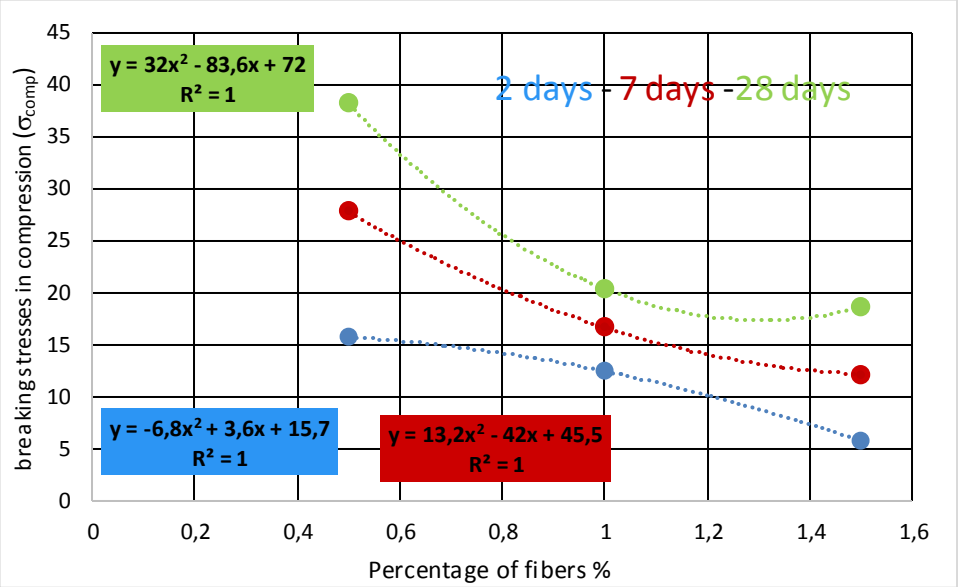


Fig. 4.12 Compression stresses values versus percentage of fibers for varying drying times (2 days, 7 days, and 28 days)

Part III: Statically analysis of mechanical behavior in bending and in compression of elaborated bio-composites

4.3.1 Introduction

The main objective of this research is to develop predictive mathematical models of mechanical behavior (bending and compression) and to better understand the mechanisms that govern this specific behavior. All the planned experiments and the statistical analysis of the results are performed using the Design-Expert software (version 10), which is a specialized software for the planning and analysis of experiments. **Response surface methodology (RSM) was used to study and statistically analyze the mechanical behavior of processed bio-sourced composites. In particular, this statistical method permits to build mathematical models involving different parameters that affect the bending and compression responses of these composites. It is based essentially on the results obtained by experiments**.

4.3.2 Implementation of experimental designs

4.3.2.1 Responses

The chosen responses are the breaking stress in flexion (σ_{flex}) and the breaking stress in compression (σ_{comp}).

4.3.2.2 Factors influencing

Several factors influence the stress at breaking in bending noted (σ_{flex}) and the stress at breaking in compression (σ_{comp}). The previous experiments carried out and described in chapter III have revealed that these two responses obtained by 3-point bending and compression tests are influenced by the percentage of *Agava Americana* fibers (X_1) and by the drying time of the specimens (X_2). The levels: minimum, average and maximum, of each variable are designated respectively in the form coded by: -1, 0 and +1. The Table 4.5 below outlines the different factors considered and the range of variation allowed for each factor (study area).

Table 4.5 Levels of the factors used by the centered composite design methodology

Factors	Coded variable symbols	Low level (-1)	Average level (0)	High level (+1)
Drying time (days)	X_1	2	15	28
Percentage of fibers (%)	X_2	0.5	1	1.5

4.3.2.3 Coded mathematical model

The coded quadratic mathematical model that describes the response variations as a function of the different coded factors is expressed as:

$$y = a_0 + \sum_{i=1}^{n} a_i X_i + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} a_{ij} X_i X_J + \sum_{i}^{N} a_{ii} X_i^2 \qquad (4.6)$$

where:

y is the response.
X_i represents the level of factor i.
X_j represents the level of factor j.
a_0 is the constant coefficient of the model.
a_i is the coefficient of factor i, (the effect of factor i).
a_{ij}_is the coefficient of the term X_iX_j, (the interaction between factors i and j).
a_{ii}_is the coefficient of the quadratic terms X_iX_i

n is the number of experiments.

In this study two models have been considered, the first one polynomial quadratic and the second one quadratic logarithmic. This choice can be justified by the varying responses according to the factors involved in the uni-factorial models already discussed in the previous section.

4.3.2.4 Design of experiments

To realize this part of the work, we have opted to implement the central composite design, which is a second-degree design (**Fig.4.13**).

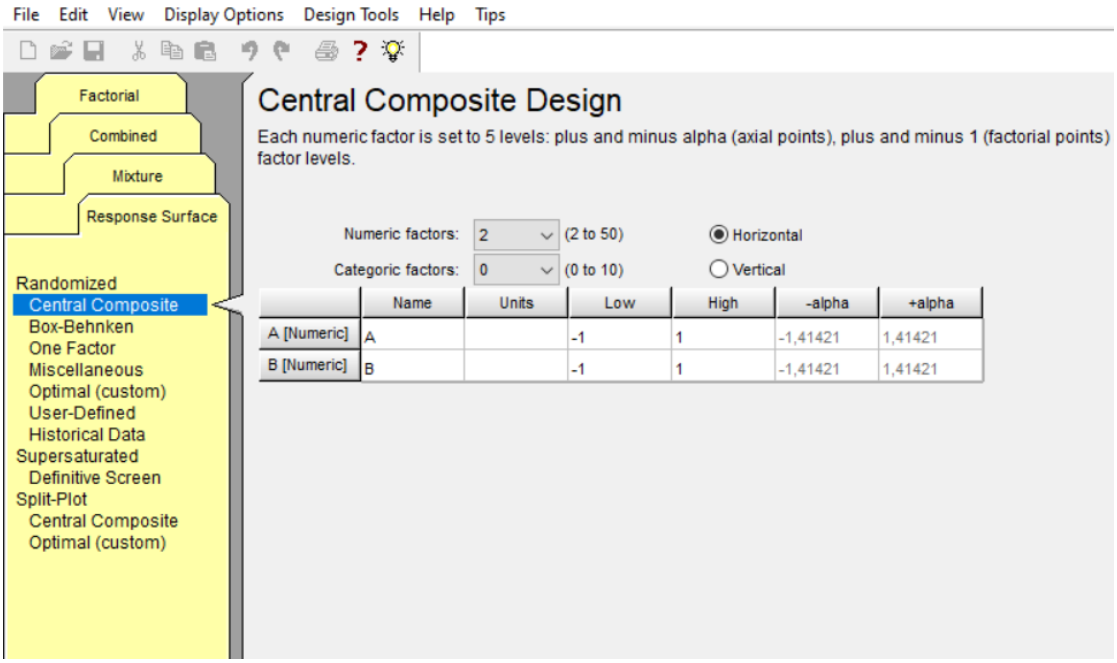


Fig. 4.13 Central composite design considered in this study

4.3.2.5 Central composite design

A central composite design is a type of experimental design used in response surface methods to create a second-order (quadratic) 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. 4.13**). A set of 11 experiments were performed (three points in the center of the model). The analysis was performed to evaluate the response functions. The quadratic model shown in the previous Equation **4.6** can predict the stresses at failure in bending and compression. The tests have been numbered from 1 to 11, according to the following relationship:

$$N = 2^f + 2f + C_p \qquad (4.7)$$

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 gives the different combinations of the basic factors (T) and (P). Table **4.6** shows the planning matrix based on the coded factors. The one based on decoded ones could be seen in Table **4.7.**

