

Mineralogical, Geotechnical and Mechanical Characterization of Clay/Alfa Short Fiber Composites for Sustainable Building

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Abstract

The present work aims to investigate experimentally and analytically the mechanical behavior of a new and ecological material based on clay reinforced with different mass percentages of short Alfa fiber (0%, 1%, 2%, 3%, and 4%). The characterization was carried out by X-ray diffraction (XRD), X-ray fluorescence (XRF), and infrared spectroscopy, as well as by microscopic observations using scanning electron microscopy (SEM). The results indicated that the clay consists of a significant proportion of kaolinite, quartz, and muscovite. Moreover, the addition of Alfa fibers to the clay resulted in a decrease in compression strength. This trend is justified by the length of fibers, which is insufficient to prevent the propagation of cracks. Also, this decrease can be attributed to porosity, where samples with higher fiber content induce more voids that were entrapped during the mixing process. On the other hand, the reinforced samples resist beyond the breaking load. Indeed, this resistance is due to the transfer of internal forces from the matrix to the fibers.

Keywords

Alfa fiber, Clay, Mechanical characterization

Introduction

The growing awareness of socioeconomic and environmental issues related to the impacts of climate change has fueled efforts to develop innovative, new, and environmentally friendly materials [1, 2]. Bio-composite materials, namely clay-based natural fiber bio-composites, have become the center of attention over the past few years. This attention is primarily due to several factors, such as their high strength and stiffness, low density, low cost, availability, biodegradability, and low environmental impact [3, 4]. These advantages have attracted great interest from both the academic world and various industries, specifically the construction and building industries. Traditionally, composite materials are reinforced with synthetic fibers such as glass, carbon, ceramic, or aramid fibers. However, these fibers are not biodegradable and cannot be recycled at the end of their life [5]. In the literature, there are other studies that discuss the physical and mechanical properties of similar materials [6, 7]. Furthermore, their production requires a significant amount of energy and the use of non-renewable resources, which are generally accompanied by the production of harmful by-products. These findings have therefore encouraged the emergence of bio-composite materials reinforced with natural fibers. Nowadays, a variety of natural fibers are being utilized in the composite industry to replace synthetic fibers, such as hemp, flax, jute, bamboo, palm, and Alfa [2]. Due to their outstanding properties, natural fiber-based composites offer very promising perspectives. But despite their multiple benefits, they also have negative points and limitations that compromise their use [8, 9]. For

example, the poor compatibility and adhesion of fibers with certain polymers, poor mechanical properties, and porosity are drawbacks that may lead to undesirable properties of the composites. In this paper, the mechanical characterization of an eco-friendly composite material from clay and short Alfa fibers has been performed.

Materials and Method

Materials

Kaolin

All used materials are nanoscale materials. The kaolin used in this study is a material marketed by a Moroccan company in Casablanca. Our laboratory carried out its principal physical, chemical, and geotechnical characterization tests, following international standards and recommendations. Indeed, the knowledge of this material's properties and characteristics is essential for identifying the potential performances taken in the manufacture of new products or the improvement of existing products, such as raw clay bricks and terracotta clay bricks. Granulometric analysis was determined using sieving for particles larger than 0.063 mm in diameter and laser particle size analysis for the finest particles. The sieving test was performed under the standard ISO 17892-4:2016 [10]. In addition, the particle size distribution was performed by the BT-9300S laser particle size analyzer. Figure 1 illustrates the granulometric curve of the kaolin clay used for sample production. Figure 1 shows that kaolin is primarily a very fine-grained soil. It contains a significant number of fine particles, about 95% of the total mass (Particle passing the 0.063 mm sieve). Following the distribution of granular fractions defined by the standard ISO14688-1:2002 [11], the results indicated that the kaolin mainly comprises 5.69% clay fraction, 8% fine silt, 20% medium silt, 61% coarse silt, and 5.2% sand. The Atterberg limit test was conducted to find the plastic and liquid limits according to the standard NF P94-051 [12]. The test results demon-

strated that kaolin has a liquid limit of 150%, a plastic limit of 55%, and a plasticity index of 95%. In addition, the methylene blue index was determined according to the standard NF EN 933-9+A1 [13]. It was found that the value of the methylene blue and the specific surface area for kaolin are about 0.4 and 15 m²/g, respectively. The calcium carbonate (CaCO₃) content was determined according to the French standard NF P94-048 [14], using a Dietrich-Fruhling calcimeter. The carbonate content was 6.14%, showing that kaolin is classified as non-calcareous since the calcium carbonate content does not exceed 10%. The solid particle density was determined using helium pycnometry according to the standards NF P94-054 [15] and NF EN ISO 17892-3 [16]. The results revealed that the particle density of kaolin was approximately 2.51 g/cm³. The mineralogical examination using the XRD technique (Rigaku Smart Lab X-ray Diffractometer) was carried out to study the composition of the kaolin sample. The XRD patterns in figure 2 show that kaolin is composed of quartz, kaolinite, and muscovite. A low proportion of quartz was found in the sample, which leads to high plasticity, as shown by the Atterberg limit test.

