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Article

Formulation, Characterization and Future Potential of Composite Materials from Natural Resources: the case of Kenaf and Date Palm Fibers

Aslain Brisco NGNASSI DJAMI [1,*], Ulrich NGNASSI NGUELCHEU [2], Serge DOKA YAMIGNO [2]

[1] Department of Fundamental Sciences and Techniques of Engineer, Chemical Engineering and Mineral Industries School, University of Ngaoundere, Ngaoundere, Cameroon
[2] Department of Physics, Faculty of Science, University of Ngaoundere, Ngaoundere, Cameroon

*Correspondence: Aslain Brisco NGNASSI DJAMI (ngnassbris@yahoo.fr)

Abstract: Thanks to their interesting mechanical properties, recyclability and low production costs, plant fiber-reinforced composites, derived from agricultural residues, are of particular interest to both manufacturers and scientists looking to incorporate new environmentally-friendly and biodegradable materials to replace synthetic fibers, particularly glass fibers. The growing use of these composites in fields such as the automotive, construction and building industries, and soon in aeronautics, raises concerns about the reliability of the structures with which they are manufactured. This reliability must be guaranteed at the design stage, by a good knowledge of the properties of the material used. In this case, for composites, it is necessary to know the mechanical properties of their constituents, fibers and matrix, etc. In this context, this paper focuses firstly on the economic and industrial recovery of Kenaf (K) and Date Palm (DP) fibers, and secondly on their incorporation as a reinforcing element in cementitious matrix composites, for subsequent use in non-structural applications. This research highlights the development of cementitious matrix bio-composites reinforced with this type of fiber, based on Taguchi's statistical methodology, in order to minimize the cost and number of tests. The bio-composites developed are then mechanically characterized under static loading in compression and 3-point bending after a 30-day drying period.

Keywords: Vegetable fibres; Kenaf fibre; Date palm fibre; Bio-composites; Mortar; Chemical treatment; Compression; 3-point bending

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1. Introduction

Today, most industrial sectors (automotive, aerospace or construction) are systems whose energy consumption, production costs or environmental impact are very high, with a large percentage of the contribution coming from the construction sector. Many countries have passed laws seeking alternative solutions to reduce the use of synthetic materials, as current construction materials are not biodegradable.

That's why many researchers are now taking action to promote the use of natural materials such as plants (wood, straw, hemp, flax, kenaf, date palm, etc.). Plant materials can make a significant contribution to limiting greenhouse gas emissions thanks to their ability to trap CO_2. What's more, using natural materials in innovative constructions also helps to reduce construction costs.

In terms of the development of composite construction materials, concrete and mortar are no exception. Researchers and fiber producers have thought about incorporating fibers as reinforcing materials in cement matrices.

Since then, the use of fibers in concrete and mortar has become increasingly common practice. Applications are being developed thanks to the proven ability of these new composites to limit cracking and improve resistance to bending, compression and impact. In this context, Cameroon is one of the countries with extraordinary plant fiber resources (Kenaf, Date Palm, Cork, etc.). Unfortunately, however, little use is made of them in practical applications [1].

In this paper, we will focus on the reinforcing fibers of Kenaf and Date Palm, which constitute one of the most abundant plant resources in Cameroon and which are still little exploited today. The main objective of this paper is to implement a bio composite reinforced with these two plant fibers (Kenaf and Date Palm), with a view to characterizing them mechanically and statistically, for subsequent use.

2. Literature review

This section, which consists of a literature review, is divided into three parts. In the first part, we review the state of the art concerning the environmental context and climate change. The impact of the industrial construction sector and the contribution of building materials will be presented.

In the second part, we present the specific characteristics of plant fibers in general, a study of their properties (physical, mechanical, etc.), their chemical compositions and their applications.

The third part explores composite materials reinforced with plant fibers, and more specifically cementitious matrix composites.

2.1. Environmental context

2.1.1. Climate change and the impact of the construction sector

Environmental climate change represents a serious threat to human society and the planet, caused by human activity. This threat is now documented by scientific and interdisciplinary work that makes it indisputable.

In this context, global agreements reaffirm the need for the widest possible cooperation between all countries to accelerate the reduction of global greenhouse gas emissions and limit the increase in average global temperature to less than 2°C (following efforts to limit global warming to 1.5°C by 2100). In this situation, several solutions have been proposed to limit the effects of global warming due to greenhouse gas emissions, in particular CO_2, including [2]: the gradual reduction in the use of fossil fuels; the transition to a low-carbon or decarbonized economic model.

The production of building materials and the installation of heating and air-conditioning equipment in industrial structures and buildings have become "energy-intensive" and very costly (in Algeria, for example, consumption in the building sector has been estimated at more than 40% of overall energy consumption, according to APRUE (the agency for the promotion and rationalization of energy use) [3-5].

2.1.2. Contribution of building materials

With regard to building materials, life cycle assessments (LCA) of buildings are generally based on environmental declarations for construction materials. The main materials used in construction are steel, wood and glass, but concrete is the most widely used. This material is difficult to replace in construction because of its compressive strength, which is associated with a reasonable cost.

However, concrete's ecological footprint is its downfall, as its manufacture is based on a material that is not very environmentally friendly: cement. To make one tonne of cement, around 400 kg of $C\boldsymbol{O_2}$ can be emitted into the atmosphere.

Given these environmental concerns, choosing the right materials is a good way of limiting the impact on the environment and ensuring a comfortable indoor environment. This choice must be based on a number of criteria: (i) economy (from an energy and financial point of view), (ii) aesthetics and environmental impact. In this context, the development of environmentally-friendly building materials is a priority issue. These materials must limit the energy consumption of the building sector linked to their thermal quality, and help to reduce greenhouse gas emissions over their entire lifespan [6].

2.2. Natural fibres as bio-sourced materials

Renewable, environmentally friendly, low cost and high thermal resistance are the characteristics most in demand in the field of building materials. In this context, biobased materials are materials whose raw material is derived from animal or plant biomass. They mainly include insulation materials in rolls, panels or loose (wool, hemp, straw, etc.). Biosourced concrete is a composite material made from plant particles or fibers and a mineral-based binder. These bio-based materials have a number of advantages over other types of material, including low environmental impact, renewable raw materials and economic competitiveness [7].

Plant fibers offer considerable environmental benefits: they are annually renewable, biodegradable, recyclable and carbon-neutral. From an environmental point of view, the use of fibers is justified insofar as they retain their ecological character following various treatments, even though these may affect their degradation kinetics. The origin of natural fibers has a major influence on their physical and mechanical properties. For example, sheep's wool has good thermal characteristics but poor mechanical performance [8].

There are a number of advantages to using natural fibers, particularly plant fibers, as reinforcement in composite materials. First of all, their use allows us to make the most of a local resource. What's more, they are often available at a lower cost than synthetic or artificial fibers. Secondly, using these fibers in composite materials reduces the environmental impact compared with conventional composites, since they are renewable raw materials that are biodegradable, have a neutral carbon footprint and require little energy to produce [9].

2.2.1. Definition

Under the name "natural fibers" lies a large family of materials that have been introduced into industry as new applications. They are classified according to their origin (natural, synthetic and artificial), their shape (straight, wavy, needle, etc.), their size (macro or micro fiber) and also by their mechanical properties. A plant fiber is a thread-like cellular expansion, mainly composed of cellulose, hemicelluloses, lignins and pectins. It can be found in two forms: isolated or grouped with other fibers. A single fiber corresponds to an elementary fiber cell, and forms a fiber bundle when it is grouped with other fibers. In bundles, the fibers are bound together mainly by pectins and hemicelluloses [10].

Natural fibers have been used for 3,000 years as a reinforcing element in composites, (for example in ancient Egypt, where straw and clay were mixed to build walls). Over the past decade, natural fiber-reinforced composites have received increasing attention from both academia and industry [11,12].

2.2.2. Classification

The large family of natural fibers can be subdivided into three sub-families (Figure 1) [13]:

- fibers of animal origin (proteic), which are subdivided into:
    - Wool (sheep, goat, rabbit, llama, vicuna);
    - Silk (silkworm);
    - Hair (alpaca, cashmere, camel, mohair);
- fibers of mineral origin (silicate fibers): peat, glass, asbestos, basalt, carbon, metals);

- fibers of plant origin (cellulosic), the sources of which are described in more detail later in this section.

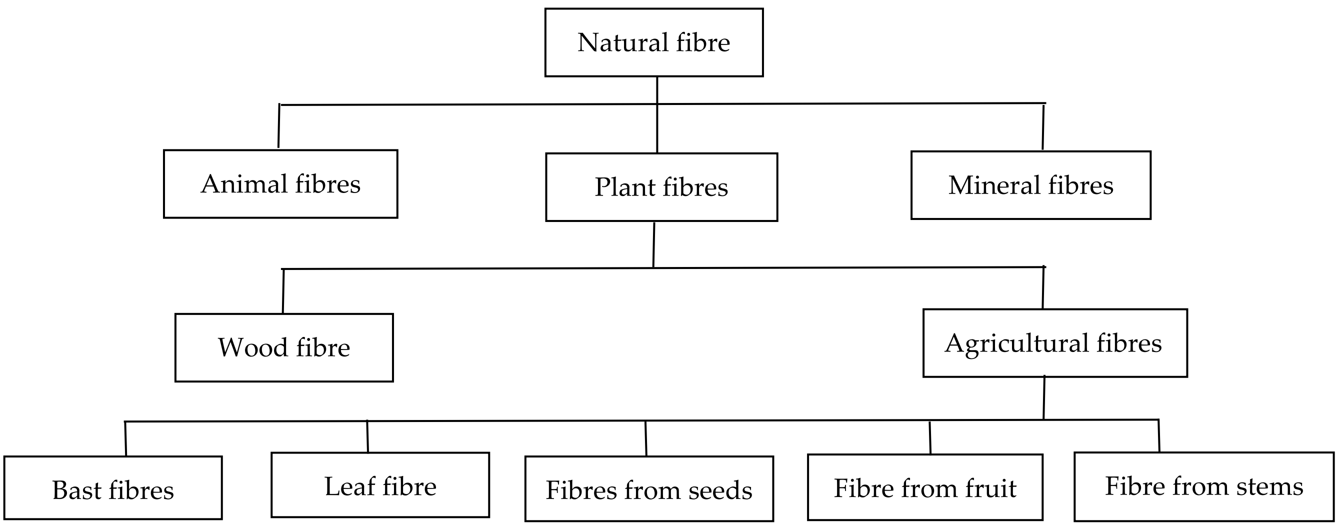

Figure 1. Classification of natural fibers [13,14].