Table 4.6 Composite central coded matrix: factors (X_1) and (X_2), responses (Y_1) and (Y_2) obtained experimentally

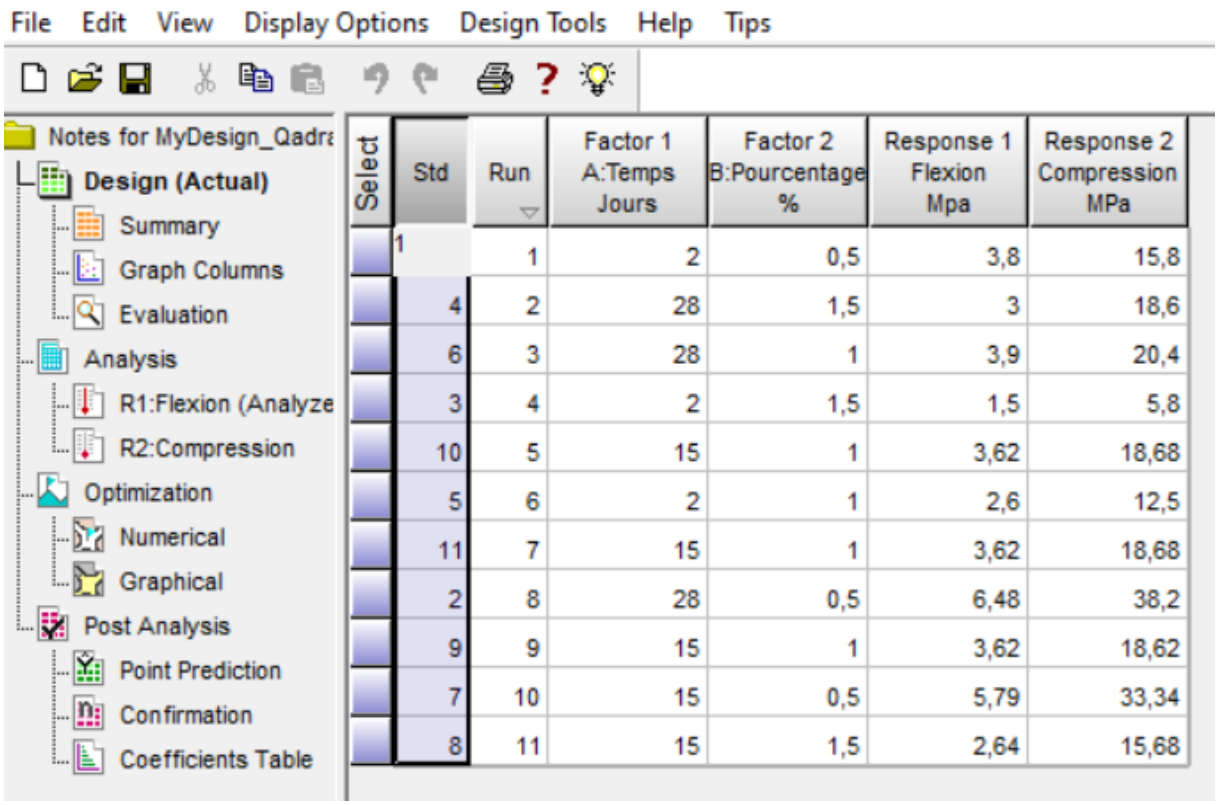
	Factor 1 (X_1)	Factor 2 (X_2)	Response 1 (Y_1)	Response 2 (Y_2)
N°	Drying times (days)	Percentage of fibers (%)	Bending break stress (MPa)	Compression break stress (MPa)
1	-1	-1	3.80	15.80
2	-1	0	2.60	12.50
3	-1	+1	1.50	5.80
4	0	-1	5.79	33.34
5*	0	0	3.62	18.68
6	0	+1	2.64	15.68
7	+1	-1	6.48	38.20
8	+1	0	3.90	20.40
9	+1	+1	3.00	18.60
10*	0	0	3.62	18.68
11*	0	0	3.62	18.68

*Three points in the center of the model

Table 4.7 Composite central decoded matrix: factors (T and P), responses (σ_{flex} and σ_{comp})

	Factor 1 (T)	Factor 2 (P)	Response 1	Response 2
N°	Drying times (days)	Percentage of fibers (%)	Bending break stress σ_{flex} (MPa)	Compression break stress σ_{comp} (MPa)
1	2	0.5	3.80	15.80
2	2	1	2.60	12.50
3	2	1.5	1.50	5.80
4	15	0.5	5.79	33.34
5*	15	1	3.62	18.68
6	15	1.5	2.64	15.68
7	28	0.5	6.48	38.20
8	28	1	3.90	20.40
9	28	1.5	3.00	18.60
10*	14	1	3.62	18.68
11*	14	1	3.62	18.68

*Three points in the center of the model



Select	Std	Run	Factor 1 A:Temps Jours	Factor 2 B:Pourcentage %	Response 1 Flexion Mpa	Response 2 Compression MPa
	1	1	2	0,5	3,8	15,8
	4	2	28	1,5	3	18,6
	6	3	28	1	3,9	20,4
	3	4	2	1,5	1,5	5,8
	10	5	15	1	3,62	18,68
	5	6	2	1	2,6	12,5
	11	7	15	1	3,62	18,68
	2	8	28	0,5	6,48	38,2
	9	9	15	1	3,62	18,62
	7	10	15	0,5	5,79	33,34
	8	11	15	1,5	2,64	15,68

Fig. 4.14 Central composite matrix used in this investigation

The data in this matrix will be statistically processed to estimate the coefficients of the mathematical model.

4.3.3 Bending behavior (polynomial quadratic model)

As a first part the analysis was performed to estimate the response function (bending stress at break) as a function of the factors (drying time and fiber percentage) through a quadratic polynomial model (Equation 4.2).

