Structural, Mechanical and Morphological Analysis of Treated Sisal Fibers and Cellulose Extracted from Sisal and it effect on Improving the Plaster-based Composites Mechanical Properties

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Abstract

In the context of attempts to protect the environment and the ongoing exhaustion of fossil resources, using biomass raises significant environmental concerns. From this approach, sisal fibers and the cellulose extracted from them are used as bio-reinforcement in the production of composite materials. The first part of this paper will examine the effects of sodium hydroxide treatment on the mechanical characteristics of natural sisal fibers. Then, the morphological properties of the surface of the fibers were determined using scanning electron microscopy (SEM). X-ray diffraction was performed on the treated and untreated fibers. The results showed that the cellulose and the treated fibers had a greater crystallinity index than the untreated fibers. The results of the FTIR spectroscopic examination were strongly congruent with the results of the XRD and SEM analyses. The ATG, which was performed on both natural sisal fibers, treated sisal fibers, and cellulose fibers, also showed that treatment of the fibers increased their thermal resistance and stability. According to research on composite materials made of plaster reinforced by fibers, increasing the sisal treated fibers content in plaster molasses to 1.25% led to a greater improvement in the mechanical and physical characteristics with a rise of 35% in resistance to bending. Additionally, when applying the cellulose fibers extracted from sisal as a reinforcement to the plaster-based composite material, we obtain an incensement in the resistance bending by 48%.

1. Introduction

Major interest in the use of bio-resources in the domain of materials in the last years. As materials designed for industrial uses or for civil engineering have grown during the past three decades. Due to a number of factors and benefits, such as strong mechanical and physical qualities, cheap cost, low density, biodegradable, non-toxic, and there are significant amounts that go wasted, the focus on plant fibers and plant wastes are of considerable interest [1–4].The development of biomaterials for use in a variety of industries, such as shipbuilding (composite with an epoxy resin matrix, polyester...), aviation, automotive (composite has a plastic matrix, polymer...), and even the composite developed for civil construction applications, such as composites based on cement and plaster, has received significant research interest[5–7]

Natural fibers and waste have replaced synthetic fibers as reinforcement for these composites because they have high mechanical qualities in their natural condition or as well in chemical, thermal, or mechanical treatments [8].

Therefore, cellulosic fibers bring many attractive advantages such as resource diversity, continuous renewal, abundance, low density, ease of processing and availability with different chemical compositions and morphologies, good specific mechanical properties, and end-of-life biodegradability among others [9]. Due to these major properties, a biomaterial such as bio-composites can be obtained by using a small amount (not exceeding 10% of the total weight of the material) of cellulosic fibers with improved properties [10]. In addition, the improved properties of the bio-composite depend on several critical parameters, such as the shape, size, polarity, distribution, content, and orientation of the reinforcing fiber), the adhesion matrix/reinforcement, and these physical properties (the density and the state of fiber surface). [11–12]

Cellulose is a natural polysaccharide, it is the most abundant biomass with favorable thermal and mechanical properties. natural cellulose also has a modulus of elasticity, E, of 138 GPa in the direction parallel to the chain axis for its crystalline regions. This is comparable to the modulus of elasticity of high-performance synthetic fibers such as poly (pphenylene terephthalamide) (156 GPa), Ekonol (130 GPa), Vectran (126 GPa)[13–14]. Also, Young's modulus of natural vegetable cellulose can reach up to 128 GPa (5) is strongly higher than the value of Young's modulus of aluminum which is limited by 70 GPa, and glass fibers which are equal to 76 GP [15]. Therefore, cellulose has been used in the manufacture of composite materials with success, it gives very encouraging results to continue research on the valuation of cellulose fibers and cellulose in the manufacturing of bio-based composites, especially in polymer composites reinforced by cellulose microfibers [15–18].

As mentioned above, the properties of cellulose are numerous and encouraging to be valorized in the field of bio-composites, especially its strong mechanical properties, and its morphology that can be a solution to the phenomenon of slippage of fibers in matrices. Therefore, this study will focus on the use of extracted cellulose from sisal fibers as reinforcement of bio-based composite materials for civil engineering applications (construction and building), with a focus on the properties of cellulosic fibers and the effect of chemical treatment by alkali on their mechanical, chemical, thermal and physical properties. The Extraction of cellulose from sisal is done according to TAPPI protocols. The effect of alkaline treatment with sodium hydroxide on the mechanical properties of natural sisal fibers such as tensile strength and elongation at break was studied. To study the effects of surface treatments, the surfaces of untreated, and treated fibers and the surface of the cellulose fibers were analyzed by SEM, EDX, and FTIR. a TGA thermogravimetric analysis was carried out and the crystallinity of the three materials was determined by XRD analysis. a bio-composite material based on a plaster matrix reinforced with sisal fibers treated with NaOH or cellulose fibers with different percentages (0.5%, 0.75%, 1.00%, 1.25%, 1.50%) whose purpose is to determine the optimum percentage of reinforcement to obtain the best mechanical and physical properties and also to compare between reinforcement methods (treated fiber or cellulose fibers). the flexural strength, flexural modulus, cracking behavior, and physical properties of this material such as density and absorption rate were studied.