Plant fibers themselves are subdivided according to their origin. These include bast fibers, which are extracted from plant stems (hemp, flax, ramie, jute), hard fibers, which are extracted from the leaves of trunks or fruit bark (sisal, coconut), and fibers derived from the seminal hairs of seeds (cotton, kapok), which are subdivided into two parts: wood fibers and agricultural fibers. The main categories of agricultural fiber are [15]:

- Bast fibers: extracted from the outer part of the stalk (flax, hemp, jute, kenaf,
- Ramie, etc.);
- Fibers extracted from leaves: sisal, banana and palm fibers, etc;
- Fibers extracted from the seminal hairs of seeds: cotton fiber, kapok fiber, etc;
- Fibers extracted from fruit husks: coconut fibers, etc.;
- Fibers extracted from stems or trunks: straw from wheat, rice, barley or other crops such as bamboo, etc.

2.2.3. Classification

Plant fibers are an interesting alternative to synthetic fibers because of their biodegradability and recyclability. These fibers have many advantages as reinforcements for composite materials. On the other hand, certain disadvantages can be an obstacle to industrial development. The main advantages and disadvantages are listed in Table 1.

2.2.4. Chemical composition of vegetable fiber

Plant fibers, which are biological structures, mainly contain cellulose, hemicellulose and lignin [17]. There are also small proportions of extractable constituents such as proteins and inorganic compounds. The proportion of holocellulose (comprising cellulose and hemicellulose) and lignin varies according to the natural fiber and its age. The composition depends on the growing conditions, the age of the plant, the climatic conditions and, of course, the nature of the fibers [15]. The composition of some natural fibers is shown in Table 2.

Table 1. Main advantages and disadvantages of plant fibers [16].

Advantages	Disadvantages
- Low density and low cost - Easy to recycle and biodegradable - CO_2 neutral - No skin irritation when handling fibres - No residues after incineration - Renewable resource, non-abrasive for tools - Requires little energy to produce - Good thermal and acoustic insulation properties - Highly specific mechanical properties (strength and rigidity)	- Water absorption - Poor dimensional stability - Poor ageing resistance - Poor fire resistance (200 to 230 ºC max) - Anisotropic fibres - Variation in quality depending on growing location, weather, etc. - For industrial applications, requires stock management - Discontinuous reinforcement

Table 2. Chemical composition of natural fibers in %.

Fibre	Cellulose	Hemicellulose	Lignin	Pectin	Waxes	Reference
			Stem fibre			
Linen	71 – 78	18.6 – 20.6	2.2	2.3	1.7	[18,19]
Hemp	70.2 – 74.4	17.9 – 22.4	3.7 – 5.7	0.9	0.8	[20]
Jute	61 – 71.5	13.6 – 20.4	12 – 13	0.2	0.5	[19]
Kénaf	45 – 57	21.5	8 – 13	3 – 5	N.S.	[21]
Ramie	68.6 – 76.2	13.1 – 16.7	0.6 – 0.7	1.9	0.3	[22]
Banana	63 – 64	10	5	N.S.	N.S.	[23]
Nettle	86	4.0	5.4	0.6	3.1	[24]
			Leaf fibre			
Sisal	67 – 78	10 – 14	8 – 11	10	2	[25]
Abaca	56 – 63	21.7	12 – 13	1.0	0.2	[26]
Curaua	73.6	9.9	7.5	N.S.	–	[20]
Pineapple	80 – 83	15 – 20	8 – 12	2 – 4	4 – 7	[28]
Henequen	77.6	4 – 8	13.1	N.S.	N.S.	[21]
			Fruit/seed fibre			
Cotton	85 – 90	5.70	0.7 – 1.6	0 – 1	0.6	[28]
Coco	36 – 43	0.15 – 0.25	41 – 45	3 – 4	N.S.	[29]
Palm oil	65	0 – 22	19	N.S.	N.S.	[30]
			Wood			
Hardwood	43 – 47	25 – 35	16 – 24	N.S.	N.S.	[23]
Softwood	40 – 44	25 – 29	25 – 31	N.S.	N.S.	[31]

N.S.: Not specified

2.2.5. Plant fiber applications

For many years, natural resources have been used for the production of basic products and as a reinforcing element for a large number of technical products [32]. Currently, in the production of vehicles, around 75% of their total end-of-life weight is recycled, of which 25%, known as automobile residue shredding, is disposed of as landfill because of its complexity. Reducing this percentage remains a major challenge, as the European End-of-Life Vehicle (ELV) Directive stipulates that from 2015, only 5% of vehicle weight can be disposed of in landfill sites.

Using materials that degrade naturally after use is one way of avoiding the complexity of recycling. With this in mind, researchers have for some time been developing the use of derivatives of jute, hemp, flax, sisal and coconut fibers, from plant materials, to reinforce the polymer. These cellulose fibers offer high specific rigidity (in relation to weight) and have little environmental impact compared with products made from synthetic fibers.

Today, the solution to ecological problems is offered by green materials.

In Europe, carmakers are using carpets made from abaca. Flax and hemp are used in the manufacture of doors with pressed panels, rear parcel shelves, seat backs, engine shields and headrests. Natural fiber composites also offer great potential as replacement materials, particularly as wood substitutes in the construction market. Various composite products are based on natural fibers, such as laminates, panels, partitions, door frames, shutters and roofing, which provide an alternative to existing materials [33].

In the construction industry, fibers of all kinds are widely used in various structures to improve their mechanical and physical performance. The use of plant fibers in cement reinforcement is relatively recent. In fact, a great deal of research is underway with the aim of replacing asbestos fibers with plant fibers [33-35]. At present, plant fibers are increasingly used in the production of tiles and car park paving, as well as for reinforcing plaster.

2.2.6. Properties of plant fibers

2.2.6.1. Mechanical properties

The great constitutive and anatomical variability of natural fibers implies a variability in their mechanical properties, so natural fibers are induced by their intrinsic characteristics, in particular by their chemical compositions (cellulose, hemicellulose, lignin and pectins), by the structure of the fiber (section, porosity, microfibrillar angle, form factor, length/diameter ratio, etc.), and by their anthropogenic characteristics (type of defibration, technical itinerary, etc.) or by independent and variable characteristics (moisture content, location of fibers in the stem, natural defects, growing conditions, etc.). ...) or by independent and variable characteristics (moisture content, location of fibers in the stem, natural defects, growing conditions, etc.). Table 3 provides a brief summary of the mechanical properties of different natural fibers.

The properties of natural fibers clearly show large variations, both within and across different fibers. Such wide variation in the mechanical properties of natural fibers can pose critical problems for the overall mechanical reliability of composites made from these fibers. Furthermore, it is not clear whether these mechanical properties (Table 3) were measured on single fibers or fiber bundles. For example, banana fiber demonstrated extremely high tensile strengths between 430 MPa and 914 MPa. Although flax fiber can have a maximum strength of 1834 MPa, its strength can reach 343 MPa. Similarly, the modulus of elasticity of flax covers a wide range (8 - 100 GPa). Overall, it can be said that the properties of natural fibers are highly variable and that their tensile strength is significantly lower than that of synthetic fibers. Some natural fibers, such as flax, can recover the modulus of elasticity of synthetic fibers [36].

Table 3. Mechanical properties of natural fibers.

Fibre	Deformation at break (%)	Tensile strength (MPa)	Young's modulus (GPa)	Reference
Lin	1.5 – 3.2	450 – 1500	2.6 – 38	[37,38]
Hemp	1.6 – 4.7	690 – 873	9.93	[27,39]
Jute	1 – 2	393 – 773	2.5 – 26.5	[40]
Kénaf	2.7 – 5.7	223 – 624	11 – 14.5	[39,41]
Sisal	3 – 15	80 – 640	1.46 – 15.8	[42]
Abaca	2.9	756	31.1	[41]
Coir	15 – 59.9	106 – 175	1.27 – 6	[43,44]
Banana	1.8 – 3.7	529 – 914	7.7 – 32	[43,44]

2.2.6.2. Physical properties

Generally, a plant fiber is characterized physically by its diameter, density, water content and percentage water absorption. The physical characteristics of some plant fibers are shown in Table 4.

In the case of fibers with a high cellulose content, such as banana and pineapple, the cracks propagate through the weak links between the cells, causing intercellular rupture without damaging the microfibrils. On the other hand, in the case of fibers with a low cellulose content, cracks propagate through the cells causing intracellular rupture with microfibril shrinkage [45]. Fiber elongation depends on the degree of crystallinity and the orientation and angle of the microfibrils in relation to the fiber axis. The dimensions and properties of lignocellulosic fibers change according to their moisture content. An increase in moisture content decreases the electrical resistivity of the fibers. The ability of fibers to absorb or desorb moisture should be taken into account when assessing their behavior in different applications. Hemicellulose plays an important role in the fiber's plastic properties, while lignin is an adhesive. It also stiffens and hardens the lignocellulosic fiber [16].

Table 4. Physical properties of different plant fibers [16, 46].