4.3.3.1 Statistical results and interpretation

4.3.3.1.1 Descriptive analysis

Figs. 4.15 and **4.17** describe the variation of the response (bending failure stress) as a function of the two factors studied (drying time (T) and percentage of fibers (P)) and the correlation between them expressed by the corresponding coefficient (R).

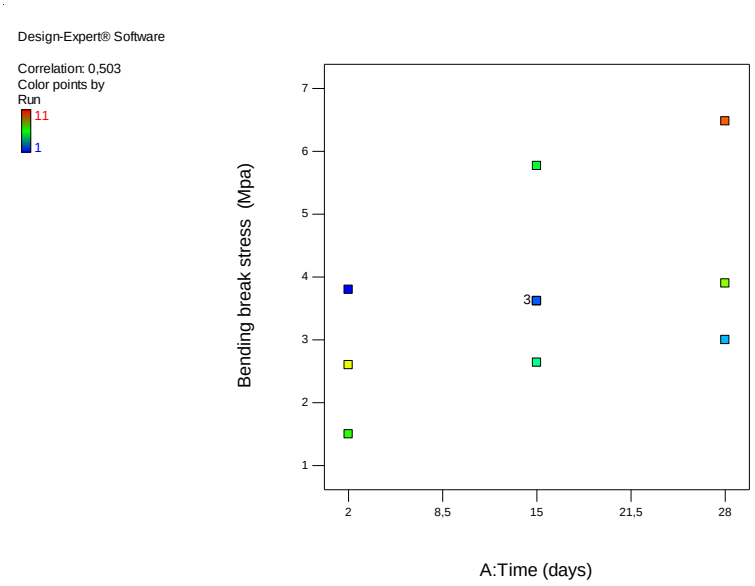


Fig. 4.15 Correlation between the response (bending stress) and the factor (drying time)

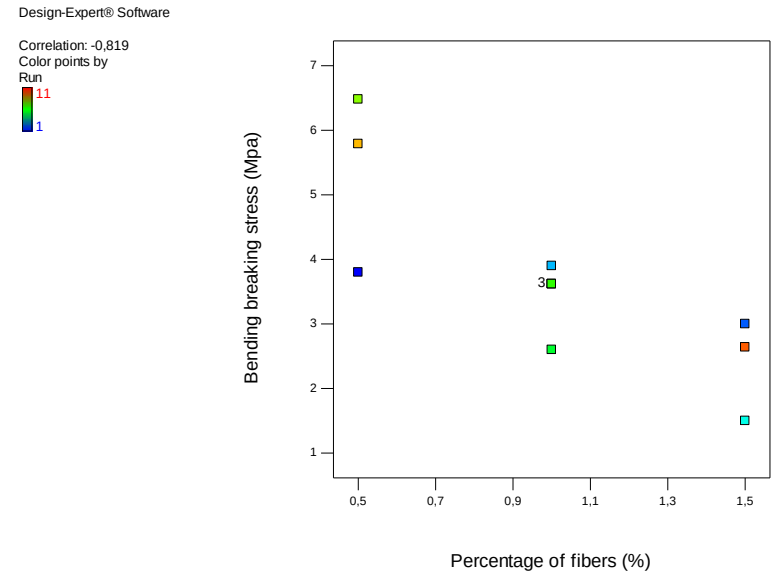


Fig. 4.16 Correlation between the response (bending stress) and the factor (percentage of fibers)

According to this descriptive analysis, it can be observed that the drying time factor (T) has a positive effect on the bending stress response (R=+0.503), whereas the percentage of fibers factor (P) has a negative effect on this response (R=-0.819). The results are summarized in the following Table 4.8.

Table 4.8 Correlation between the response and the two factors considered separately

Response	Factor	Correlation	Interpretation
bending stress (σ_{flex})	drying time (T)	0.503	a positive linear relationship
bending stress (σ_{flex})	percentage of fibers (P)	-0.819	a negative linear relationship

The Design Expert software allows to plot the bending stress response predicted by the model versus the actual experimental values.

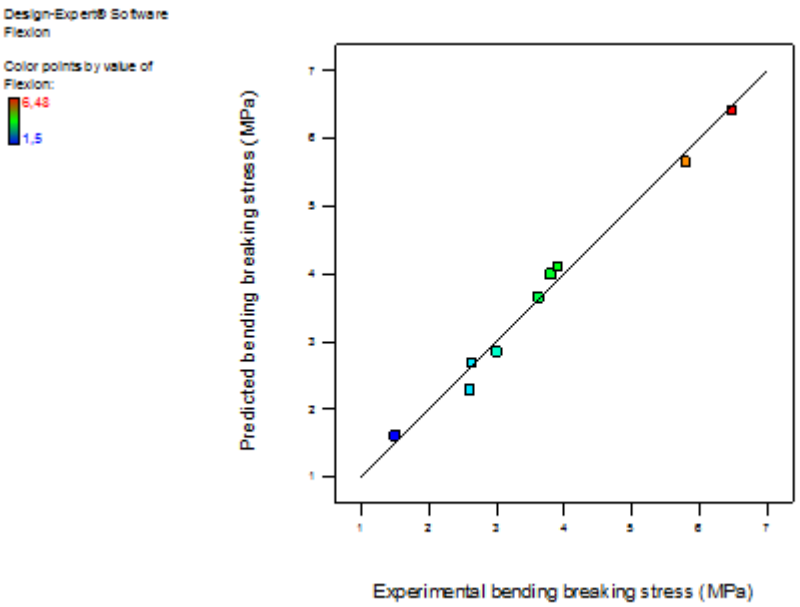


Fig.4.17 Curve of predicted versus experimental values of bending stress at break.

The data in the graph, which shows the actual versus predicted response values (**Fig.4.17**), should be distributed evenly and by a 45 °line **[1]**. The actual response values are uniformly dispersed with regard to a 45° line, as one can see. ANOVA was used to check the significance and form of the model in question.

4.3.3.2 Analysis of variance (ANOVA)

We have used the (P-value) and (F-value) values as statistical indicators to judge which terms in the model are significant. According to the ANOVA Table provided by Design Expert software. The Model F-value of 80.00 less than 0.05 implies that the model is significant. In this case, A, B, AB, A^2, B^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The "Pred R-Squared" of 0.8718 is in reasonable agreement with the "Adj R-Squared" of 0.9753; i.e. the difference is less than 0.2. "Adeq Precision" measures the signal to noise ratio. A ratio greater than

4 is desirable. Your ratio of 29.394 indicates an adequate signal. This model can be used to navigate the design space. Furthermore the value of the coefficient of determination R-Squared (0.9877) is good and Adj R-Squared (0.9753), which indicates that the model is relatively well fitted. Pred R-Squared (0.8718) represents the response values predicted by the model. The difference between Pred R-Squared and Adj R-Squared should always be in the range of 0 to 0.200 for a good model. In this case, the difference between the two is 0.1035 so the model chosen is adequate for the proposed study of the bending behavior of the elaborated composites by 3-point bending tests. The coefficient of variation "CV" which represents the ratio of the standard error of the estimate to the mean value of the observed response is a measure of the reproducibility of the model. Generally a model can be considered reasonable if its CV does not exceed 15% **[9]**. In this study, the value of the coefficient of variation obtained of 6 % indicating high accuracy and reliability of the experiments. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 29.394 indicates an adequate signal. This model can be used to navigate the design space.