2. Materials And Methods

2.1 Raw Material and Chemical products

The sisal plant fibers were chosen as the primary material of this project. In the Gafsa area (southern Tunisia), sisal leaves were gathered. Cuttings were used to detach them from the mother plant, and the sharpest edges were removed. then the fibers were extracted using nails to prevent any deterioration of the fibers that may happen during the use of the chemical extraction procedure. After the unit fibers were removed, the last one was washed with distilled water and dried at standard temperature.

Sodium chlorite, acetic acid, and sodium hydroxide have all been employed as bleaching agents. These substances were all bought from SIGMA ALDRICH and CARLO ERBA, and no additional purification was used.

2.2 Extraction of Cellulose from sisal Fibers

The extraction of cellulose from sisal fibers requires two steps, respectively delignification and bleaching. Dry fibers were treated with a 10% sodium hydroxide solution at 160°C for two hours to remove the majority of the lignin and hemicellulose. Then, water was employed to wash the acquired fibers. The final stage was taking the hemicellulose out of the fibers that had been extracted. The goal of this step was to obtain fibers with a high degree of purity. The fibers were combined with tampon acetate and a (1.7%) sodium chlorite solution. At 80°C, the reaction was carried out in the water while the suspension was mechanically agitated. After two hours, the suspension underwent filtering and washing with distilled water until its pH matched that of the water. All the procedure was carried out twice [19–20].

2.3 Chemical treatment

In order to compare the mechanical properties of the treated and untreated fibers, we employed sodium hydroxide to treat sisal fibers. We varied a number of parameters, including the concentration of the NaOH solution (1%, 2%, 3%, 4%, and 5%) and the reaction time (1h, 2h, 3h, 4h, and 5h). The natural fibers were first washed in distilled water, dried in an electric oven, and then treated to mercerization with NaOH at the conditions we already specified. The final step was a second wash of the treated specimens in distilled water. the chemical process, as seen in the accompanying figures.

2.4 Characterization

2.4.1 Strength test

Single fibers were subjected to tensile tests on a computer-guided Shimadzu tensile machine which recorded the tension pattern. According to ASTM D 3822-07, the strain rate was 2 mm/min. Gauge length (GL) was 40 mm between clips according to Mouna, et al. [21]), and measurements were taken at room temperature. Using the program (Trapeze), which automatically calculates all characters (based on force, deformation, and deformation). To determine the Tensile Strength (σ) which was calculated as follows the equation:

$$\sigma = \frac{F}{S}$$

1

Where S is the fiber's section, expressed in mm2, and F is the maximum breaking load, expressed in Newton. However, from the graph, we can deduce the strain (ε) and Young's modulus (E).

2.4.2 Chemical Composition

Sisal fibers' chemical composition was measured using established techniques and customary practices. The extracts were made using standard TAPPI procedures, namely extracts in hot and cold water (T207 cm-08), extracts in 1% sodium hydroxide solution (T212 om-07), and extracts in an ethanol-toluene system (T224 om-07). Then, the quantities of lignin and cellulose were measured using the TAPPI techniques T222 om-06 and T203 cm-99, respectively. Utilizing Álvarez et al. approach. [22], the quantity of holocellulose was calculated. The organic matter was calcined for 8 hours at 600°C in accordance with TAPPI Standard Method T221 om-07 to estimate the ash concentration.

2.4.3 Morphology Analysis(SEM)

The surface morphology of the natural fibers, the treated fibers, and the cellulose extracted from the Tunisian cactus plant is examined using a scanning electron microscope (SEM) brand JEOL JSM-IT 100, which has an acceleration voltage of 0.5 to 30 kW and a resolution of 4.5 nm. Before analysis, the samples' surfaces were coated with gold to make them conductive.)

2.4.4 Spectroscopic Analysis (DRX)

The crystallinity of all samples was evaluated at room temperature (22°C) using an X-ray diffractometer (D8-Advance Bruker AXS GmbH) with a monochromatic Cu Kα radiation source in step-scan mode with an angle 2θ (5–60°), with a current of 4 mA and a scan time of 5 min in the “research center of the Gafsa Phosphate Company” in Mettlaoui, Tunisia

2.4.6 FTIR

The three samples of untreated fibers (UN. F), alkali-treated fibers (NaOH. F), and cellulose fibers (CELL. F) were thoroughly ground before being formed into pellets for Fourier-transform infrared spectroscopy (FTIR) analysis at the “research laboratory -Nanomaterials and Systems for Renewable Energies-Borj Cédria Energy Research and Technology Center, Tunisia”. The scanning range was 400 to 4000 cm-1 using FTIR “infrared spectrometer EQUINOX 55”

2.4.7 TGA

Untreated fibers (UN. F), alkali-treated fibers (NaOH. F), and cellulose fibers (CELL.F) samples were all subjected to thermogravimetric analyses at the "Unit of Joint Service of Research - National School of Engineers of Gabes, University of Gabes, Tunisia." utilizing a “TGA/TDA 1600°C/DSC 800°C thermobalance” with a heating rate of 10°C/min in a nitrogen environment and a temperature range of 25 to 800°C

2.5 composite material making and its characterization

2.5.1 composite material making

according to the AFNOR standard EN13279-2, Test specimens of the material (plaster/cactus fibers) are created in the dimensions 40*40*140 mm with a hardening period of 28 days in order to determine the mechanical and physical proprieties of the composite. To discover the optimal technique of reinforcement, the composite was made utilizing two types of reinforcement obtained from natural sisal fibers: alkali-treated sisal fibers and cellulose extracted from sisal. Three samples for each of the six percentages (0.00%, 0.5%, 0.75%, 1.00%, 1.25%, 1.50%, and 1.75%) were created for each form of reinforcement. Figure 1-a shows that the cellulose fibers or sisal fibers were placed in the lower part of the samples because this positioning of the fibers is the best position for the material to become very resistant to bending according to [23].