Type of fibre	Diameter (μm)	Length (mm)	Form factor (L/D)	Microfibrillar angle (degrees)	Density (kg/m³)	Recovery of Moisture (%)
Abaca	17.0−21.4	4.6−5.2	257	–	1500	14.00
Banana	–	2−3.8	–	11−12	1300−1350	–
Bamboo	10−40	2.7	–	–	1500	–
Coconut	16.2−19.5	0.9−1.2	64	39−49	1250	13.00
Cotton	11.5−17.0	20−64	2752	20630	1550	8.50
Flax	17.8−21.6	27.4−36.1	1258	5	1400−1500	12.00
Hemp	17.0−22.8	8.3−14.1	549	6.2	1400−1500	12.00
Jute	15.9−20.7	1.9−3.2	157	8.1	1300−1500	17.00
Kenaf	17.7−21.9	2.0−2.7	119	–	1220−1400	17.00
Pineapple	20−80	–	–	6−14	1520−1560	–
Ramie	28.1−35.0	60−250	4639	–	1550	8.50
Sisal	18.3−23.7	1.8−3.1	115	10−22	1300−1500	14.00

2.2.7. Some treatments carried out on plant fibers

2.2.7.1. Chemical treatment of plant fibers

The hydrophilic characteristics of natural fibers and the hydrophobic characteristics of the matrix (polymer, organic or other) are the main fundamental problems associated with the use of natural fibers as reinforcement in composites. However, using a chemical treatment on natural fibers reduces the hydrophilic characteristics of the fibers [32].

Toledo et al. [47] compared the effect of immersing sisal and coir fibers in water at PH=8.3, in a $Ca(\boldsymbol{OH})_2$ lime solution at PH=12 and in NaOH soda at PH=11 to distinguish between the effect of PH and that of the presence of calcium ions on the surface of the plant fibres after immersion. Baley et al. [48] studied the influence of different chemical treatments (soda, formic acid, acetic anhydride) on the bond (flax fibres-polyester resin), and compared it with the system (glass-polyester). Interfacial characterisation was carried out by removing a unit fibre from a microdrop of matrix to determine the apparent interfacial shear stress and the critical energy of rupture. The results show that there is a very significant increase in the bond (flax fiber-resin) after the treatments.

Claire et al. [49] studied the impact of different chemical treatments (NaOH, EDTA, lime-saturated water and a polyethylene solution) on hemp fibers, and found that soda treatment increased the crystallinity index of the fibers, decreased the setting time and increased the experimental stiffness of the composites. EDTA treatment, which also increased the stress at break of the composites, and the other two treatments had little effect on the mechanical properties. Bahloul et al. [50] treated esparto fibers at different percentages (20%-25%) for 48 h with NaOH and found that the final results were better than those obtained with untreated fibers. Rokbi et al. [51] also treated esparto fibers with NAOH soda at different concentrations (1, 5 and 10%) for 0h, 24h and 48h, and the results obtained showed that the strength and flexural modulus of composites reinforced with fibers treated with 10% NaOH for 24h improved by about 62% compared with composites reinforced with untreated fibers. composites reinforced with untreated fibers.

2.2.7.2. Physical treatment of plant fibers

The products most commonly used in this type of treatment are: oils and resins for various reasons [32]:

- Cleaning and purification of the surface;
- Oxidation and activation of sites on the surface.

These methods lead to a physical and chemical change in the compound's structural properties. Treatments include dielectric discharges such as cold plasma and exposure to laser, ultraviolet or gamma radiation. The application of plant fibers as reinforcement requires, on the one hand, strong adhesion between the fiber and the matrix and, on the other hand, limited absorption of water by the fibers.

2.2.7.3. Heat treatment of plant fibers

The treatment consists of heating the fibers in an inert atmosphere up to 280°C and holding them there long enough for the structure to be modified. This technique, known as torrefaction, was studied in particular by Bourgois et al. [9]. The process that has been adapted involves heat-treating wood in an inert atmosphere at temperatures below 280°C. The length of the treatment is sufficient to obtain the desired properties. It has the advantage of degrading the fiber less and preserving its mechanical properties as much as possible.

Merzoud et al. [52] heat-treated dissolving fibers by boiling them in a pot for four hours, then washing them to remove the sugars and water-soluble components contained in the fibers.

2.3. General information on fibre-cement composite materials

The term "composite material" refers to a material formed by the assembly of two or more materials, the final assembly having properties superior to those of each of the constituent materials separately [13]. The production of a composite material therefore requires the association of at least two components: the reinforcement and the matrix.

The combination of these two phases creates a third, called the interface or interphase. The properties of this assembly depend on the properties of the constituents, their geometric distribution, their proportions and their interactions. It is therefore necessary to know the mechanical and physicochemical properties of the constituents, the volume fraction of each, the orientation and distribution of the reinforcement, and the nature of the matrix/reinforcement interactions.

2.3.1. Composite materials include

Industrially, composite materials are made up of a matrix (providing protection and load transfer) and reinforcements (providing mechanical strength). The matrix is one of the basic constituents of composite materials, and has three main functions: to distribute the mechanical load throughout the reinforcement, to hold the fibres together for better overall homogeneity, and to protect the reinforcements from the external environment (mechanical or thermal shocks). The polymer resins most commonly used in composite materials are thermosetting resins (polyester, polyurethane, etc.) and thermoplastic resins (PVC, PS, PET, etc.) [53].

- Thermosets harden permanently when they are synthesized. They include polyester resins which, when combined with glass fibers, have good resistance to temperature and chemical agents, epoxy resins which have excellent mechanical, thermal and chemical properties, and phenolic resins which are fire-resistant [54].
- Thermoplastics come in the form of linear chains, which must be heated to shape them and cooled to fix them. This operation is reversible. Thermoplastic matrices have poor mechanical properties; reinforcement by incorporating fillers gives them improved thermal and mechanical resistance and good dimensional stability. The most common matrices are polyvinyl chloride, which is used in the manufacture of pipes; polyethylene, which is used to make toys, shampoo bottles and supermarket bags; polypropylene for food cans and floor coverings; and polystyrene, which is used in the manufacture of food containers, disposable crockery, toys and windows [55].

2.3.2. Composite materials based on natural fibers

Choosing the right materials is a good way of limiting their impact on the environment and ensuring a comfortable indoor environment. To this end, the use of composite materials based on plant fibers (hemp, straw, flax, wood, date palm, etc.) is a good response to the need to protect human health, meet well-being and comfort requirements and reduce environmental impact. In addition, the use of composite materials based on plant products has the following advantages:

- the use of abundant, renewable, recyclable and sustainable resources;
- plants renew themselves periodically and therefore provide an abundant resource, given that other resources (coal, oil, gas) will be exhausted within a few decades;
- reducing greenhouse gas emissions;
- low grey energy (the sum of all the energy required to produce, transport, manufacture, use and finally demolish the material or dispose of it at the end of its life [56]).

2.3.3. Properties of composite materials reinforced with plant fibers

2.3.3.1. Mechanical properties

Mechanical properties are the main properties studied in natural fiber-reinforced composites. These properties include strength, stiffness, toughness, shear, compression, fatigue and creep. The mechanical strength of composite materials is assessed by a number of test methods that are mechanically simple in concept but extremely sensitive to sample preparation and test execution procedure [57].

Mechanical tests mainly include tensile, compression, shear, bending and fatigue tests. These test methods are governed by standards developed by (ASTM), (ISO) and (SACMA). In the present study, we restrict ourselves to the study of the flexural and compressive properties of plant fiber-reinforced cementitious matrix composites. The previous tests were carried out to determine the flexural and compressive strength, Young's modulus and elongation at break of the material [58].

2.3.3.2. Composite failure under tensile loading

On a macroscopic scale, a composite under tensile loading can exhibit ductile or brittle fracture. These two terms describe the degree of macroscopic plastic deformation preceding failure.

- Ductile fracture occurs through the coalescence of micro-voids, which lead to internal cracking by normal fracture. This mode of fracture is accompanied by sudden plastic deformation, which gives the fractured surface a fibrous appearance.

- Brittle fracture is characterized by rapid crack propagation without sudden plastic deformation; the behavior of a brittle material is almost exclusively elastic until fracture. In this type of material, the fracture surface is characterized by the presence of radial striations on the fracture surface that are parallel to the direction of crack propagation. It should be noted that a ridge is produced when two cracks, which are not coplanar, become connected by tearing of the intermediate material. Brittle fracture in tension has a granular appearance with little or no necking [58,59].

On a microscopic scale, two fracture mechanisms can occur depending on the properties of the interface, namely debonding at the fiber/matrix interface or disbonding of the fibers from the matrix [60]. If the interface disbonds easily, crack propagation is interrupted by the disbonding process and instead of the crack passing through the fiber, it propagates along the fiber surface, allowing the fiber to withstand higher loads. Fiber delamination occurs because the fibers break at random locations away from the plane of the crack. One of the broken fiber ends loosens from the matrix as the crack opens with increasing applied load [60].

2.3.3.3. Effect of chemical treatments of fibers on the mechanical behaviour of composites

The long- and short-term mechanical performance of fiber-reinforced composites depends mainly on three factors: the strength and modulus of the fiber, the strength and chemical stability of the resin and the effectiveness of the interracial bonding that transfers the load from the matrix to the fiber. However, the quality of the fiber-matrix interface remains the key factor to optimize for a high-performance composite. Because of the strong dependence between fiber polarity and inter-racial adhesion, researchers have been interested in chemically modifying fibers using a number of treatments as mentioned in the previous paragraphs, with the aim of improving composite performance. These treatments have achieved varying degrees of success in improving the fiber-matrix adhesion of lignocellulosic fiber composites [46].