Table 4.9 ANOVA for response (bending breaking stress) quadratic polynomial model

Analysis of variance table [Partial sum of squares]					
	Sum of	Mean	F	p-value	
Source	Squares	Square	Value	Prob > F	
Model	19.58	3.92	80.00	< 0.0001	significant
A-Temps	5.01	5.01	102.24	0.0002	
B-Pourcentage	13.29	13.29	271.49	< 0.0001	
AB	0.35	0.35	7.11	0.0445	
A^2	0.51	0.51	10.43	0.0232	
B^2	0.67	0.67	13.78	0.0138	
R-Squared	0.9877				
Adj R-Squared	0.9753				
Pred R-Squared	0.8718				
Adeq Precision	29.394				
C.V. %	6.00				

4.3.3.3 Mathematical Model Determination

The models generated by the Design Expert in coded and decoded form are presented below:

Coded model:

$$Y_1 = 3.65 + 0.91 \times X_1 - 1.48 \times X - 0.3 \times X_1 X_2 - 0.44 \times X_1^2 + 0.51 \times X_2^2 \quad (4.8)$$

Decoded model:

$$\sigma_{flex} = 6.33942 + 0.1944 \times T - 6.37976 \times P - 0.045385 \times T \times P - 2.62535 \times 10^{-3} \times T^2 + 2.04526 \times P^2 \quad (4.9)$$

The statistical analysis conducted by Design Expert software revealed that all factors are significant (T^2, P^2, T, P and T×P). The details of this analysis were presented in the Table 4.9. We obtain the final expression of the model with a exellent quality fit.

4.3.3.3.1 Validation of the Model

The validation of the model is one of the fundamental stages of the design of experiments. It consists in comparing the theoretical result of an experiment calculated by the model, with the real result of a test. If the real response is very close to the predicted response, the model is validated. If the real response is far from the predicted response, the model is not approved.

4.3.3.3.2 Primary validation of the model

Primary validation of the model consists of checking that the calculated and measured responses are correlated (Table **4.10**). This table allows us to judge more precisely the quality of the adjustment carried out. The comparison between the columns Y_{exp} (measured responses) and Y_{cal} (responses predicted by the model) confirms that the fit is of good quality.

Table 4.10 Comparison between the experimental values and the values computed using the model

	Factor 1 (T)	Factor 2 (P)	Response (Experimental)	Response (Predicted)
N°	Drying times (days)	Percentage of fibers (%)	σ_{flex} (MPa)	σ_{flex} (MPa)
1	2	0.5	3.8	3.9938
2	2	1	2.6	2.2925
3	2	1.5	1.5	1.6137
4	15	0.5	5.79	5.6458
5	15	1	3.62	3.6494
6	15	1.5	2.64	2.6758
7	28	0.5	3.8	3.9938
8	28	1	2.6	2.2925
9	28	1.5	1.5	1.6138
10	15	1	3.62	3.6494
11	15	1	3.62	3.6494

4.3.3.3.3 Graphical representation of the theoretical responses versus the measured responses

This correlation can also be illustrated by plotting the measured responses against the calculated responses **[10]**, as illustrated in Figure **4.18**, which demonstrates a strong correlation between them, with a correlation coefficient of R^2=0.9878.

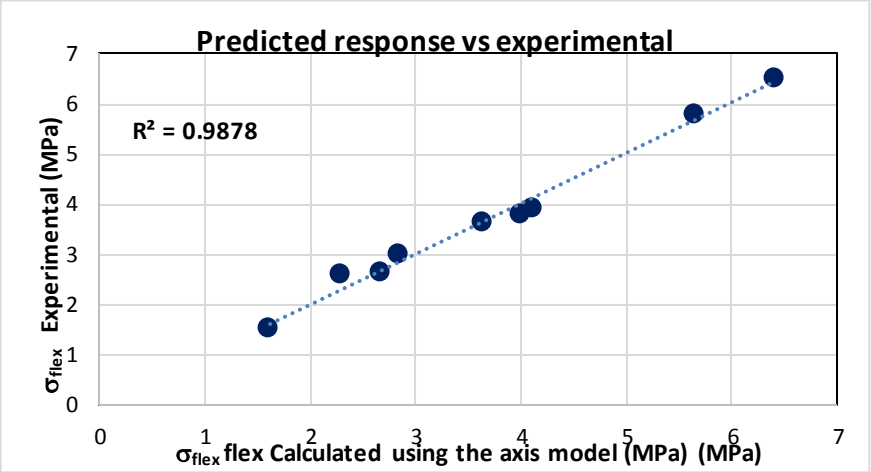


Fig.4.18 Measured response versus calculated response

4.3.3.3.4 Residual analysis

An analysis of the residuals (see Table **4.11**) can achieve the appreciation of the quality of the second order model.The average value of the residuals according to Equation 4.10 is of the order of 0.1827 % and is close to 0 **[11]**.

Table 4.11 Computation of the residues

N°	Response (Experimental)	Response (Computed)	Residue
1	3.8	3.9938	-0.1938
2	2.6	2.2925	0.3075
3	1.5	1.6137	-0.1137
4	5.79	5.6458	0.1442
5	3.62	3.6494	-0.0294
6	2.64	2.6758	-0.0358
7	3.8	3.9938	-0.1938
8	2.6	2.2925	0.3075
9	1.5	1.6138	-0.1138
10	3.62	3.6494	-0.0294
11	3.62	3.6494	-0.0294
		Sum	0.0201
		Average residuals	0.00182727

4.3.3.3.5 Effects of process variables

The software Design Expert may generate 3D response surface plots for the bending break stress of the examined bio-composites based on the predictive quadratic model (Equation **4.9**) in order to better understand the effect of independent variables. (**Fig. 4.19**) displays the 3-D surface plot for the bending stress-to-break response as a function of drying time (T) and fiber percentage (P) inputs. It can be noticed that the bending stress at break increases with increasing drying time and decreases with increasing fiber percentage.