2.5.2 characterization

three-point bending test and physical properties

Three-point bending tests are necessary to evaluate the mechanical characteristics and behavior of materials as well as their damage. For each reinforcing condition under test, three specimens were evaluated. The computer connected to the bender automatically calculated the reinforced plaster specimen's flexural strength using the formula in equation [24]:

$$\sigma_f = \frac{3FL}{2bh^2}$$

2

Where: F is the maximum load applied, L is the distance between the supports (100 mm), b is the width of the specimens (40 mm), h is their thickness (40 mm)

As indicated in Fig. 2, the sample was placed on two supports spaced 100 mm apart. The loading actuator delivered increasing stresses to the sample's middle until it broke. Finely an absorption test was done according to EN13279-2 standards to determine the absorption rate and the density before and after absorption.

3. Results

1. Strength Test

The results reported in the following paragraph are the averages of the five micro-tensile samples from each condition. Figure 3 shows that after the alkalization of sisal fibers, we obtain a better tensile strength compared to natural fibers, with different percentage increases for each of the treatment conditions. This is clearly illustrated in figure (10), and with a tensile strength equal to 490 MPa, the alkaline treatment with a 3% NaOH solution for 3 hours is the optimum treatment combination. (about 4 times compared to raw fibers) with a percentage of increase equal to 292% compared to untreated fibers. Also, the cellulose fibers extracted from sisal fiber have good tensile strength despite their diameter which becomes smaller than the natural fiber which reaches 530 MPa with a percentage of increase equal to 324% compared to untreated fibers

2. Chemical Composition

The chemical composition of natural and treated sisal fibers was determined according to the TAPPI protocol. Table 1 presents the results of the analysis. The extraction rate in water is relatively low compared to the majority of other cellulosic fibers (in cold water equal to 13% and in hot water equal to 10.9%) and in a 1% NaOH solution, the extraction rate is equal to 13.6%) as well as in ethanol/toluene the solution has an extraction capacity equal to 8.8% of the total mass of raw materials. However, the fibers treated with sodium hydroxide have an extraction rate in cold water that is relatively equal compared to natural fibers with a percentage equal to 12.3%, unlike extractions in hot water which are greatly reduced

compared to extractables in cold water with a percentage equal to 3.5%. in a 1% NaOH solution, the extraction rate is equal to 13.6% and 1.0%), so it can be observed that the alkaline treatment eliminated most of the extractions with 1% NaOH. as well as in ethanol/toluene the solution has an extraction capacity equal to 8.8% and 7.5% of the total mass of raw materials successively for natural fibers and treated fibers. Lignocellulosic fibers have two main components, one is lignin and the other is hollocellulose which divides into cellulose and hemicellulose. For sisal fibers, the lignin content is generally between 10% and 14% and for this Tunisian sisal fiber, the lignin content is equal to 13.2% with a reduction of 4.9% for treated sisal according to Mária et al. [25]. this reduction can be expressed by the extractive effect of sodium hydroxide on lignin. The holocellulose content of 68%, with an α-cellulose content of 58.6% and 9.4% hemicellulose, which was similar to the study of Mária et al [25], but for treated fibers and because of the effect of NaOH which eliminates many extracts such as lignin and extracts in hot water, it is observed that the cellulose rate increases by 10.8% and reaches 69.4%. Thus, the high content of α-cellulose is advantageous for cactus fibers whose objective is to apply them in industrial applications such as composite materials, paper manufacturing, etc.

Table 1
Chemical composition of natural and alkali treated sisal fibers and other lignocellulosic fibers

	Chemical composition							
	Ash	**C.W**	**H.W**	**1%NaOH**	**E.T**	**Lin**	**Hol**	**Cell**
Natural Sisal (other reserches) [25]	n.a	n.a	n.a	n.a	n.a	10–14,2	67–78	59–66
Astragalus armatus [26]	3	26.2	33	32.7	13	16.7	54	35
Retama raetam [27]	3.5	32	32.5	47	10	20.5	58.7	36
Plam [28]	1.2	n.a	n.a	n.a	n.a	35	51	43
Alfa [29]	5-7.2	n.a	n.a	n.a	n.a	18.7–24	59–69	39.5–47.6
Natural sisal (this work)	**1.20**	**13**	**10.9**	**13.6**	**8.8**	**13.2**	**68**	**58.6**
Alkali Treated sisal (this work)	**1.05**	**12.3**	**3.5**	**1.0**	**7.5**	**8.1**	**82.7**	**69.4**

Note : n.a: not available; C.W: extractables in cold water (%); H.W.: extractables in hot water (%); E.T: ethanol–toluene extracts (%); NaOH 1%: extractables in sodium hydroxide 1% (%); Hol: holocellulose (%); Lig: Klasson lignin (%); Cell: Cellulose (%)