2.3.4. Cementitious matrix composites

2.3.4.1. Generality

Fiber-reinforced concrete or mortar is a material that combines fibers with a cementitious matrix to enhance its mechanical properties. The fibers act as reinforcement, while the matrix ensures the cohesion of the material and the transfer of forces to the fibers.

Fibers have long been used to reinforce fragile materials. Straw has been used to reinforce mud bricks, and horsehair to reinforce masonry mortar and plaster. More recently, large-scale commercial use of asbestos fibers in a cementitious matrix began with the invention of the Hatschek process in 1898. However, mainly because of the health hazards associated with asbestos fibers as we know them, alternative fiber types were introduced in the 1960s and 1970s. Today, a large number of technical materials (ceramic, metallic or organic matrices) incorporate fibers to improve material properties. Industrial interest in fiber-reinforced construction materials continues to grow due to the large amount of research being carried out in this area [61].

2.3.4.2. Motar

Mortar is a construction material containing binder (cement, plaster or lime), fine aggregate (such as sand, which distinguishes it from concrete) and water. Because of its fine granularity, it is lighter than concrete [62,63].

Mortars can be very different from one another depending on the nature and percentages of the constituents, the mixing, processing and curing. They are used in construction as bonding or sealing elements, or as plaster. These materials are highly resistant to chemical attack, humidity and heavy mechanical stress.

Mortar is obtained by mixing a binder (lime, cement or other), sand and water, to which mineral additions and/or organic additives may be added [63].

2.3.4.3. Mechanical behaviour of fiber cement composites

2.3.4.3.1. Bending behaviour

Studies carried out on composite materials based on raw plant fibers show a change in the mechanical behaviour of the composite. Whereas the pure cementitious matrix exhibits brittle linear elastic behaviour, fiber composite specimens tested in three-point bending retain their integrity even beyond the maximum load, and continue to support a significant load in the post-peak part [64].

2.3.4.3.2. Behaviour in compression

The compressive strength of composites is a property significantly affected by the incorporation of plant fibers. According to studies by Kirker et al.[65], the compressive strength of composites incorporating jute fibers varies significantly with the addition of these fibers. Compressive strength is either increased or decreased as a function of increasing fiber content with varying lengths.

3. Materials and Methods

This section describes the different experimental techniques, starting from the preparation of plant fibers, the test conditions used, up to the development of bio composites in cementitious matrix mortar reinforced by two types of plant fibers, Kenaf fiber (K) and date palm fiber (DP). The development of bio-composites is carried out by statistical experimental planning based on the Taguchi method. The objective of this method is to minimize the number of experiments as well as to reduce the cost of mechanical tests. The characterization of the mechanical behavior of the bio-composites developed was carried out by two essential mechanical tests, namely the 3-Point bending test and the compression test.

3.1. Fibre preparation

In this paper, we seek to develop bio-composites consisting of a cementitious matrix reinforced with different percentages of two types of plant fibre, in order to give the bio-composites developed quite remarkable strength and durability. The choice is reserved for Kenaf and Date Palm plant fibres, which are found in Cameroon and grown locally.

To do this, the Kenaf or Date Palm fibers used in this paper were extracted by hand from their plants.

The fibers thus obtained are called raw fibers, with different diameters ranging from 0.3 to 1 mm and lengths from 400 mm to 800 mm. Each of the two raw fibers is cut into different lengths of 5mm, 10mm and 20mm, in order to determine the effect of varying the length of the fibers on the mechanical properties of the bio-composites produced. Figure 2 shows the Kenaf fiber (a) and Date Palm fiber (b) used.

(a)

(b)

Figure 2. Kenaf fiber (a) and Date palm fiber (b) used.

The variation in the length of the fibers used for reinforcement leads to a variation in their quantities in mass (grams), and the relationship that expresses this variation is given by:

Volume of the mould used for the 3-point bending test (4x4x16) cm^3 = 256 cm^3 (0.000256 m^3).

According to standard EN 196-1 [66] used for mortar preparation, a volume of 0.000256 m^3 translates into a quantity of 1350 g.

Therefore

1350 g 100%

X g 1%

Table 5 summarizes the results in mass of the quantities of fibers (Kenaf or Date Palm) for each percentage used.

Table 5. Results in mass of the quantities of Kenaf and Date Palm fibers.

% of fibre	Fibre lengths (mm)	Quantity by mass (g)
1 %	5 / 10 / 20	4.5
2%	5 / 10 / 20	9
5%	5 / 10 / 20	22.5

3.2. Taguchi's experimental design

The Taguchi statistical method, invented to carry out experimental designs applied in all industrial disciplines, focuses above all on minimizing variations around the target value (responses). The Taguchi method therefore requires a minimum of data to determine the factors that have a significant influence on the response (in our case, the mechanical characteristics) and to optimize it using a minimum of experiments, thus saving time and resources [67,68].

In order to establish an experimental design based on the Taguchi method for evaluating the flexural and compressive mechanical properties of the bio-composites produced, we considered the following factors: percentage fiber reinforcement (%) and fiber length (mm), each with three levels 1%, 2%, 5% and 5 mm, 10 mm, 20 mm respectively (Table 6).

The Taguchi method was employed using Minitab 17 software. The application of the two factors, each with three levels, resulted in the design of a reduced Taguchi L9 experimental design (33) (Table 7). In other words, the orthogonal table obtained leads to having only 9 types of experiments instead of 27, but sufficient to optimize the factors considered. Next, the experimental results obtained can be analyzed using a signal-to-noise ratio to assess the effect of the parameters and their levels on the response.

3.3. Chemical treatment of fibres

The hydrophilic characteristics of natural fibres followed by the hydrophobic characteristics of the matrix are the main fundamental problems associated with the use of natural fibres as reinforcing elements in bio-composites. However, using an alkaline chemical treatment on plant fibres reduces the hydrophilic characteristics of the fibres.

To do this, it is necessary to clean the outer surface of the fibres used with NaOH, in order to eliminate impurities and dust from their natural environmental exposure. The aim is to disrupt the absorption of water and moisture, as well as to remove the lignin (which is responsible for the hydrophilicity of the fibres) that surrounds the fibre and obtain rough surfaces.

To achieve this, Kenaf or Date Palm fibre bundles were chemically treated to obtain rough surfaces in order to improve fibre/matrix adhesion. The fibre bundles were immersed in a sodium hydroxide (NaOH) solution at different concentrations (1%, 2% and 5%) for immersion times of 1h, 4h and 8h at ambient temperature and pressure. They were then rinsed with water before being immersed in sulphuric acid (H_2SO_4) for 5 min, then immersed in distilled water for 15 min to achieve a neutral PH, and finally dried naturally before being taken for reinforcement.

The reaction of Kenaf (K) or Date Palm (DP) fibre during chemical treatments with NaOH is given by equation 1.

$$K(DP) - OH + NaOH \rightarrow K(DP) - O - Na^{+} + H_2O \quad (1)$$

Table 6. Factors and levels used in the design of a Taguchi experimental plan (raw fiber).

Factor	Symbols	Level 1	Level 2	Level 3
Fibre (%)	(A)	1	2	5
Length (mm)	(B)	5	10	20

Table 7. Taguchi orthogonal experimental design for raw fibers.

Run	Fibres (%) (A)	Length (%) (B)
1	1	5
2	1	10
3	1	20
4	2	5
5	2	10
6	2	20
7	5	5
8	5	10
9	5	20

The application of the Taguchi method, still using Minitab 17 software, for the four factors mentioned above, i.e. % of fibres, their lengths, % of alkaline element (NaOH) and immersion time, each of which has three levels, led to the design of a Taguchi L9 (33) reduced experimental design, which is shown in Table 8.

Table 8. Taguchi orthogonal experimental design for treated fibers.

Run	Fibre (%) (A)	Lengths (mm) (B)	NaOH (%) (C)	Time (h) (D)
1	1	5	1	1
2	1	10	2	4
3	1	20	5	8
4	2	5	2	8
5	2	10	5	1
6	2	20	1	4
7	5	5	5	4
8	5	10	1	8
9	5	20	2	1

3.4. Development of bio-composites with a cementitious matrix reinforced with K and DP fibres

As part of the recovery of local agricultural fibrous waste from Kenaf and date palm plants, bio-composites with a cementitious matrix reinforced by this type of fibre are being developed for subsequent use in non-structural applications in buildings in arid and semi-arid zones in northern Cameroon.

To achieve this objective, and in order to minimise the number of tests as much as possible, and to enable the development of bio-mortars at the lowest possible experimental costs, a design of experiments for the development of 9 types of tests was established using Taguchi's methodology implemented by Minitab 17 software, combining four development parameters (Table 8), namely:

(A)the percentage of fibre incorporation in the mortar in %,

(B) fibre length in mm,

(C) the percentage concentration of the alkaline element NaOH in % ,

(D) the immersion time of the fibres in the chemical solution in hours (h).

This could be used to assess the influence of the above-mentioned processing parameters on the mechanical properties of bio-mortars in terms of strength and ultimate stress, by means of 3-point bending and compression tests after 30 days' curing of the samples.

Based on the experimental design L9 established for the preparation of bio-mortars reinforced with raw K and DP (Table 7) and those chemically treated (Table 8), and in accordance with the European standard NF-EN 196-1[66] for the preparation of a standardised mortar, sand/cement mortar composites reinforced with K and DP fibres were prepared for testing in this study.

To do this, the mixture of cementum, sand and water are mixed according to certain criteria, for the preparation of (03) specimens for testing in 3-point bending (40×40×160) mm^3or compression (5 x 5 x 5) mm^3, the following quantities were used:

- Dry sand: 1350 g (a standard sand),
- Cement: 450 g (composite portland cement "CEM II/A-M (P-L) 42.5 R"),
- Water: 225 g (tap water).