The chemical composition test was conducted using the XRF technique (Epsilon 4 Benchtop XRF-EDS) to investigate the sample's composition. Kaolin was calcined at 1100 °C for 24 h. The oxide composition and loss on ignition (LOI), presented in table 1, shows that the kaolin contained a significant amount of silica, primarily from alumina silicates and quartz. It also has a high concentration of Al₂O₃, which is generally bound to silicate clay and contributes to good plasticity. Other trace oxides (Mg, Na, Ti, etc.) have been detected in the chemical analysis of the sample.

The microstructure of the kaolin was observed and analyzed using a Tescan Vega SEM. Figure 3 shows the microscopic images of the sample. The microscopic observation indicated that the kaolin has a relatively compact texture, formed by several overlapping flat sheets exhibiting turbostratic disor-

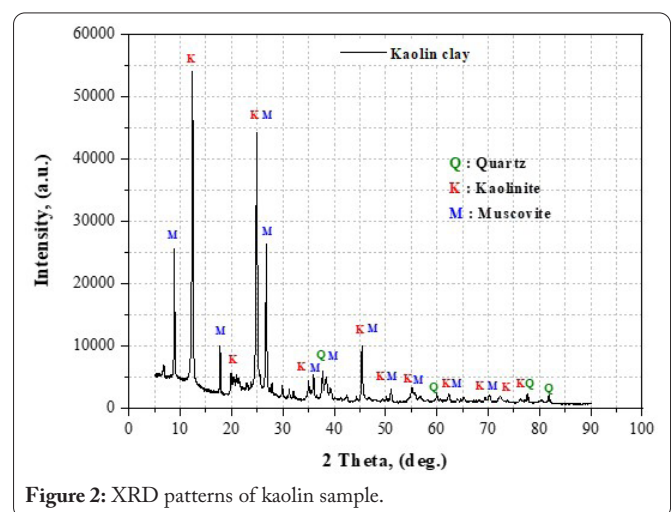
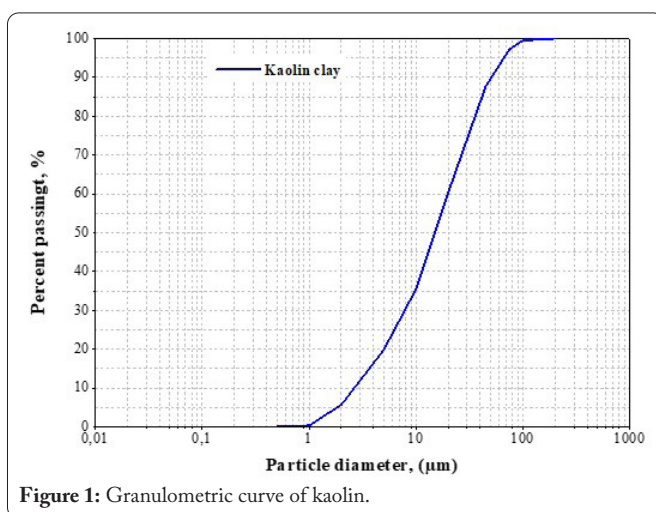


Table 1: Chemical composition of kaolin clay.

Chemical composition (%)	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	LOI
Kaolin clay	48.1	0.20	36.00	1.40	0.20	0.10	0,10	2.40	11.50