3. Ftir

FT-IR spectroscopy was carried out to examine the changes in the chemical structure of treated and untreated sisal fibers, namely: untreated sisal fibers (N.T), sisal fibers treated with sodium hydroxide (NaOH. T), and the cellulose extracted from sisal fibers (Cellulose). The FTIR spectra of the three samples presented in Fig. 4 are comparable, with minor differences due to the influence of the alkaline treatment, and they show a classic lignocellulosic band. [30]

A broad band with a maximum of around 3300 cm-1 is presented in the first pick, which can be associated with the stretching vibration of hydroxyl (-OH) groups of carbohydrates and N-H groups of high hydrogen content glycoproteins, with a small increased absorbance intensity ratio for mercerized fibers and cellulose compared to raw fibers, which may be related to the fact that the a-cellulose content remains almost constant after mercerization [31–32]. The second peak is a sharp peak in the 2800–3000 range, exactly at 2930, representing the process of ion exchange between symmetric or asymmetric C-H protons and the symmetric stretching vibration of CH2[33]. Two absorbance bands at 1460 cm-1 and 1630 cm-1 are attributed to (asymmetric) CH3 strain in lignin (ionized COOH (COO − asymmetric stretching)), it is pointed out that the intensity ratios decrease considerably for the fibers treated with sodium hydroxide and cellulose (especially for cellulose), this can be mainly due to the loss of lignin during the alkaline treatment and the total loss of the latter during the extraction of the cellulose [32, 34].

4. Tga

Thermogravimetric analysis (TGA) is the most important test to determine the thermal stability of materials and their decomposition because organic fibers are subjected to thermal stress during their application, especially in composite materials. like most of the works which are interested in the study of cellulosic fibers, we make a comparison between the thermal stability of natural fibers, fibers treated with NaOH, and cellulose extracted from sisal.

The thermogravimetric analysis (TGA) curves are divided into four main parts, in the first part of the curves, there is a slight increase and some disturbance in the mass due to the instability of the mass at the start of the test. the second part between room temperature and 300°C for the untreated fibers and 260°C for the treated fibers and cellulose, the mass of all the samples makes a small loss which does not exceed 5% of its initial masses, this decrease is due to the loss of moisture and water in the fibers under high-temperature exposure. In the third part, the loss of mass continues but quickly and uniformly until reaching a temperature equal to 480°C for the untreated fibers with a loss equal to 27% and 360°C for the treated fibers and the cellulose with a mass loss successively equal to 35% and 32%. This is due to the breakdown of hemicellulose and cellulose in the fibers which are more susceptible to heat stress. Thus the fourth interval presents the degradation of lignin since it needs a high temperature to decompose. Thus the loss remains continuous but more slowly before having the stability of the mass at the temperature of 620°C with a loss of total mass equal to 38% for the untreated fibers, 45% for the fibers treated with NaOH, and 41% for cellulose.

The alkaline treatment of sisal fibers and the extraction of cellulose gives a material that is mechanically stronger and cleaner compared to raw fibers by eliminating the impurities responsible for the reduction of the mechanical properties of the fibers. Thus, as reported by Fig. 5, the mass loss of treated fibers was greater than untreated fibers, especially cellulose, so it could be said that fiber treatments improve the thermal stability of fibers when applied to composite materials.

5. Morphology Analysis(Sem)

Scanning Electron Microscopy (SEM) is a technique to observe the morphological details of the surface of materials, as in our case, natural and treated cellulose fibers, as shown in Fig. 6, the change in morphology of the surface between the fibers is very visible. The figure Fig. 6-A corresponds to the morphology of the surface of the natural sisal, and as it is clear, the surface is covered by a layer of amorphous materials mainly composed of hemicellulose, pectin, wax, and lignin, since its macromolecular composition is mainly composed of cellulose (58.6%), hemicellulose (9.4%) and lignin (13.2%). Other significant impurities may also be present on the surface of the fiber. The layer of impurities weakens the interfacial adhesion between the fiber matrix [35] and negatively influences the stiffness of the fiber [36].

By observing the sisal fibers treated with sodium hydroxide under the following conditions (concentration of 3% NaOH and duration of treatment 3 hours) (NaOH.T), it is noted in Fig. 6-B that the number of pores on the surface of the fibers has increased. The external details and notches become more visible. Also, the figure Fig. 6-C shows a big difference between the fibers treated with NaOH or the untreated fibers compared to the cellulose extract from the cellulose at the level of the overall shape of the structure which was completely changed, the fiber becomes cleaner and more rigorous whether the fiber is natural or treated. Therefore, cellulose fibers can be said to have a positive effect on fiber/matrix adhesion by increasing the bond between the fiber and the matrix [35].