The samples and experiments were prepared as follows:

1. **Mixing and moulding the specimens**
    - ***a.*** With the mixer running, water is poured into the container, then cement is added.
    - ***b.*** The mixer is then run at low speed for one minute, during the last 30 seconds sand is added, and the mixer is then run at high speed for two minutes.
    - ***c.*** Once the mixer has stopped moving, the walls and bottom of the container are scraped to ensure that no part of the mortar escapes the mixer.
    - ***d.*** Resume mixing for two minutes at a fast speed.
    - ***e.*** The specimens are moulded using impact equipment.
    - ***f.*** Place the fibre-reinforced mortar on a non-cement-reactive, absorbent plate.
    - ***g.*** Form an approximately rectangular cake divided into three (03) roughly equal fractions.
    - ***h.*** Place the mould on the impact table and introduce a fraction of the cake into each of the compartments. Set the machine in motion for 60 impacts.
    - ***i.*** Repeat the same operations for the second layer of cake and remove the mould from the machine, clearing and removing the excess fibre-reinforced mortar with a metal ruler moved in a sawing motion along the length of the test specimens.
    - ***j.*** The mould filled with fibre-reinforced mortar is kept in a damp cupboard (98%) at a temperature of 20°C ± 2 and not stacked on top of each other.

2. **Demoulding the test tubes**

- ***a.*** After 24 hours of storage, carefully remove the test tubes from the mould.
- ***b.*** Mark the test specimens "sample code, sample location, etc." without using the compression face, then immerse in water at a temperature of 20°C ± 1 until the time of testing (30 days).
- ***c.*** The age of the specimens is counted from the time of mixing until the start of the test.

3.5. Mechanical testing machines

3.5.1. Compression test on bio-composites

The compression test is one of the most widely used tests for the mechanical characterisation of cement-based construction materials (mortar, concrete, reinforced concrete, etc.). It is used to determine a number of standardised quantities in accordance with ASTM standard C109 / C109M [69], and involves applying a compressive force to a sample resting on a non-deformable surface. This load causes the specimen to be crushed, and stresses and strains are measured simultaneously during the test.

The compression tests on the mortar/K or DP bio-composite samples were carried out at ambient temperature and pressure, on an Instron universal compression/bending machine (available from the Mechanical Engineering laboratory of the University Institute of Technology, University of Ngaoundéré, Cameroon) (Figure 3), at a speed of 2 mm/min. The technical characteristics of the compression machine are given in Table 9.

Table 9. Technical characteristics of the compression machine.

Model	Instron 1125
Force Capacity	100 kN
Column Spacing	559 mm
Crosshead Travel	914 mm
Minimum Speed	0.05 mm/min
Footprint	1022 x 21 x 78 mm
Others	Complete computer

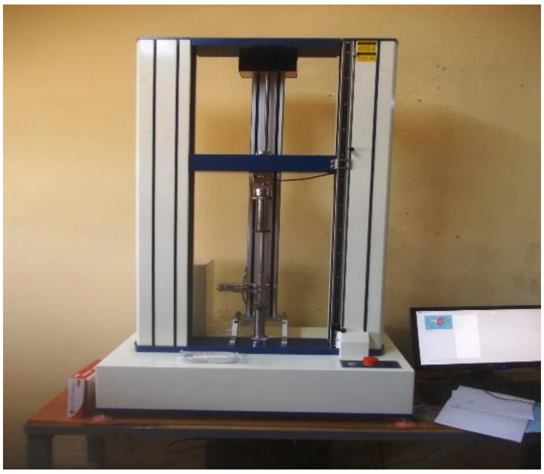

Figure 3. Compression testing machine.

3.5.2. Resistance to bending

The same Instron machine (Figure 3) was used for static 3-point bending tests carried out on bio-composites made and reinforced with K and DP fibres in accordance with ASTM standard C348-14 [70]. The tests were carried out under the same conditions with a displacement speed of 50 N/s ± 10 N/s until failure, at a temperature of 22°C and an average relative humidity of 25%.

4. Results and discussion

The main objective of this section is to assess the mechanical behaviour of bio composites produced with a cementitious matrix reinforced with short plant fibers from the Kenaf plant and the Date Palm, for possible use in non-structural applications.

4.1. Mechanical characterisation of bio-mortars reinforced with raw K and DP

4.1.1. Statistical analysis of mechanical behaviour

Based on Table 7, which represents the Taguchi experimental design (L9) of the raw fibers, we carried out this experimental design on the processed bio-mortar reinforced with K and DP and which were loaded in 3-point bending and compression, which enabled us to determine the ultimate force and stress summarized in Table 10 and 11 respectively.

Table 10. Results of the mechanical properties of K-reinforced bio-mortar subjected to 3-point bending and flexural stress.

Run	Fibres (%) (A	Length (mm) (B)	Bending		Compression	
			Forces (N)	Stress (MPa)	Forces (N)	Stress (MPa)
Control	0	0	2303	5.39	13290	31.1
1	1	05	2786	6.52	16153	37.8
2	1	10	2649	6.20	13290	31.1
3	1	20	2602	6.09	12307	28.8
4	2	05	1816	4.25	10384	24.3
5	2	10	2098	4.91	10042	23.5
6	2	20	1397	3.27	9145	21.4
7	5	05	1217	2.85	13803	32.3
8	5	10	1094	2.56	10512	24.6
9	5	20	901	2.11	7735	18.1

Table 11. Results of the mechanical properties of DP-reinforced bio-mortar subjected to 3-point bending and flexural stress.

Run	Fibres (%) (A	Length (mm) (B)	bending		Compression	
			Forces (N)	Stress (MPa)	Forces (N)	Stress (MPa)
Control	0	0		5.39		31.1
1	1	05		4.85		27.8
2	1	10		4.20		21.1
3	1	20		3.89		28.8
4	2	05		4.05		24.3
5	2	10		3.91		23.5
6	2	20		3.27		21.4
7	5	05		5.52		32.2
8	5	10		5.20		29.6
9	5	20		4.11		18.1

Initially, the results obtained for the K-reinforced bio-mortar samples compared to the reference mortar (without fibers) mentioned in Table 10, show either increases or decreases in the mechanical properties in terms of bending or compressive strength or stress. The highest stress is evaluated by a % Kenaf fiber reinforcement of 1% with a length of 5 mm, this increase is translated by a value of 20.96% and 21.5% respectively in flexural and compressive strength. The lowest stress is evaluated by 5% Kenaf fiber reinforcement with a length of 20 mm. This reduction is reflected respectively by a value of 60.85 % in the flexural strength and 41.8 % in the compressive strength, compared with those of unreinforced mortars.

On the other hand, Table 11 shows the results obtained for the DP-reinforced bio mortar samples compared with the reference mortar. Slight increases were obtained in both the 3-point bending test and the compression test, which were 2.41% and 3.5% respectively for bio mortars reinforced with 5% date palm fiber 5 mm long.

Table 12 shows the ranking order of the influence of elaboration factors for Kenaf (K) and Date Palm (DP) raw fibers on the mechanical responses for signal-to-noise ratios (Prefer larger). It is noted that the percentage factor (%) of fiber incorporation for both types of fiber has a greater effect than the fiber length factor.

Table 12. Classification of the influence of manufacturing factors on bending stress (K and DP).

	Kenaf fibre	
Level	fibre (%)	Length (mm)
1	18.788	14.177
2	14.059	12.801
3	3.234	9.102
Delta	15.554	5.074
Rank	1	2
	date palm fibre	
Level	fibre (%)	Length (mm)
1	12.66	13.57
2	11.43	12.88
3	13.81	11.46
Delta	2.38	2.11
Rank	1	2

4.1.2. Analysis of variance ANOVA

The analysis of variance ANOVA performed by Minitabe.17 allows us to provide information on the relative significance attributed to each test parameter, either flexural or compressive, on the mechanical properties of the K or DP reinforced mortars.

The contribution of each test parameter in terms of % of K (or DP) and their lengths (in mm) has been determined in Tables 13 and 14 for the stress responses (MPa).

Table 13. Analysis of variance for bending stress.

Source	DL	SC ajust	CM ajust	F-value	P-value	Remarks
Regression	2	19.6937	9.8469	17.33	0.003	
Fibre (%)	1	18.7680	18.7680	33.02	0.001	significative
Length (mm)	1	0.9257	0.9257	1.63	0.249	N- significative
Error	6	3.4101			0.5683	
Total	8	23.1038				
R square				95.24%		
R square ajust				90.32%		
R square prev				96.75%		

DL: *Degree of freedom;* ***CM***: *Medium square;* ***SC***: *Sum of squares.*

In this context, given the reliability evaluation index F-value and the probabilistic parameter P-value measured and compared with the established experimental data, the hypothesis is that, if the parameter P-value is less than 0.05, the responses extracted from the mechanical bending or compression tests can be considered significant. For this purpose, the independent parameter fiber concentration (%) is considered significant for the above response, while the analyzed length parameter is considered non-significant.

Table 14. Analysis of variance for compressive stress.

Source	DL	SC ajust	CM ajust	F-value	P-value	Remarks
Regression	2	149.88	74.94	2.99	0.126	
Fibre (%)	1	44.37	44.37	1.77	0.023	significative
Length (mm)	1	105.51	105.51	4.20	0.086	N- significative
Error	6	150.63			25.11	
Total	8	300,52				
R square				89.88%		
R square ajust				93.17%		
R square prev				90.00%		

4.2. Mechanical characterization of bio-mortars reinforced with K and DP chemically treated

The experimental results reveal the overall and mean mechanical properties and their standard deviations from the 3-point bending and compression tests in terms of stress, obtained for the different chemically treated K and DP reinforced bio-mortars and made according to the Taguchi L9 experimental design are presented in Tables 15 and 16 respectively.