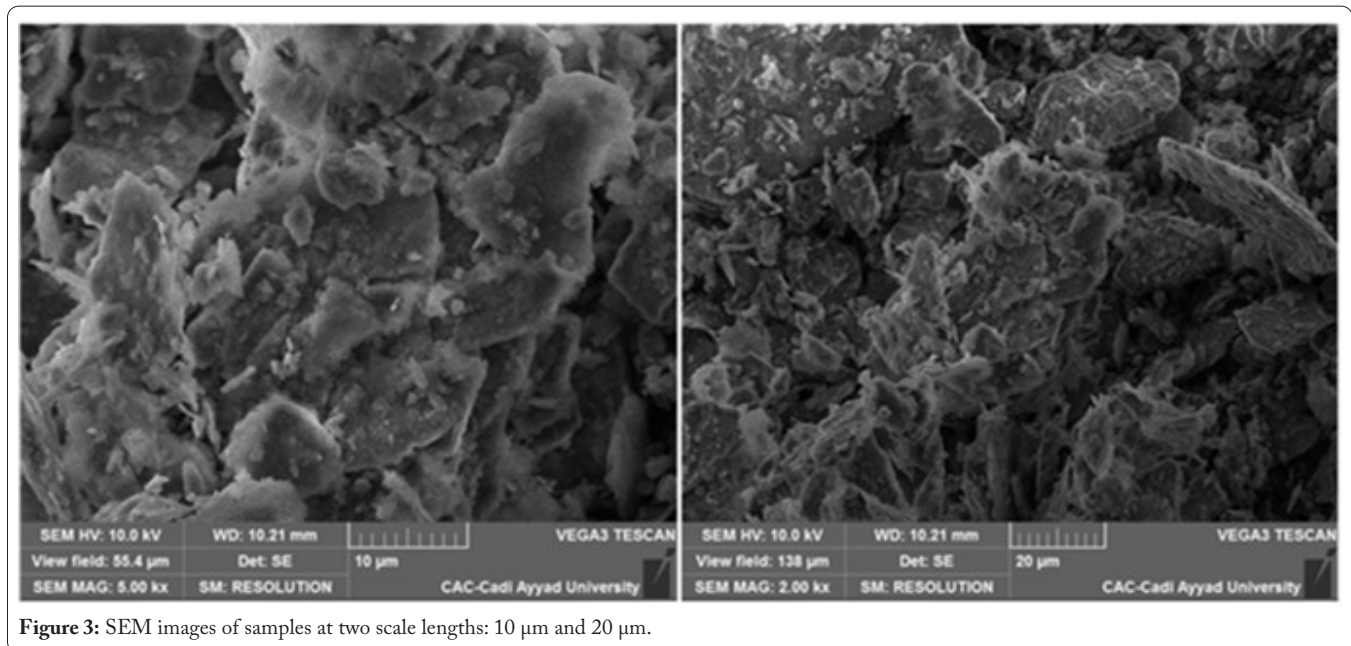


Figure 3: SEM images of samples at two scale lengths: 10 μm and 20 μm.

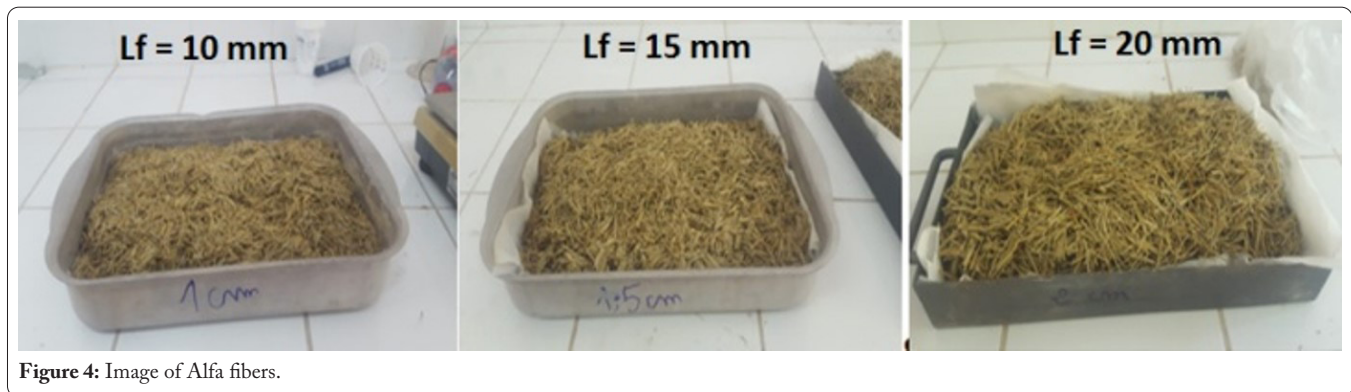


Figure 4: Image of Alfa fibers.

der. It was observed that the kaolin matrix is dense with some pores and micro-cracks.