6. Energy Dispersive X-ray Analysis (Edx)

Energy dispersive X-ray spectroscopy (EDX) is a very important technique for determining the material's elementary composition. This technique is based on the interaction of the sample and the source of X-ray excitation. This technique allows for displaying a specific set of peaks on its X-ray emission spectrum. Essentially, each element has a unique atomic structure. In this study, an EDX analysis was carried out to observe and study the elementary composition of Tunisian sisal fibers in several cases, firstly in their natural form (N.T) then when they are alkali-treated by sodium hydroxide (NaOH.T), and finely, the EDX analysis was carried out to the cellulose fibers extracted from the sisal plant. The results are shown in Fig. 7-A, which shows that the sisal fibers are mostly composed of the following main components: Oxygen (O), and Carbon (C) are the most dominant elements in all EDX spectra because as it is known, they are the main elements that make up the lignocellulose fiber structures [37]. Also, there are other elements with fewer quantities like nitrogen, silicon, chlorine, magnesium, and calcium. As shown in Table 2, the chemical treatment removed certain elements or reduced their percentages such as silicon, sulfur, magnesium, potassium, and iron. Thus, the chemical composting of the fibers treated by alkaline, which has become mainly composed of carbon, oxygen, and sodium, and traces of very small amounts of calcium and silicon Fig. 7–B, but for cellulose, there is a total elimination of impurities and wax as shown in the figure Fig. 7-C. Therefore, it can be concluded that the chemical treatment of natural sisal fibers has greatly improved the quality of the surface, it makes it clean of all impurities, pores, and cavities become more visible as is clear in the SEM image

Table 2
Elementary composition of Sisal fiber, NaOH treated Sisal fiber, and Cellulose

	Sisal fiber		*NaOH treated Sisal fiber*		*Cellulose extracted from Sisal fiber*	
Element	*Weight %*	*Atom %*	*Weight %*	*Atom %*	*Weight %*	*Atom %*
C	35.14	41.86	32.05	38.35	36.79	43.05
N	13.92	14.21	12.59	12.92	13.32	13.37
O	47.09	42.10	53.38	47.95	49.22	43.25
Na	0.63	0.39	N.A	N.A	0.10	0.06
Mg	0.50	0.29	0.21	0.13	N.A	N.A
Al	0.48	0.25	N.A	N.A	0.38	0.20
P	N.A	N.A	0.10	0.04	N.A	N.A
Si	0.56	0.28	N.A	N.A	0.07	0.04
S	0.09	0.04	0.07	0.03	N.A	N.A
Cl	0.06	0.03	N.A	N.A	N.A	N.A
K	0.12	0.04	0.89	0.33	N.A	N.A
Ca	1.34	0.48	0.71	0.25	0.11	0.04
Fe	0.07	0.02	N.A	N.A	N.A	N.A
Total	100.00	100.00	100.00	100.00	100.00	100.00

7. Spectroscopic Analysis (Drx)

According to the approach of Segal et al.[38], the crystallinity of the untreated or treated cactus fibers may be determined from the crystallinity index (CI) using the empirical equation shown below:

$$CI\,(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

3

I_{am} is the intensity related to the diffraction of amorphous matter only, where the intensity is low, while I_{002} is the highest diffraction intensity of the peak in reference to the crystalline zone that represents both crystalline and amorphous materials simultaneously.

Figure 8, shows the XRD spectra of untreated sisal fibers (N.T), NaOH-treated cactus fibers(NaOH.T, and cellulose extracted from sisal fibers(Cellulose). Picks at 15.6° and other picks at 22.5° that are provided by planes (101) and (002) demonstrate that all XRD curves may have a Type I cellulose-specific curve [39]. Moreover, look for a tiny pick that might change as a result of the treatment. But, the pick diffraction intensity, which varies from one type of treatment to another, is what makes the curves different from one another.

The results of the calculations of the crystallinity index using the segal et al method presented in (Table 3) show that the CrI for untreated cactus fibers, treated with NaOH, and the cellulose are 60.0%, 60.1%, and 67.1%, respectively. According to these results, the treatment of sisal fibers by sodium hydroxide has no influence or very small influence on the crystallinity of the fibers that was affected in a low way as CrI increased by 0.1%. Even if a significant amount of hemicellulose and wax has been removed from sisal fibers by this treatment, the crystalline region of lignocellulose cannot be deeply increased with low-concentration alkali within a short treatment time. However, the cellulose extracted from natural sisal fibers has a crystallinity equal to 67.1% with an increment of 7.1% compared to the untreated fibers[40].

Table 3
Crystallinity index of natural, treated sisal fibers, and Cellulose

samples	I_{am} (15.5%)	I_{cr}(22.5%)	CI (%)
N.T	496	1240	60.0%
NaOH.T	487	1246	60.1%
Cellulose	541	1525	64.5%

8. Mechanical Proprieties Of Composite

8.1 *Mechanical behaviour of plaster composite with alkali-treated sisal fibers and cellulose reinforcement*

Figure 9, shows the behavior of the plaster-based composite, Fig. 9-a for the composite reinforced with treated sisal, and Fig. 9-b for the composite reinforced with cellulose. the two curves can be divided into three parts and two characteristic points: the point of maximum applied force (σmax), and the point at which the first macroscopic damage of the composite can be observed (σ*). The first part presents the quasi-linear behaviour of the composite, it corresponds to the behaviour of the gypsum only (matrix) [41]. The second part, which is between the characteristic point σ* and the characteristic point σ_{max}, which characterizes a drop in the load, corresponds to the first macroscopic damage of the composite, then the load is taken up by the reinforcement (sisal or cellulose) until the maximum strength (σ_{max}) is reached. In the third part, a sudden drop in load is observed, which corresponds to a rupture of the reinforcement-matrix interfaces or the complete damage of the fibers [42].