Initially, from Table 10 onwards, the chemically treated K-reinforced bio-mortar samples compared with the reference mortar (without fibers) always show either increases or decreases in flexural and/or compressive strength. The highest resistance is estimated by a % of alfa fiber reinforcement of 1% with a length of 5 mm treated with 1% NaOH for 1 hour. This increase is reflected by a value of 32.5% and 29.3% in flexural and compressive strength respectively. The lowest stress is evaluated by a % Kenaf fiber reinforcement of 5% with a length of 20 mm, this decrease is translated respectively by a value of 40.63 % in flexural strength and 66.88 % in compressive strength, compared to those of unreinforced mortars.

Table 15. Average mechanical characteristics of K-reinforced bio-mortars obtained in the bending and compression test.

Run	K (%)	Lengths (mm)	NaOH (%)	Time (h)	Flexion			Compression		
					results	Average value	STD	results	Average value	STD
0	0	0	0	0		5.39	/		31.1	/
1	1	5	1	1	6.32	7.14	1.73	40.5	40.2	1.37
					7.70			41.4		
					7.42			38.7		
2	1	10	2	4	6.89	6.33	1.03	36.4	35.2	4.84
					5.65			34.0		
					6.46			35.4		
3	1	20	5	8	5.55	5.37	3.44	25.0	24.3	1.42
					4.98			25.3		

					5.60			22.7		
					7.01			21.1		
4	2	5	2	8	7.81	6.44	0.23	20.6	20.6	0.50
					6.49			20.1		
					4.71			18.9		
5	2	10	5	1	3.95	4.63	1.60	17.2	18.2	0.91
					4.27			18.6		
					2.70			12.2		
6	2	20	1	4	2.53	2.53	1.63	17.2	14.9	2.53
					2.38			15.4		
					5.07			19.0		
7	5	5	5	4	5.23	5.54	0.68	20.9	20.4	0.36
					5.23			21.2		
					3.17			12.60		
8	5	10	1	8	2.76	3.60	1.73	13.04	12.60	0.13
					3.22			12.01		
					3.20			10.7		
9	5	20	2	1	3.17	3.20	1.41	10.0	10.3	0.52
					4.13			10.2		

STD: Standard deviation.

Table 16. Average mechanical characteristics of DP-reinforced bio-mortars obtained in the bending and compression test.

Run	**DP (%)**	**Lengths (mm)**	**NaOH (%)**	**Time (h)**	**Flexion**			**Compression**		
					results	Average value	STD	results	Average value	STD
0	0	0	0	0	/	5.39	/	/	31.1	/
					5.32			17.9		
1	1	5	1	1	3.91	4.67	1.17	18.7	19.51	2.11
					4.68			21.9		
					3.39			11.32		
2	1	10	2	4	3.62	3,57	7.06	11.98	11.50	3.49
					3.54			11.20		
					5.39			18.8		
3	1	20	5	8	4.76	4.90	3.64	17.7	18.23	0.56
					4.76			18.1		
					3.25			16.6		
4	2	5	2	8	4.50	3.80	6.28	16.6	15.60	1.86
					3.77			13.4		
					2.72			18.6		
5	2	10	5	1	3.02	2.80	1.86	16.4	16.20	4.10
					2.68			14.5		
					3.64			13.0		
6	2	20	1	4	4.23	3.96	2.99	11.9	13.18	1.41
					4.02			14.7		
					6.81	7.70	0.81	32.6	33.53	0.42

7	5	5	5	4	8.40			36.2		
					7.90			31.8		
					5.99			28.46		
8	5	10	1	8	6.00	6.43	2.47	27.79	27.97	0.43
					7.31			27.67		
					4.35			21.00		
9	5	20	2	1	5.73	4.14	0.76	21.96	21.33	0.55
					3.22			21.03		

Table 17 shows the ranking order of the influence of each processing factor for Kenaf (K) treated fibers on the mechanical responses (ultimate stress) for the signal-to-noise ratios (Prefer greater). It is noted that the percentage (%) factor of fiber incorporation has a greater effect (rank 1) for bending stresses, and a medium effect (rank 2) for compressive stresses.

Table 17. Classification of the influence of processing factors on bending and compressive stress for K.

Level	K (%)	Length (mm)	NaOH (%)	Time (h)
	Bending stress			
1	15.90	16.04.12.09.13.50		
2	12.52	13.4914.10.12.99		
3	12.03	10.92.14.26.13.97		
Delta	3.87	5.12 2.17 0.98		
Rank	2	1 3 4		
	Compressive stress			
1	30.24	28.18	25.85	25.85
2	24.98	26.05	25.82	26.86
3	22.82	23.81	26.37	25.33
Delta	7.42	4.37	0.55	1.53
Rank	1	2	4	3

In addition, the ANOVA regression analysis carried out to identify the significance of each processing parameter, i.e.: K (%); Length (mm); NaOH (%) and Time (h) and those on flexural strength and compressive strength is represented in Tables 18 and 19 respectively.

Table 18. ANOVA regression analysis of output responses (σ_f) as a function of processing parameters.

Source	DL	SC ajust	CM ajust	F-value	P-value	Remarks
Regression	4	15.7798	3.9449	3.50	0.126	
K (%)	1	5.2410	5.2410	4.66	0.009	significative
Length (mm)	1	10.0245	10.0245	8.90	0.041	significative
NaOH (%)	1	0.4703	0.4703	0.42	0.553	N- significative
Time (h)	1	0.0439	0.0439	0.04	0.853	N- significative
Error	4	4.5033	1.1258			
Total	8	20.2830				
R squared (%)	97.80%					
R-squared (adjusted)	95.60%					

R squared (forecast)	90.00%

Table 19. ANOVA regression analysis of output responses (σ_f) as a function of parameters.

Source	DL	SC ajust	CM ajust	F-value	P-value	Remarks
Regression	4	582.214	145.554	0.126	0.188	
K (%)	1	391.828	391.828	7.03	0.057	Significative
Length (mm)	1	162.975	162.975	2.92	0.162	Significative
NaOH (%)	1	3.949	3.949	0.07	0.803	N-Significative
Time (h)	1	23.461	23.461	0.42	0.552	N-Significative
Error	4	222.98	55.747			
Total	8	805.202				
R squared (%)	97.80%					
R-squared (adjusted)	95.60%					
R squared (forecast)	90.00%					

In addition, using Design software, a regression equation is established for the predicted responses symbolized by (Y) fitted by ANOVA analysis for bending stress (Y_{σ_f}) and compressive stress (Y_{σ_c}). These were evaluated respectively to deduce the final expression of the mathematical model represented by Equations 2 and 3.

$$\sigma_f = 7.68 - 0.449 \times K\,(\%) - 0.1692 \times Length\,(mm) + 0.134 \times NaOH\,(\%) + 0.024 \times Time\,(h) \quad (2)$$

$$\sigma_c = 43.65 - 3.88 \times K\,(\%) - 0.682 \times Length\,(mm) - 0.39 \times NaOH\,(\%) - 0.563 \times Time\,(h) \quad (3)$$

Table 20 shows the ranking of the influence of each processing factor for processed Date Palm (DP) fibers on the ultimate stresses for signal-to-noise ratios (Prefer greater). The results only address the percentage (%) factor of incorporation of fibers with a greater effect (rank 1) for bending and compression stresses. Rank 2, on the other hand, is reserved for immersion time and the alkaline element for bending and compressive stress respectively.

Table 20. Classification of the influence of processing factors on bending and compressive stress for DP.

Level	DP (%)	Length (mm)	NaOH (%)	Time (h)
	Bending stress			
1	12.75	14.24	13.83	11.56
2	10.83	12.05	11.66	13.58
3	15.41	12.70	13.49	13.85
Delta	4.58	2.18	2.17	2.30
Rank	1	3	4	2
	Compressive stress			
1	24.08	26.73	25.71	25.53
2	23.48	24.78	23.89	24.71
3	28.67 5.19	24.73	26.64	26.00
Delta	1	1.99	2.75	1.30
Rank		3	2	4

The ANOVA regression analysis carried out to examine the significance of each processing parameter on flexural strength and compressive strength is shown in Tables

21 and 22 respectively. This analysis shows that the parameters (%) of DP as well as their length are significant for both responses. However, the NaOH parameter (%) is non-significant for flexural strength and significant for compressive strength, and vice versa for the immersion time parameter.

Table 21. ANOVA regression analysis of output responses (σ_f) as a function of processing parameters.

Source	DL	SC ajust	CM ajust	F-value	P-value	Remarks
Regression	4	10.6784	2.6696	1.35	0.0389	
DP (%)	1	7.2083	7.2083	3.65	0.0129	**Significative**
Length (mm)	1	1.2361	1.2361	0.63	0.0473	**Significative**
NaOH (%)	1	0.3841	0.3841	0.19	0.0682	**N-Significative**
Time (h)	1	1.8499	1.8499	0.94	0.0388	**Significative**
Error	4	7.9014	1.975			
Total	8	18.5798				
R squared (%)	97.47 %					
R-squared (adjusted)	94.95 %					
R squared (forecast)	90.00%					

Table 22. ANOVA regression analysis of output responses (σ_f) as a function of processing parameters.

Source	DL	SC ajust	CM ajust	F-value	P-value	Remarks
Regression	4	317.160	79.290	3.68	0.117	
DP (%)	1	253.698	253.698	11.78	0.026	Significative
Length (mm)	1	35.129	35.129	1.63	0.0271	Significative
NaOH (%)	1	24.399	24.399	1.13	0.0347	Significative
Time (h)	1	3.935	3.935	0.18	0.0691	N-Significative
Error	4	86.159	21.540			
Total	8	403.319				
R squared (%)	98.64 %					
R-squared (adjusted)	97.28%					
R squared (forecast)	90.00 %					

5. Conclusions

The aim of this paper was to develop and characterize new cementitious matrix bio-composites reinforced with Kenaf and Date Palm plant fibers. In general, these fibers are known for their hydrophilicity (water absorption), which affects the fiber/matrix interfacial adhesion property. To this end, alkaline treatments using sodium hydroxide (NaOH) with different concentrations and immersion times were carried out on the fiber bundles. Experimental 3-point bending and compression tests were carried out on the bio-mortars produced, enabling us to determine the ultimate strength and stress.