From the results of the energy dispersive X-ray analysis, the following chemical elements are observed: O, Si, Al, Na, Mg, Fe, Ca, K, and Ti. The crystalline phases are formed mainly by quartz, kaolinite, and muscovite.

Alfa fiber

The Alfa plant, also known as esparto grass stems (*Stipa tenacissima*), is a typically Mediterranean perennial herb that grows in clumps about 1 m to 1.2 m high. It is a large grass widespread in north Mediterranean Africa and the dry regions. The Alfa fibers used in our study are extracted from the Oujda region in northeast Morocco. The fibers are prepared in several stages. First, the stems are washed with water to remove dust and impurities deposited on the surface. Subsequently, they are air-dried at ambient temperature for 72 h, which will eliminate most of the moisture they contain. The dried stems are cut into short fibers with 1 mm diameter and lengths of 10 mm, 15 mm, and 20 mm. This operation is followed without grinding. The resulting fibers are designated as untreated fibers (Figure 4). The SEM representation of the external surface of Alfa fiber is shown in figure 5.

The crystallinity index (C_rI) based on the “Segal method

[17]” was calculated from the XRD analysis using the following equation:

$$C_r I (\%) = \frac{100 \times (I_{002} - I_{am})}{I_{002}}$$

Where I_{002} is the maximum peak intensity of the crystalline phase corresponding to the crystallographic plane (002) of cellulose (I) and I_{am} is the minimum intensity of the amorphous form. Table 2 shows the C_rI values of Alfa fiber.

The Alfa fiber diffractogram obtained from an XRD test is shown in figure 6. It was remarked that the spectrum contains two prominent peaks. The first one is located between 13.20°

Table 2: C_rI of Alfa fiber.

Alfa plant fiber	I_{002}	I_{am}	C _r I (%)
Alfa fiber	47980	30200	37

and 17.65°, corresponding to the crystallographic plane (110). The second one appeared at 22°, associated with a crystallographic plane of (002). These two peaks are characteristic of the presence of cellulose I.

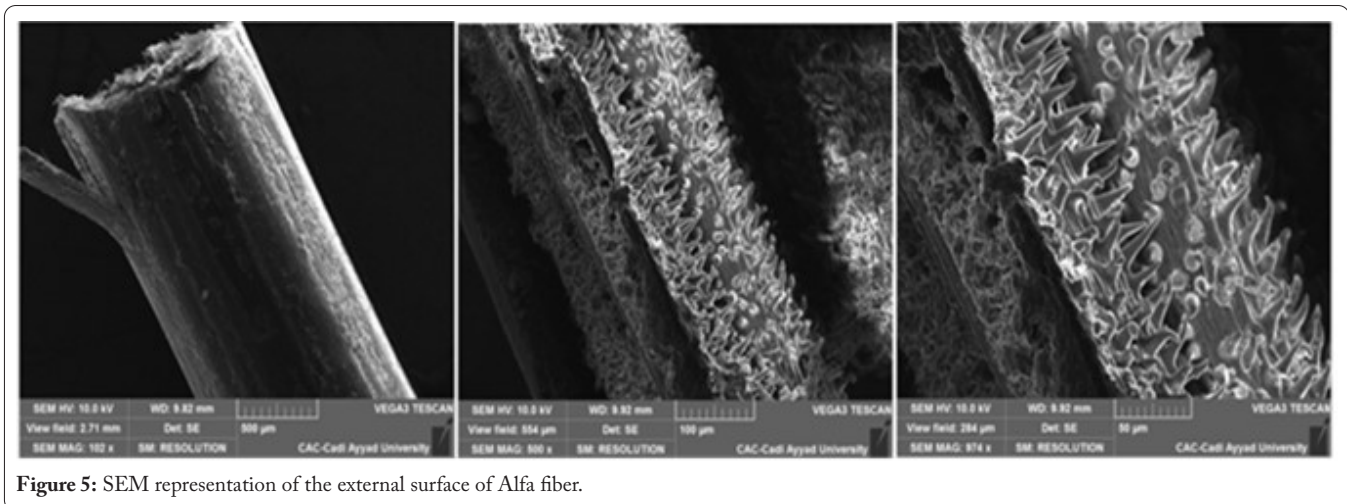


Figure 5: SEM representation of the external surface of Alfa fiber.