8.2. Fibers mass fraction effect on the flexural strength of plaster-based composites using treated Fibers and cellulose fibers

The results of the three-point bending test for the samples of plaster reinforced with the two types of fibers (the treated fibers of sisal and the fibers of cellulose) with different percentages (00.00%, 0.50%, 0.75%, 1.00% 1.25%, 1.50%, and 1.75%) are presented in the Fig. 10. The study of the properties of tensile strength in bending with 2 different methods of reinforcement and 7 different percentages of reinforcement gives that the use of cellulose fibers extracted from the natural fibers of sisal is always more favourable than the utilization of the fibers. treated with sodium hydroxide, especially when increasing the percentage of reinforcing fibers (1.25% and 1.50%). A gradual increase in the resistance to bending linked to the addition of sisal or cellulose fibers, which goes from a value of 0.82 MPa for the non-reinforced plaster samples to a value of 1.11 MPa for the plaster reinforced by the fibers treated with alkaline with an increase of 35.3% at a mass fraction of the fibers equal to 1.25% before observing the decrease in the resistance to bending. also for the samples in plaster reinforced by cellulose fibers, there is a progressive increase in the resistance to bending linked to the increase in the percentage of fibers until reaching the mass fraction of cellulose 1.50% with a maximum resistance equal to 1.22 MPa before observing the decrease in flexural strength with a percentage increase compared to plaster without reinforcement equal to 48.70%.

These results confirm the results of microscopic observation (SEM) about the surface condition of cellulose fibers which becomes rougher with larger cavities than the surface of the fibers after treatment with sodium hydroxide [43]. So this increase of the adhesion between the matrix and the fibers guides to avoiding the slippage phenomenon as shown in Fig. 11-a, and Fig. 11-b. The increase in adhesion in the case of cellulose helps the composite material resist more bending before the break. The measurement of the diameters of sisal fibers and cellulose fibers gives us that the cellulose fibers have a smaller diameter and a tensile strength almost the same as the sisal fibers, it is remarkable that a quantity of cellulose has more fibers than the same quantity of fibers is sisal, therefore the resistance of a well-determined quantity of cellulose will be better than the same quantity of sisal fibers and this will positively influence the properties of the composite materials.

9. Absorption Rate, And Density

Table 4 shows the absorption rate (%) of raw plaster and plaster-based composite reinforced with alkali-treated sisal fibers and cellulose fibers with different volume fractions. Because in the field of building, good physical properties are always required. From Table 4 and Fig. 12, it can be seen that the rate of absorption has increased dramatically as a result of the increase in the percentage of fibers. Given this large absorption capacity, the properties of the material are greatly improved.

The bulk density was calculated according to ASTM (D1895). The procedure was as follows: The weight of the sample was measured by Using an electrical balance. Also as the absorption rate in the field of building construction, the weight of materials is very important because the weight reduction gives to reduce the charges for mechanical properties such as the capacity to support the loads. In addition, since this composite material is lighter than dry plaster, it imposes lower loads on the building structure, as shown in Fig. 13.

Table 4
physical proprieties of plaster-based composite material

Fibers Mass fraction (MF)	MF = 0.00%	MF = 0.5%		MF = 0.75%		MF = 1.0%		MF = 1.25%		MF = 1.5%	
		NaOH.T	Cell	NaOH.T	Cell	NaOH.T	Cell	NaOH.T	Cell	NaOH.T	Cell
Front mass absorption test (g)	354	337	342	312	316	288	287	280	284	266	273
mass after absorption(g)	472	456	462	434	440	420	418	416	419	412	415
Absorption rate (%)	33	35.3	35.8	39,1	39.2	45.8	45.6	48.5	47.5	55.0	52.0
Density (g/cm^3)	1.38	1.31	1.31	1.21	1.23	1.12	1.12	1.09	1.10	1.03	1.06

Conclusion

The use of sisal cellulose fibers for the production of composite materials is achieved by alkaline treatment with NaOH under different treatment conditions (different concentrations and immersion times) and with sisal cellulose. The following conclusions can be drawn:

- Alaklin treatment with 3% alkaline solution for 3 hours is the best treatment condition with tensile strength reaching 490 MPa and 530 MPa for cellulose compared to 125 MPa for natural fiber.

- The morphology of the fiber surface was radically modified after the alkaline treatment, becoming more rigorous, clean, and visible. The details of the cellulose fibers were more visible and porous, and the cavities and notches were cleaner from wax and impurities.

- The changes in the fibers were confirmed by the results of EDX, FTIR XRD, and chemical composition analysis, which showed that the treatment removed most of the lignin, hemicellulose, and pectin, as well as the impurities.

- 3-point flexural tests on cellulose/fiber reinforced plaster-based composites show that the alkali-treated fiber reinforcement improves the flexural strength by 35.3% while the cellulose reinforcement improves the strength by 48.70%. This can be explained by the strong adhesion between the cellulose and the matrix.