The characterization analyses carried out in this paper have led to a number of conclusions that allow us to envisage various research perspectives on K and DPand their bio-composites:

- Initially, the samples of bio-mortars reinforced with K compared with the reference mortar (without fibers), show either increases or decreases in the mechanical properties in terms of strength or stress in bending or compression.
- The highest stress is evaluated by 1% of Kenaf fiber reinforcement with a length of 5 mm. This increase is reflected in a value of 20.96% and 21.5% in flexural and compressive strength respectively.
- Slight increases in the mechanical properties were obtained for the bio-mortar samples reinforced with 5% date palm fiber of 5 mm length, translated respectively by 2.41% and 3.5% in the 3-point bending test and, in the compression, test compared with the reference mortar.
- Taguchi's methodology was used to determine the classification of the influence of each processing factor for Kenaf (K) and Date Palm (DP) raw fibers on the mechanical responses for the signal-to-noise ratios (prefer the greater). It should be noted that the percentage (%) fiber incorporation factor for both types of fiber had a greater effect than the fiber length factor.
- The analysis of variance ANOVA performed by Minitabe 17, provided information on the relative significance attributed to each parameter of either the flexural or compressive test, and those on the mechanical properties of K or DP reinforced mortars. In this context, given the reliability evaluation index F-value and the probabilistic parameter P-value measured and compared with the experimental data established, the independent parameter fiber concentration (%), is considered significant for the above response, while the analyzed length parameter is considered non-significant.
- On the other hand, the resulting mechanical properties of the bending and compression test, processed following an experimental programme established by the Taguchi L9 statistical method, highlighted the influence of the NaOH alkaline treatment and the immersion time of the K and DP in the alkaline solution. They showed that an increase or decrease in mechanical properties was obtained. The best mechanical properties, in terms of ultimate stress for both tests, were obtained following treatment with 1% NaOH for 1 hour of immersion time, and with 5% NaOH for 4 hours of immersion time, leading to an increase of 32.5% and 29.3% respectively in flexural strength and compressive strength compared with untreated K and DP.
- For the manufacturing parameters of DP-reinforced bio-mortars that are chemically treated, the results show that the mechanical strength in terms of ultimate bending stress is evaluated under the conditions of: 5% fiber reinforcement, 5 mm length, 5% NaOH treated for 4 hours immersion time. This analysis is always carried out in the case of compressive strength evaluation.
- The classification of the degree of influence of each processing factor for chemically treated Kenaf (K) fibers on mechanical responses (ultimate stress), shows that the percentage (%) factor of fiber incorporation has a greater effect (rank 1) for bending stresses, and a medium effect (rank 2) for compressive stresses.
- The results obtained by ANOVA for ranking the influence of each processing factor for chemically treated date palm fibers show that the percentage fiber incorporation factor has a greater effect (rank 1) for bending and compressive stresses. On the other hand, rank 2 is reserved for immersion time and the alkaline element for bending and compressive stress respectively.

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References

[1] Benmansour, N. (2015). Development and characterization of local natural composites suitable for thermal insulation in homes. Doctoral dissertation, UB1.
[2] Jules, J., Paul, B., Adam, M., & Andrieu, N. (2023). Co-design with producers of adaptation strategies to climate change: the case of farms in Haiti. Cahiers Agricultures, 32, 27.
[3] Kamouche, F. and Mokrane, A. (2022). Energy efficiency policy in the building sector: theoretical and empirical elements, case of Algeria.
[4] Fares, K. A. M. O. U. C. H. E., & Ali, M. O. K. R. A. N. E. (2022). Energy efficiency policy in the building sector: theoretical and empirical elements, case of Algeria. Journal of Financial, Accounting & Managerial Studies, 9(2).
[5] Salhi, R. (2023). Composite materials from natural resources: formulation, characterization and future potential. Doctoral dissertation.
[6] Tran Le, A.D. (2010). Study of hygrothermal transfers in hemp concrete and their application to building. PhD thesis, University of Reims Champagne- Ardenne, France.
[7] Bourhaneddine, H. (2017). In-situ study of the energetic and mechanical performances of biosourced and local materials. Doctoral Theses in Material Sciences (physics), El-Hadj Lakhdar University - BATNA 1.
[8] Baley, C. (2003). Contribution to the study of organic matrix composite materials reinforced with flax fibers. HDR thesis. University of South Brittany, Lorient, France.
[9] Bourgeois, M. (2011). Agro-sourced fibers. In Engineering techniques. Engineering techniques.
[10] Jonathan Page. (2018). Formulation and characterization of a biofibrous cementitious composite for prefabricated construction processes. PhD thesis, https://tel.archives-ouvertes.fr/tel-01713160.
[11] Abdelmouleh, M., Boufi, S., Belgacem, M.N., Dufresne, A. (2007). Short natural-fibre reinforced polyethylene and natural rubber composites: Effect of silane coupling agents and fibres loading. Composites Science and Technology 67, 1627-1639.
[12] Suganti Ramarad. (2008). Preparation and properties of kenaf bast fiber filled (plasticized) poly(lacticacid)composites. Master of science.
[13] Baley, C. (2004). Natural reinforcing fibres for composite materials. Technique de l'ingénieure, traité génie des procédés, Vol. AM5, 2004, pp. 130.
[14] Doan Thi, T.L. (2006). Investigation of jute fibres and their composites based on polypropylene and epoxy matrices. PhD thesis, University of Dresden.
[15] Mohammad Dalour, H.B. (2007). Improved interfacial bonding, weatherability and recycling of wood fibre reinforced polypropylene composites. PhD thesis, University of Waikato, Hamilton, New Zealand.
[16] Aziz Saaidia. (2018). Study of the influence of polyester resin on the mechanical behaviour of jute fibre reinforced composite. PhD thesis in Mechanical Engineering, University of 8 MAY 1945 GUELMA.
[17] Panthapulakkal, S., Zereshkian, A., Sain, M. (2006). Preparation and characterization of wheat straw fibers for reinforcing application in injection molded thermoplastic composites. Bioresource Technology; 97:265–72. https://doi.org/10.1016/j.biortech.2005.02.043.
[18] Satyanarayana, K.G., Arizaga, G.G.C., Wypych, F. (2009). Biodegradable composites based on lignocellulosic fibers—An overview. Progress in Polymer Science; 34:982–1021. https://doi.org/10.1016/J.PROGPOLYMSCI.2008.12.002.
[19] Dicker, M.P.M., Duckworth, P.F., Baker, A.B., Francois, G., Hazzard, M.K., Weaver, P.M. (2014). Green composites: A review of material attributes and complementary applications. Composites Part A: Applied Science and Manufacturing; 56:280–9. https://doi.org/10.1016/J.COMPOSITESA.2013.10.014.
[20] Faruk, O., Bledzki, A.K., Fink, H.P., Sain, M. (2012). Biocomposites reinforced with natural fibers: Progress in Polymer Science, 2000-2010; 37:1552–96. https://doi.org/10.1016/j.progpolymsci.2012.04.003.
[21] Li, X., Lope, A.E., Ae, G.T., Panigrahi, S. (2007). Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review n.d. https://doi.org/10.1007/s10924-006-0042-3.
[22] Fuqua, M.A., Huo, S., Ulven, C.A. (2012). Natural Fiber Reinforced Composites. Polymer Reviews; 52:259–320. https://doi.org/10.1080/15583724.2012.705409.
[23] Jawaid, M., Abdul Khalil, H.P.S. (2011). Cellulosic/synthetic fibre reinforced polymer hybrid composites: A review. Carbohydrate Polymers; 86:1–18. https://doi.org/10.1016/J.CARBPOL.2011.04.043.
[24] Malkapuram, R., Kumar, V., Negi, Y.S. (2008). Recent Development in Natural Fiber Reinforced Polypropylene Composites: Http://DxDoiOrg/101177/0731684407087759;28:1169– 89. https://doi.org/10.1177/0731684407087759.
[25] Sreekumar, P.A., Thomas, S.P., Saiter, J.M., Joseph, K., Unnikrishnan, G., Thomas, S. (2009). Effect of fiber surface modification on the mechanical and water absorption characteristics of sisal/polyester composites fabricated by resin