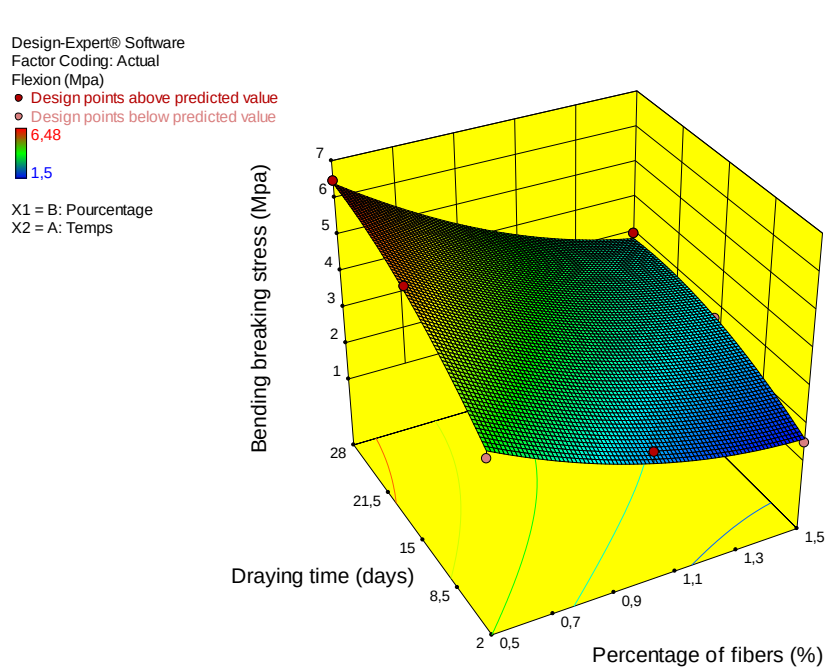


Fig.4.19 Response surface plots for the combined effects of factors (T) and (P) (quadratic model)

4.3.3.3.6 Comparison between the experimental values and those predicted by the model

The values of the bending break stress as a function of drying time (T) and fiber percentage (P), calculated from the model (Equation 4.9) were compared with the experimental values obtained by the the 3-point bending tests (Chapter 3). The results obtained are reported in Table 4.12. It can be seen that the real bending break stress values are very close to the calculated ones. The calculated and measured responses are correlated as shown in **Fig. 4.20**, which allows to validate the model.

Table 4.12 Bending break stress: experimental values and computed using the model

	Factor 1 (T)	Factor 2 (P)	Response (Experimental)	Response (Predicted)
N°	Drying times (days)	Percentage of fibers (%)	σ_{flex} (MPa)	σ_{flex} (MPa)
1	2	0.5	3.8	3.99377
2	2	1	2.6	2.292451
3	2	1.5	1.5	1.613762
4	7	0.5	4.1	4.734169
5	7	1	3.4	2.91939
6	7	1.5	2.2	2.127241
7	15	0.5	5.79	5.645771
8	15	1	3.62	3.649456
9	15	1.5	2.64	2.675771
10	28	0.5	6.48	6.410405
11	28	1	3.9	4.119094
12	28	1.5	3	2.850413

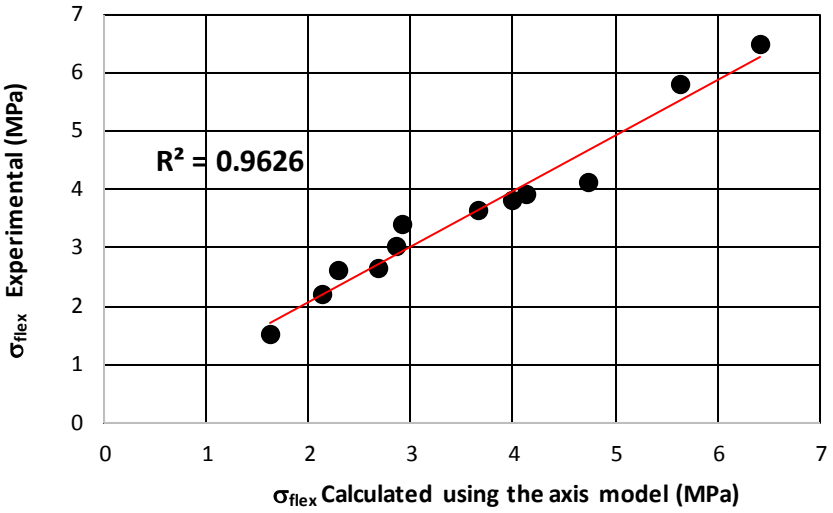


Fig.4.20 Fitting curve of the stress at break in experimental bending with respect to the one calculated by the model

4.3.4 Compression behavior (polynomial linear model)

In this section, the response function (compression stress at break) was estimated as a function of the same factors (drying time and percentage of fibers) using a linear polynomial model. ANOVA analysis excluded all second-order terms (A^2, B^2) in addition to the linear interaction term between the two factors ($A \times B$), which forced us to choose a linear model.

4.3.4.1 Statistical results and interpretation

Fig. 4.21 and **4.22** show how the response (compression stress at break) varies as a function of the two examined factors (drying time (T) and percentage of fibers (P), and the correlation between them expressed by the corresponding coefficient (R).

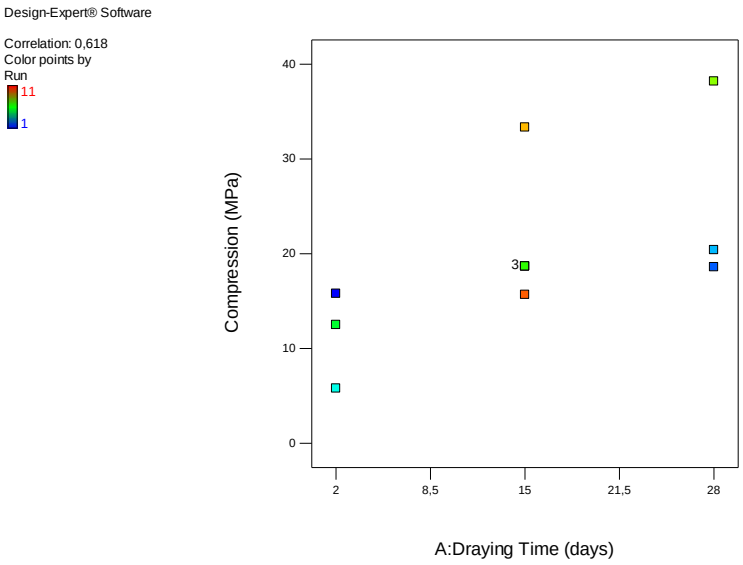


Fig. 4.21 Correlation between the response (compression break stress) and the factor (drying time)