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Figures

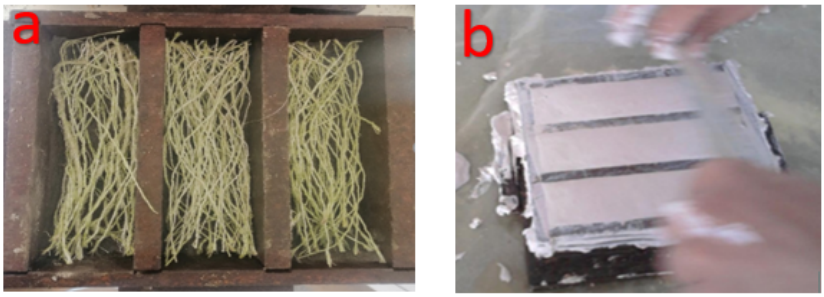

Figure 1

plaster-based Composite making: (a) reinforcement poisoning, (b) sample preparation

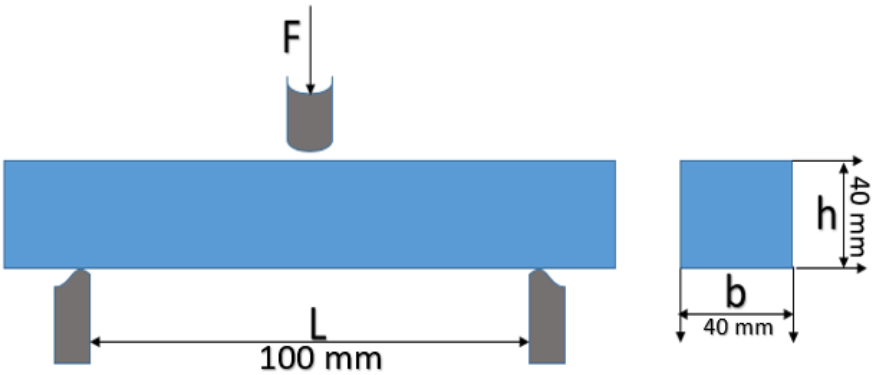

Figure 2

Three-point bending test for plaster-based Composite material

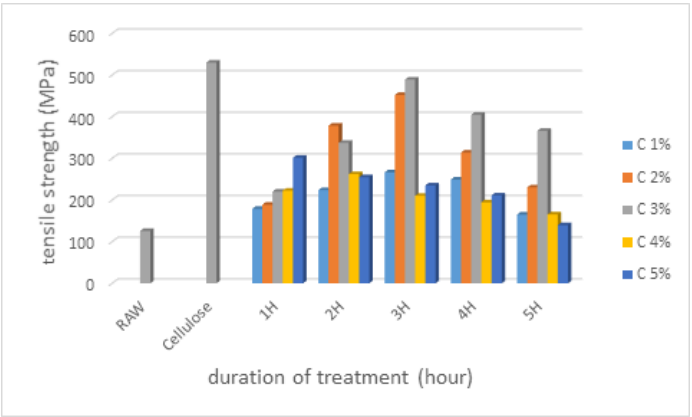

Figure 3

tensile strength of natural sisal fibers, treated sisal fibers, and cellulose

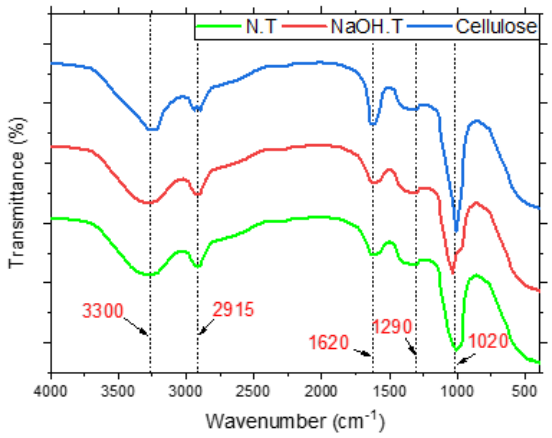

Figure 4

FTIR spectra of natural sisal fibers; NaOH treated sisal fibers and cellulose

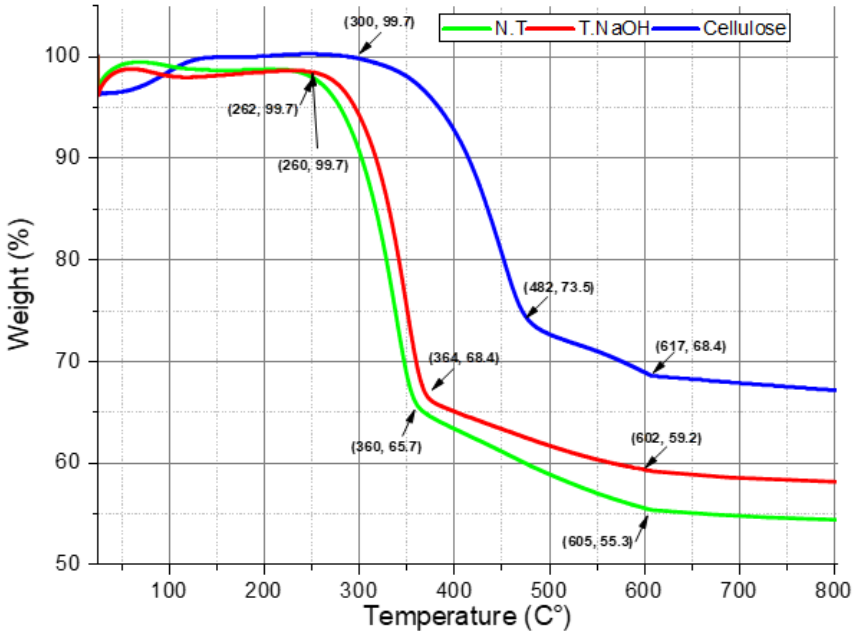

Figure 5

Thermogravimetric analysis (TGA) for natural sisal fibers; NaOH treated sisal fibers, and cellulose