transfer molding. Composites Part A: Applied Science and Manufacturing; 40:1777–84. https://doi.org/10.1016/J.COMPOSITESA.2009.08.013.
[26] Carr, D.J., Cruthers, N.M., Laing, R.M., Niven, B.E. (2016). Fibers from Three Cultivars of New Zealand Flax (Phormium tenax): Http://DxDoiOrg/101177/004051750507500201 2016; 75:93–8. https://doi.org/10.1177/004051750507500201.
[27] Swamy, R.P., Kumar, G.C.M., Vrushabhendrappa, Y., Joseph, V. (2004). Study of Areca-Reinforced Phenol Formaldehyde Composites: Http://DxDoiOrg/101177/0731684404037049 2004; 23:1373–82. https://doi.org/10.1177/0731684404037049.
[28] Mwaikambo, L.Y., Ansell, M.P. (2002). Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization. Journal of Applied Polymer Science; 84:2222–34. https://doi.org/10.1002/app.10460.
[29] Hill, C.A.S., Khalil, H.P.S.A. (2000). Effect of Fiber Treatments on Mechanical Properties of Coir or Oil Palm Fiber Reinforced Polyester Composites. https://doi.org/10.1002/1097-4628.
[30] Theapsak, S., Watthanaphanit, A., Rajaraman, R. (2012). Preparation of Chitosan-Coated Polyethylene Packaging Films by DBD Plasma Treatment. ACS Applied Materials and Interfaces; 4:2474–82. https://doi.org/10.1021/AM300168A.
[31] Bledzki, A.K., Mamun, A.A., Volk, J. (2010). Physical, chemical and surface properties of wheat husk, rye husk and soft wood and their polypropylene composites. Composites Part A: Applied Science and Manufacturing; 41:480–8. https://doi.org/10.1016/J.COMPOSITESA.2009.12.004.
[32] Sadov, F., Korchagin, M., Matetsky, A. (1978). Chemical technology of fibrousmaterials.
[33] Ofomaja, A.E., Naidoo, E.B. (2011). Biosorption of copper from aqueous solution by chemical lyactivated Pinecone: A kineticstudy, Chem Eng J; 175:260– 270.
[34] Fung, J.C.M. (2009). A New Composite Material Consisting of Flax Fibers, Recycled Tire Rubber and Thermoplastic. Thesis.
[35] Yanjun, X., Qinglin, W., Yong, L.Y. (2010). Creep behavior of Bagasse fiber reinforced polymer composites, Biore source Technol; 101: 3280–3286.
[36] Bouhemame Nasser. (2021). Study of polymer matrix composite materials filled with Date Palm fibres. PhD thesis in Mechanical Engineering, University of Kasdi Merbah Ouargla.
[37] Stamboulis, A., Baillie, C.A., Peijs, T. (2001). Effects of environmental conditions on mechanical and physical properties of flax fibers. Composites Part A: Applied Science and Manufacturing; 32:1105–15. https://doi.org/10.1016/S1359-835X(01)00032-X.
[38] Barkoula, N.M., Garkhail, S.K., Peijs, T. (2009). Effect of Compounding and Injection Molding on the Mechanical Properties of Flax Fiber Polypropylene Composites. Http://DxDoiOrg/101177/0731684409104465;29:1366–85. https://doi.org/10.1177/0731684409104465.
[39] Graupner, N., Herrmann, A.S., Müssig, J. (2009). Natural and man-made cellulose fibre-reinforced poly (lactic acid) (PLA) composites: An overview about mechanical characteristics and application areas. Composites Part A: Applied Science and Manufacturing; 40: 810–21. https://doi.org/10.1016/J.COMPOSITESA.2009.04.003.
[40] Satyanarayana, K.G., Sukumaran, K., Kulkarni, A.G., Pillai, S.G.K., Rohatgi, P.K. (1983). Performance of Banan Fabric-Polyester Resin Composites :535–48. https://doi.org/10.1007/978-94-009-6640-6_38.
[41] Shibata, S., Cao, Y., Fukumoto, I. (2005). Press forming of short natural fiber-reinforced biodegradable resin: Effects of fiber volume and length on flexural properties. Polymer Testing; 24:1005–11. https://doi.org/10.1016/J.POLYMERTESTING.2005.07.012.
[42] Jayaraman, K. (2003). Manufacturing sisal–polypropylene composites with minimum fibre degradation. Composites Science and Technology; 63:367–74. https://doi.org/10.1016/S0266-3538(02)00217-8.
[43] Satyanarayana, K.G., Sukumaran, K., Kulkarni, A.G., Pillai, S.G.K., Rohatgi, P.K. (1986). Fabrication and properties of natural fibre-reinforced polyester composites. Composites; 17:329–33. https://doi.org/10.1016/0010-4361(86)90750-0.
[44] Tomczak, F., Sydenstricker, T.H.D., Satyanarayana, K.G. (2007). Studies on lignocellulosic fibers of Brazil. Part II: Morphology and properties of Brazilian coconut fibers. Composites Part A: Applied Science and Manufacturing; 38:1710–21. https://doi.org/10.1016/J.COMPOSITESA.2007.02.004.
[45] Dungani, R., Khalil, H.P.S.A., Sumardi, I., Suhaya, Y., Sulistyawati, E., Islam, M.D.N., Suraya, N.L.M., Aprilia, A.S. (2014). Non-wood renewable materials: properties improvement and its application. In K.R. Hakeem, M. Jawaid et U. Rashid, Biomass and Bioenergy: Applications, (p. 1-30). Springer, London.
[46] Nanou, I., Yousfi, H. (2020). Comparative study on date palm fibre composites. Master's thesis in Materials Engineering, Université Mohamed Boudiaf de M'sila.
[47] Tolêdo Filho, R.D., Scrivener, K., England, G.L., Ghavami, K. (2000). Durability of alkali-sensitive sisal and coconut fibers in cement mortar composites. Cement and Concrete Composites, quoted by [MAG10], Volume 2 (2000) 127-143.
[48] Baley, C., Grohens, Y., Levesque, G., Busnel, F., Pommel, C., Sire, O. (2002). Influence of chemical treatments on the interfacial properties and adhesion of the flax fibre-polyester resin system. Matériaux.
[49] Claire, S., Peyratout, M., Le Troëdec, T., Chotard, J. (2007). Pierre Bonnet, A. Smith - Physico-chemical modification of interactions between hemp fibres and lime: impacts on the mechanical properties of mortars. 18éme congrès Français de Mécanique, 27-31 08.
[50] Bahloul, O., Bourzam, A., Bahloul, A. (2009). Use of plant fibres in the reinforcement of cement mortars. SBEIDCO, 1st International Conference on Sustainable Built Environment Infrastructures in Developing Countries ENSET Oran (Algeria) - October 12-14.

[51] Rokbi, M., Osmani, H. (2011). The effect of fibre surface treatments on the mechanical properties of Polyester-Alfa fibre composites. 20th French Mechanical Congress, 29 August to 02 September 2011.

[52] Merzoud, M. (2007). Elaboration and characterisation of a composite material based on diss fibres in the manufacture of masonry. PhD thesis, Badji Mokhtar University, pp 123.

[53] Fávaro, S.L., Ganzerli, T.A., de Carvalho Neto, A.G.V., da Silva, O.R.R.F., Radovanovic, E. (2010). Chemical, morphological and mechanical analysis of sisal fiber reinforced recycled high- density polyethylene composites, Express Polymer Letters,4; 465– 473.

[54] Upadhyaya, P., Garg, M., Kumar, V., Nema, A.K. (2012). The Effect of Water Absorption on Mechanical Properties of Wood Flour/Wheat Husk Polypropylene Hybrid Composites, Materials Sciences and Applications, 3; 317-325.

[55] Tran, H.N., Ogihara, S., Tung, N.H., Kobayashi, S. (2011). Effect of alkali treatment on interfacial and mechanical properties of coir fiber reinforced poly (butylene succinate) biodegradable composites, Composites: Part B, 42; 1648–1656, 2011.

[56] Tranle, A.D. (2010). Study of hygrothermal transfers in hemp concrete and their application to building. Doctoral thesis, University of Reims Champagne- Ardenne, pp. 221.

[57] Salit, M.S. (2014). Tropical Natural Fibre Composites : Properties, Manufacture and Applications. Springer. Singapore, pp. 53-103.

[58] Davis, J.R. (2004). Tensile Testing. 2nd Edition, ASM intemational, pp. 4-183.

[59] Bazergui, A. (2002). Résistance des matériaux, third edition. Presses lntemationales Polytechnique. p. 258.

[60] Mallick, P.K., M. Dekker. (1997). Composites Engineering Hand book. Marcel Dekker Inc. United States of America, pp. 1-129.

[61] ACI. (1996). Report on Fiber Reinforced Concrete (Reapproved). American Concrete Institute. Agopyan, V., H. Savatant, V. M. John, et M. A. Cincotto. Developments on vegetable fibre– cement based materials in São Paulo, Brazil: an over view. Cement and Concrete Composites 27, 527-536, (1996).

[62] Taoukil, D., Bouardi, A.E., Ajzoul, T., Ezbakhe, H. (2012). Effect of the Incorporation of Wood Wool on Thermo Physical Proprieties of and Mortars. KSCE, Journal of Civil Engineering, vol. 16, pp. 1003-1010.

[63] Patural, L. (2011). Method of action of cellulose ethers on the water retention of mortars in the fresh state. PhD thesis, Ecole Nationale Supérieure des Mines de Saint- Etienne, pp. 262.

[64] Aziz, M.A., Paramasivam, P., Lee, S.L. (1984). New Reinforced Concretes: 3. Concrete reinforced with natural fibres. Surrey University Press.

[65] Kriker, A., Debicki, G., Bali, A., Khenfer, M.M., Chabannet, M. (2005). Mechanical properties of date palm fibres and concrete reinforced with date palm fibres in hot dry climate. Cement and Concrete Composites 27, 554-564.

[66] EN 196-1. (2005). Methods of testing cement-part 1: Determination of stress, European committee for standarization.

[67] Li, D., Ji, D., Liu, J., Lin, Y. (2016). A Multi-Model EKF Integrated Navigation Algorithm for Deep Water AUV. International Journal of Advanced Robotic Systems ;13. https://doi.org/10.5772/62076.

[68] Babaki, M., Yousefi, M., Habibi, Z., Mohammadi, M. (2017). Process optimization for biodiesel production from waste cooking oil using multi-enzyme systems through response surface methodology. Renewable Energy; 105:465–72. https://doi.org/10.1016/j.renene.2016.12. 086. of testing cement-part 1: Determination of stress, European committee for standardization, 2005.

[69] ASTM C109 / C109M –Standard Test Method for Compressive strength of Hydraulic Cement Mortars (Using 2-in. Or [50mm] Cube Specimens), https: //www.astm.org/Standards/C109.

[70] ASTM C348-14, Standard Test Method for Flexural Stress of Hydraulic- Cement Mortars, ASTM International, West Conshohocken, PA, 2014, www. Astm.org.