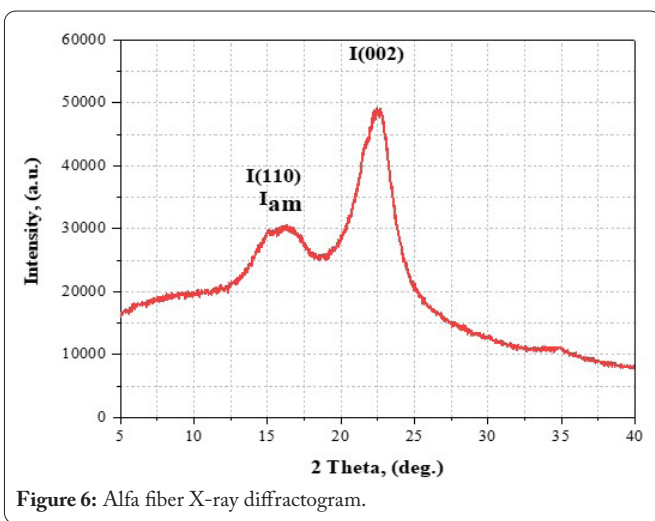


Figure 6: Alfa fiber X-ray diffractogram.

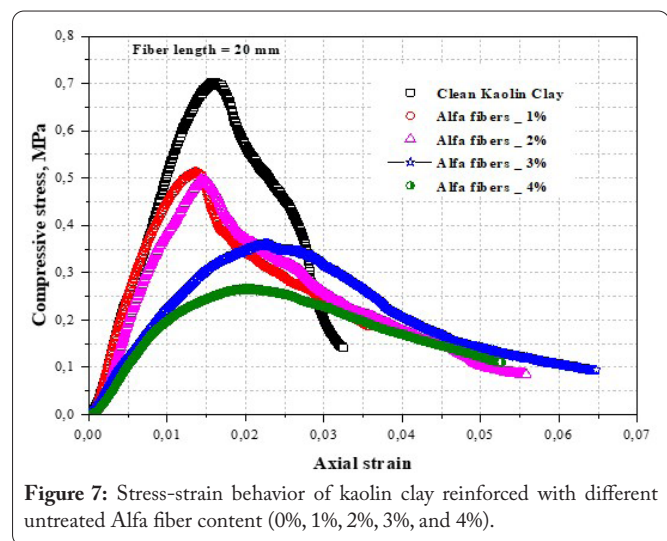


Figure 7: Stress-strain behavior of kaolin clay reinforced with different untreated Alfa fiber content (0%, 1%, 2%, 3%, and 4%).

Simple compression test

The specimens tested were in a cylindrical shape of 30 mm diameter x 60 mm height. A compaction level of 2 MPa was applied to fabricate the previous samples. The soil moisture content in volume percent was about 10%. Compression tests were carried out to determine the compressive strength. An Instron hydraulic press with a 50 kN load cell at a constant speed of 0.25 mm/min was used. The machine is an automatic device with software that processes and displays the results on a computer. The results are retrieved and analyzed afterward.