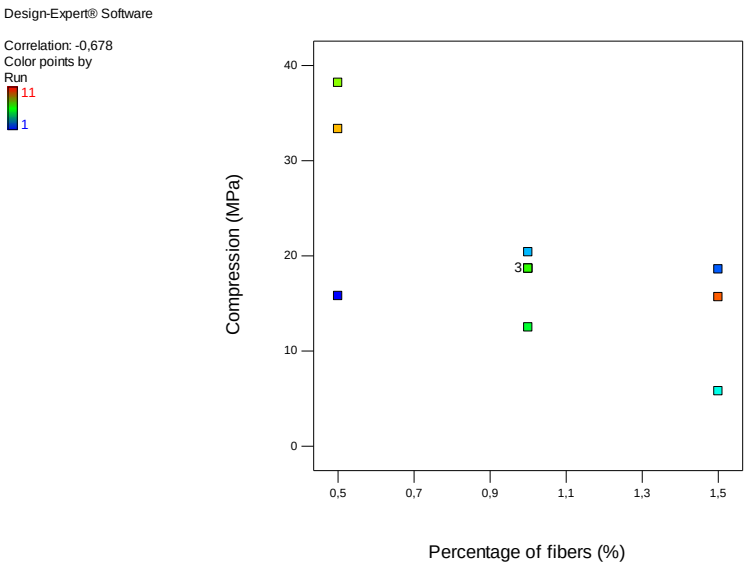


Fig. 4.22 Correlation between the response (bending stress) and the factor (percentage of fibers)

The drying time factor (T) has a favorable effect on the compression stress response (R=+0.618), however the percentage of fibers factor (P) has a negative effect on this response (R=- 0.678), as per this descriptive analysis. The results are given in Table 4.13 below.

Table 4.13 Correlation between the response and the two factors considered separately

Response	Factor	Correlation	Interpretation
Compression stress (σ_{comp})	drying time (T)	+0.618	a positive linear relationship
	percentage of fibers (P)	-0.678	a negative linear relationship

The Design Expert software enables us to compare and plot the response (stress at break in compression) predicted by the model to the experimental one.

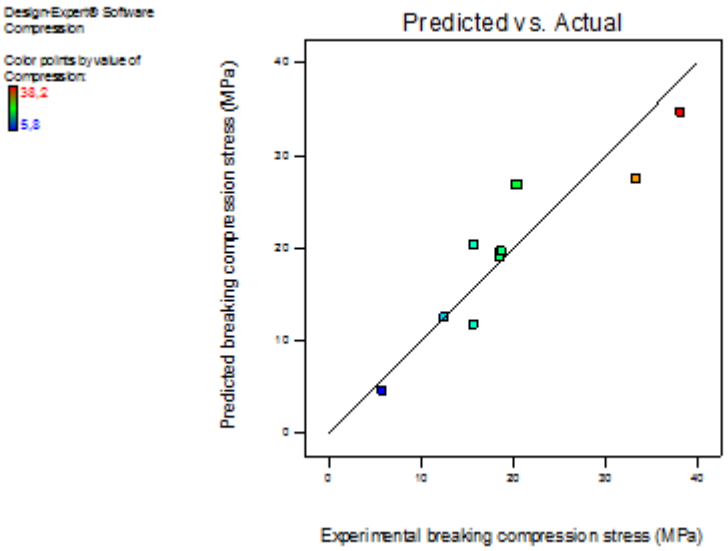


Fig.4.23 Curve of predicted versus experimental values of bending stress at break.

The data in the graph (**Fig. 4.23**) that depicts actual vs predicted response values should be distributed equally and by a 45-degree line **[1]**. As can be seen, the actual response values are equally spread in relation to a 45° line. The significance and appropriateness of the model in inquiry were checked using ANOVA.

4.3.4.2 Analysis of variance (ANOVA)

We have used the (P-value) and (F-value) values as statistical indicators to judge which terms in the model are significant. According to the ANOVA Table provided by Design Expert software. The Model F-value of 21.34 implies that the model is significant. In this case, A, B, are significant model terms. AB, A^2, B^2 terms are not significant. Values greater than 0.1000 indicate the model terms are not significant. The "Pred R-Squared" of 0.6648 is in reasonable agreement with the "Adj R-Squared" of 0.8027; i.e. the difference is less than 0.2. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 14.30 indicates an adequate signal. This model can be used to navigate the design space. Furthermore the value of the coefficient of determination R-Squared (0.8422) is good and Adj R-Squared (0.8027), which indicates that the model is relatively well fitted. Pred R-Squared (0.6648) represents the response values predicted by the model. The difference between Pred R-Squared and Adj R-Squared should always be in the range of 0 to 0.200 for a good model. In this case, the difference between the two is 0.1379 so the model chosen is adequate for the proposed study of the bending behavior of the elaborated composites by 3-point bending tests.

Table 4.14 ANOVA for response (compression breaking stress) linear polynomial model

Analysis of variance table [Partial sum of squares]				
Source	Sum of Squares	Mean Square	F Value	p-value Prob > F
Model	681.85	340.93	21.34	0.0006
A-Temps	309.60	309.60	19.38	0.0023
B-Pourcentage	372.25	372.25	23.30	0.0013
R-Squared	0.8422			
Adj R-Squared	0.8027			
Pred R-Squared	0.6648			
Adeq Precision	14.30			

4.3.4.3 Mathematical model determination

The models generated by the Design Expert in coded and decoded form are presented below:

Coded model:

$$Y_1 = 19.66 + 7.18 \times X_1 - 7.88 \times X_2 \quad (4.11)$$

Decoded model:

$$\sigma_{comp} = 27.12851 + 0.55256 \times T - 15.75333 \times P \quad (4.12)$$

Only (T) and (P) factors are significant, according to the statistical analysis performed by Design Expert software. Table 4.15 summarizes the findings of this investigation. With an acceptable quality fit, we get the model's final expression.

4.3.4.4 Validation of the model

Model validation consists of ensuring that the calculated and measured responses are related (Table 4.15). This table shows the good quality of the fit after a comparison of the columns Y_{exp} (measured responses) and Y_{cal} (responses predicted by the model).

Table 4.15 Comparison between the experimental values and the values computed using the linear model

	Factor 1 (T)	Factor 2 (P)	Response (Experimental)	Response (Predicted)
N°	Drying times (days)	Percentage of fibers (%)	σ_{comp} (MPa)	σ_{comp} (MPa)
1	2	0.5	15.8	20.3569
2	2	1	12.5	12.4803
3	2	1.5	5.8	4.6036
4	15	0.5	33.34	27.5402
5	15	1	18.68	19.6635
6	15	1.5	15.68	11.7869
7	28	0.5	38.2	34.7235
8	28	1	20.4	26.8468
9	28	1.5	18.6	18.9701
10	15	1	18.68	19.6635
11	15	1	18.68	19.6635

4.3.4.5 Graphical representation of the theoretical responses versus the measured responses

This correlation can also be seen by graphing the measured responses against the estimated responses **[10],** as shown in (**Fig. 4.24**), which shows a good correlation between them with an R^2=0.8423 correlation coefficient.

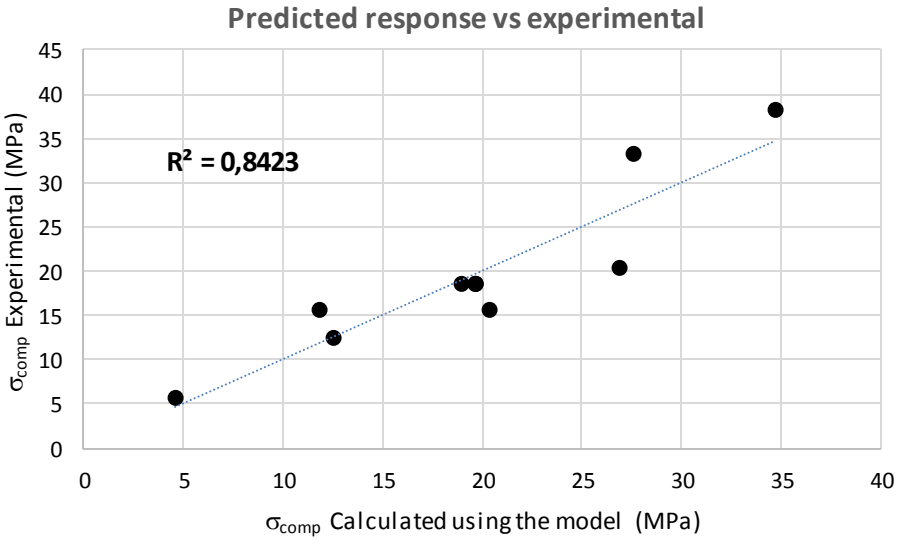


Fig.4.24 Calculated response versus measured response

4.3.4.6 Residual analysis

The quality of the second order model can be tested by glancing at the residuals (see Table 4.11) and (**Fig. 4.19**) below. According to Equation 4.10, the average residual value is on the order of 0.556364 percent and is close to 0 **[11].**

Table 4.16 Computation of the residues

N°	Response (Experimental)	Response (Computed)	Residue
1	15.8	20.3569	-4.5569
2	12.5	12.4803	0.0197
3	5.8	4.6036	1.1964
4	33.34	27.5402	5.7998
5	18.68	19.6635	-0.9835
6	15.68	11.7869	3.8931
7	38.2	34.7235	3.4765
8	20.4	26.8468	-6.4468
9	18.6	18.9701	-0.3701
10	18.68	19.6635	-0.9835
11	18.68	19.6635	-0.9835
		Sum	0.0612
		Average residuals	0.00556364

4.3.4.7 Effects of process variables

Fig. 4.25 depict 3D response surface plots based on the predictive linear model (Equation **4.12**) for the compression break stress of bio-composites in an attempt to better understand the effect of independent variables The 3-D surface plot of the compression stress-to-break response as a function of drying time (T) and fiber percentage (P) data is shown in **Fig. 4.25**. It can be seen that the compression stress at break increases with increasing drying time and decreases with increasing fiber percentage.

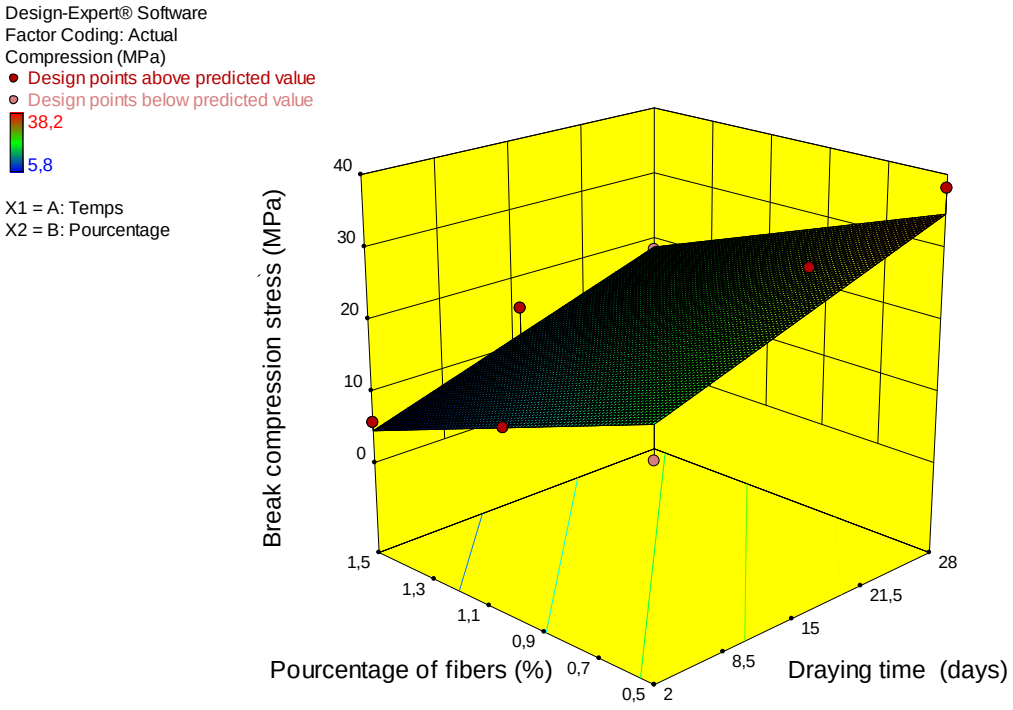


Fig.4.25 Response surface plots for the combined effects of drying time (T) and percentage of fiber (P) (Linear model)

4.3.4.8 Comparison between the experimental values and those predicted by the model

The values of the bending break stress as a function of drying time (T) and fiber percentage (P), calculated from the model (Equation 4.9) were compared with the experimental values obtained by the the 3-point bending tests (Chapter 3). The results obtained are reported in Table 4.12. It can be seen that the real bending break stress values are very close to the calculated ones. The calculated and measured responses are correlated as shown in **Fig. 4.20**, which allows to validate the model.