Results and Discussion

Effect of Alfa fibers content on compressive strength

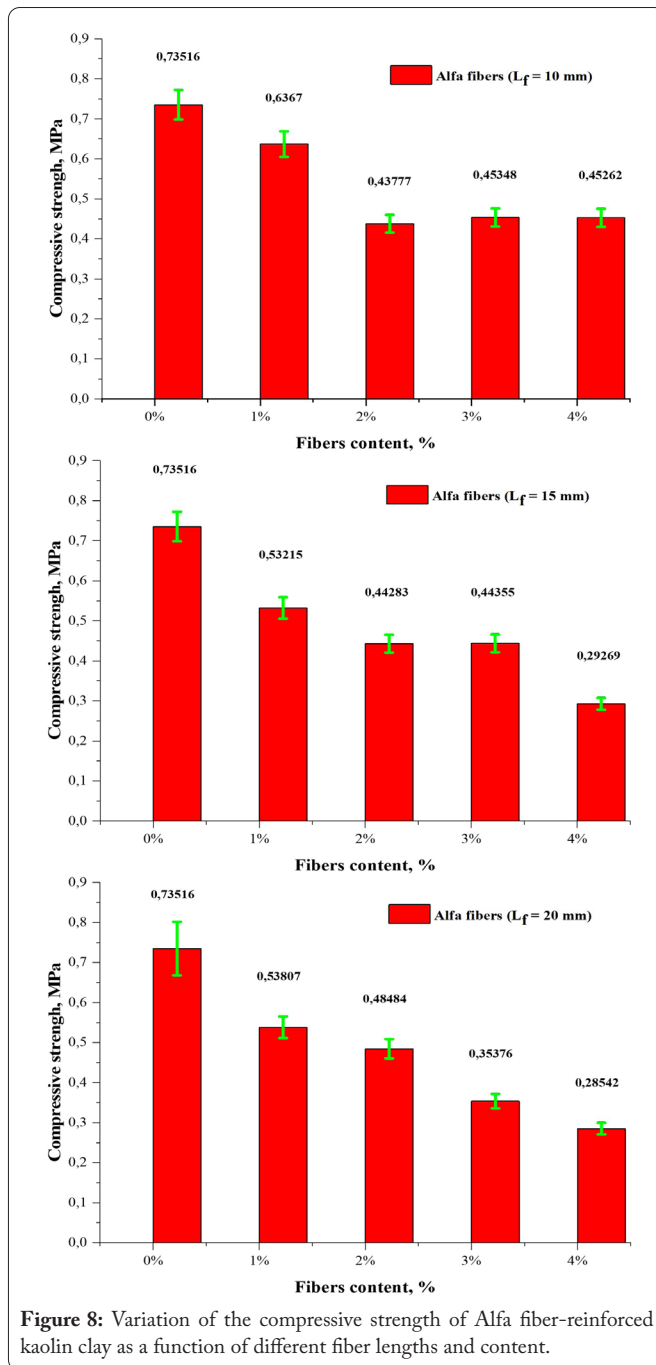
The stress-strain curves for reinforced kaolin with an Alfa fiber content of 20 mm length are shown in figure 7. The results indicated that adding fibers to the clay matrix decreases the compressive strength. This could be due to the lower density in the reinforced sample compared with the unreinforced sample. Otherwise, it can be explained by a change in soil structure and the formation of pores. Poor fiber-matrix bonding may also increase micro cracks in the fiber-matrix transition zone. In addition, it was found that the incorporation of fibers gives the mixture a higher ductility than pure kaolin clay. This might be due to the bond between fibers and kaolin clay,

which requires more energy to break bonds.

Figure 8 shows the variation of the compressive strength of Alfa fiber-reinforced kaolin clay for different fiber length sand content. The compressive strength decreases by adding Alfa fiber, regardless of the length considered (10 mm, 15 mm, and 20 mm). This trend is justified by the length of fibers, which is insufficient to prevent the propagation of cracks. Also, this decrease can be attributed to porosity, where samples with higher fiber content induce more voids that were entrapped during the mixing process. It can also be explained by the effect of size, surface condition, and the number of fibers present in a given volume. Therefore, increasing the content of Alfa fibers leads to a decrease in bond strength, resulting in a reduction in compressive strength. Finally, it was remarked that the failure of unreinforced samples occurred quickly and brutally. On the other hand, the reinforced samples resist beyond the breaking load. Indeed, this resistance is due to the transfer of internal forces from the matrix to the fibers.

Effect of Alfa fiber content on Young's modulus

By analyzing the influence of Alfa fiber percentage on Young's modulus for different fiber lengths, it was observed that Young's modulus was not improved by increasing fiber percentage. For example, for a fiber length of 20 mm, Young's



modulus drops by 42.73% when the fiber content augments from 1% to 3%. Moreover, it was noticed that the impact of fiber percentage on the elastic modulus is stronger than on the tensile strength.

Conclusion

The present work has been developed along two fundamental lines. The first consists of an experimental study on kaolin clay reinforced Alfa fibers composite. The objective of the first part was to analyze and understand this new composite's physical and mechanical properties for an Alfa fiber mass ratio ranging from 0% to 4%. It allowed for highlighting the influence of microstructural parameters such as fiber content and length on the compressive strength and Young's

modulus of the composite. The compressive strength decreases by adding Alfa fiber, regardless of the length considered (10 mm, 15 mm, and 20 mm). This trend is justified by the length of fibers, which is insufficient to prevent the propagation of cracks. Also, this decrease can be attributed to porosity, where samples with higher fiber content induce more voids that were entrapped during the mixing process. It can also be explained by the effect of size, surface condition, and the number of fibers present in a given volume. Therefore, increasing the content of Alfa fibers leads to a decrease in bond strength, resulting in a reduction in compressive strength. Finally, it was remarked that the failure of unreinforced samples occurred quickly and brutally. On the other hand, the reinforced samples resist beyond the breaking load. Indeed, this resistance is due to the transfer of internal forces from the matrix to the fibers.

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None.

Conflict of Interest

On behalf of all authors, the corresponding authors states that there is no conflict of interest.

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