Table 4.17 Bending break stress**:** experimental values and computed using the model

	Factor 1 (T)	Factor 2 (P)	Response (Experimental)	Response (Predicted)
N°	Drying times (days)	Percentage of fibers (%)	σ_{flex} (MPa)	σ_{flex} (MPa)
1	2	0.5	15.8	20.3569
2	2	1	12.5	12.4803
3	2	1.5	5.8	4.6036
4	7	0.5	27.8	23.1197
5	7	1	16.7	15.2431
6	7	1.5	12.2	7.3664
7	15	0.5	33.34	27.5402
8	15	1	18.68	19.6635
9	15	1.5	15.68	11.7869
10	28	0.5	38.2	34.7235
11	28	1	20.4	26.8468
12	28	1.5	18.6	18.9701

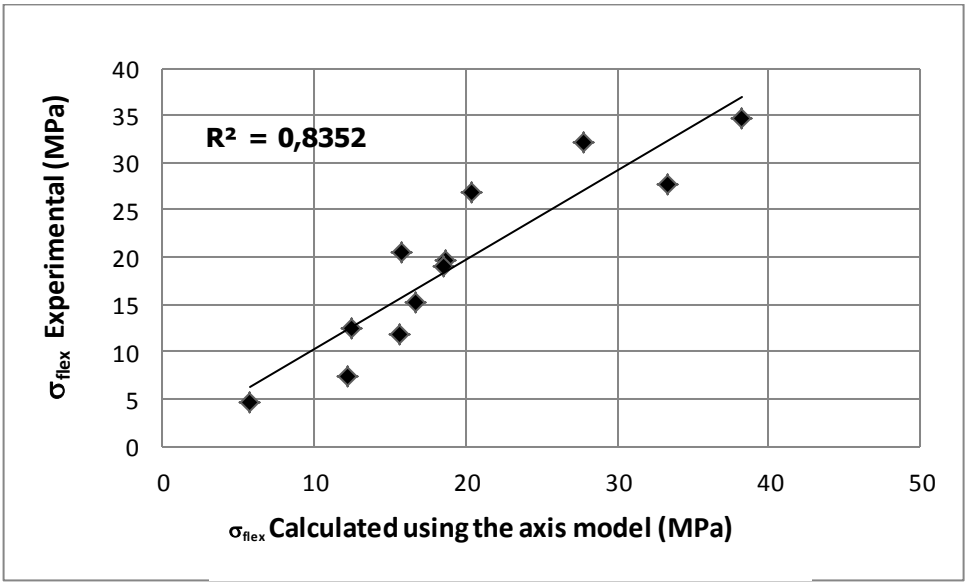


Fig.4.26 Fitting curve of the stress at break in experimental bending with respect to the one calculated by the model

Bibliographical references

[1] Iman Mezarguia, Ikram Slimani, Etude et modélisation mathématique de l'inhibition de la corrosion de l'acier au Carbone dans un milieu acide par l'extrait dichlorométhane de Chamomilla recutita, Mémoire de Master, Université Larbi Tébessi-Tébessa, 2021.

[2] Amina Belgharbi , Saadia Abib , Utilisation des plans expérience pour la formulation des pates cimentaires auto-plaçants, Mémoire de Master, Université de Bouira, 2016.

[3] Redhouane Lombarkia , Etude, simulation et expérimentation du comportement mécanique des couvercles des boites d'emballage métalliques pour produits chimiques soutenu, Mémoire de Magister, Université de Batna, 2012.

[4] François Rabier, Modélisation par la méthode des plans d'expériences du comportement dynamique d'un module IGBT utilisé en traction ferroviaire, Thèse de doctorat, Institut National Polytechnique de Toulouse,2007.

[5] Nabil Maza, Houssam Eddine Redjouh,Carractérisation d'un béton autoplaçant par modélisation, Mémoire de Master, Université Mohamed El-Baachir El-Ibrahimi Bordj Bouaririd j,2020

[6] Mohamed Ayad, Yassine Barka, Modélisation des bétons ordinaires par des plans d'expériences, Mémoire de Master ,Université Abou Bekr Belkaid - Tlemcen ,2013.

[7] Nassima Bouzar, Optimisation des paramètres d'oxydation catalytique de cyclohexène par les polyoxométalates à l'aide de la méthodologie de surface de réponse, Mémoire de Master ,Université Abou Bekr Belkaid - Tlemcen ,2019.

[8] Remidini Hadjer,Contribution à l'étude des propriétés mécaniques d'un béton de fibres, Université Mohamed khider –Biskra,2020.

[9] Rossi,. Applied Biostatistic for the Health Sciences. Wiley, New Jersey. Simsek,0, Özdemir, , Beker, 2013. Processoptimization for arsenicadsorption onto natural zeolite in corporating metal oxides by responsesurface methodology. Water Air SoilPollut. 224, (2010).

[10] Obi-Egbedi, Obot, El-Khaiary, Umoren and Ebenso, Computational Simulation and Statistical Analysis on the Relationship Between Corrosion Inhibition Efficiency and Molecular Structure of Some Phenanthroline Derivatives on Mild Steel Surface, Int. Electrochem. Sci, Vol.6, pp. 5649 – 5675, (2011).

[11] Yahiaoui, Les plans d'expériences Initiation à la construction et l'analyse des plans factoriels complets à deux niveaux et du plan composite centré, Polycopie de Cours, Université A. MIRA Bejaia, (2015).

Conclusion

• Conclusion

The principal purpose of this research is to contribute to the investigation and characterization of *Agave Americana* fiber-based composites with a cement matrix. This research was carried out both experimentally and numerically, with experimental designs that allowed us to compare the data obtained as well as get mathematical and statically modeling and characterization of the mechanical behavior of produced eco-composites. As a result of this study we have arrived at the following results:

- The vegetable fibers of the *Agave Americana* are among the natural and non-polluting sources of the environment
- For the extraction of the fibers we opted for the water retting technique which allowed to preserve the physical and mechanical properties of the fibers.
- To get rid of unwanted constituents such as lignin, pectin, waxes and fat, we performed soda treatment (alkalization) of the *Agave Americana* fibers. This resulted in a considerable improvement of the surface condition of the fibers
- After 2, 7 and 28 days of curing, the flexural and compressive strengths of the composite specimens vary with the fiber content in the specimen. The best results were obtained for the 0.5% fiber ratio for a 28-day curing period. The flexural and compressive strengths of the mortar samples decrease progressively with increasing fiber content ratio.
- In our trial we found that the raw fiber reinforced mortar samples have more strength than the treated fiber reinforced mortar samples. Unfortunately we cannot explain this behavior exactly due to the lack of conditions necessary to carry out more experiments to explain it.
- The experimental test applied to the composite cement/*Agave Americana* fiber allowed us to determine the mechanical properties related to the compressive test strength and the bending test strength of various specimens with different fiber ratios
- The measurements obtained during the tests carried out allowed us to determine the predictive models of the resistance to bending and compression by using the experimental designs
- The result of numerical models revealed to be in good agreement with those obtained from experimental models. The differences between these results are in the excellent range.
- In addition to the results found we were able to complete the gaps in our training and we managed to learn and even master some software (Design Expert ...).
- The developed models can be used to select the most resistant mixtures, while avoiding to realize a great number of tests for an optimal mixture and which answers the specifications required for trials.