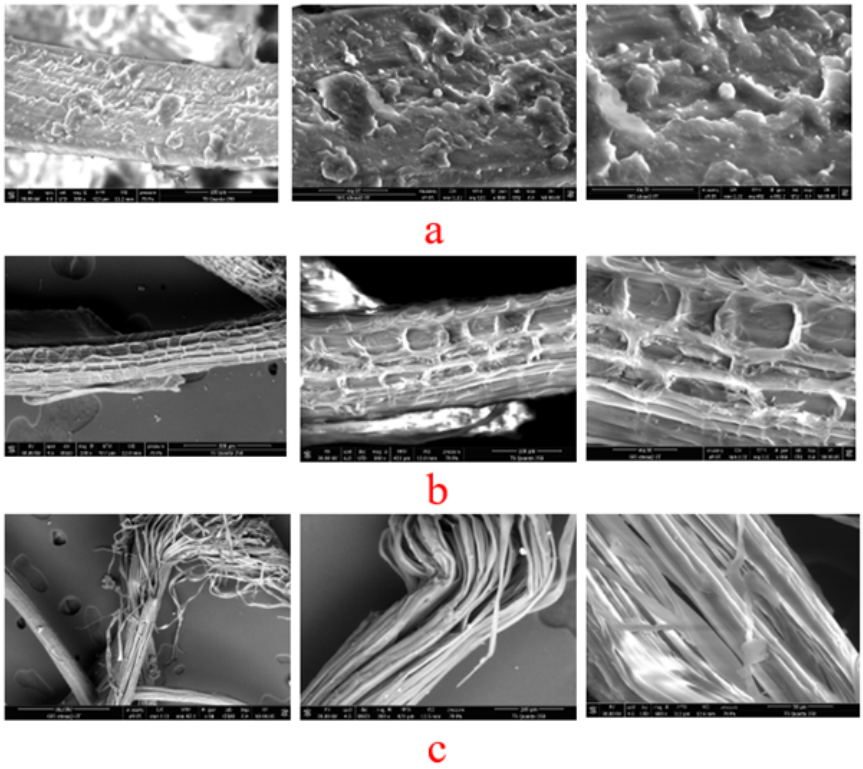

Figure 6

SEM images of (A) natural sisal fibers; (B) NaOH-treated sisal fibers, and(C) cellulose

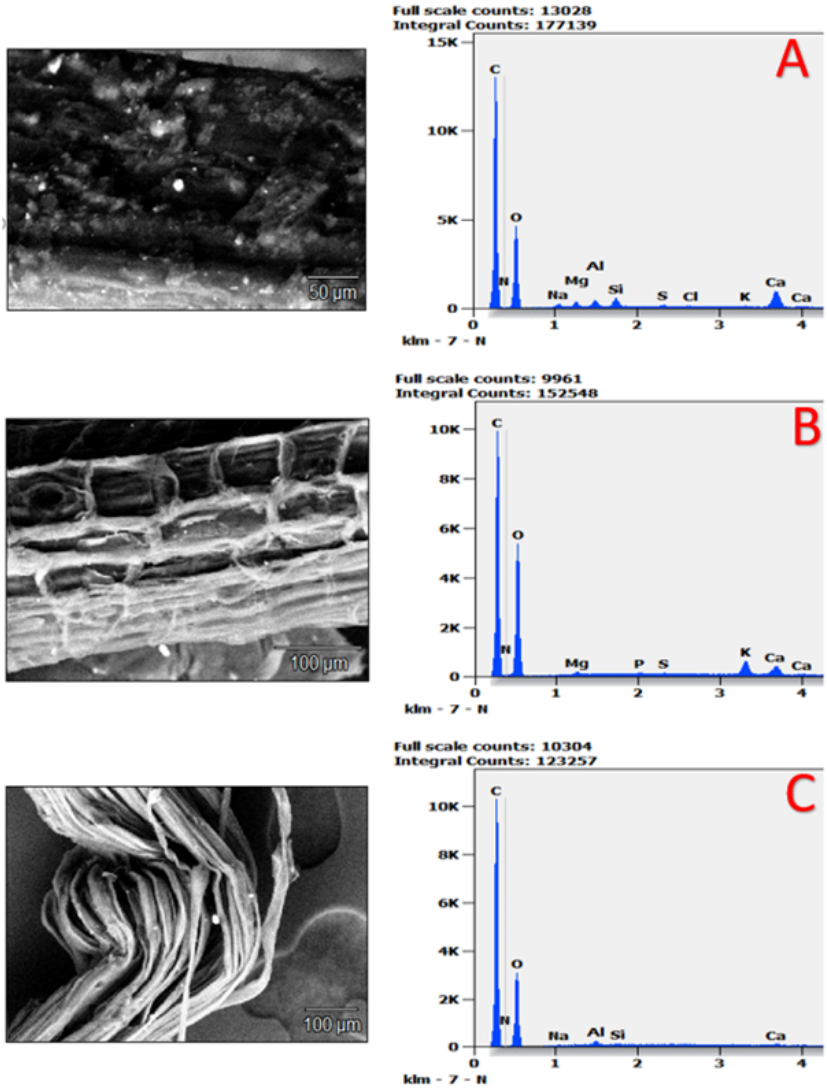

Figure 7

EDX spectra of (A) natural sisal fibers; (B) NaOH-treated sisal fibers, and(C) cellulose

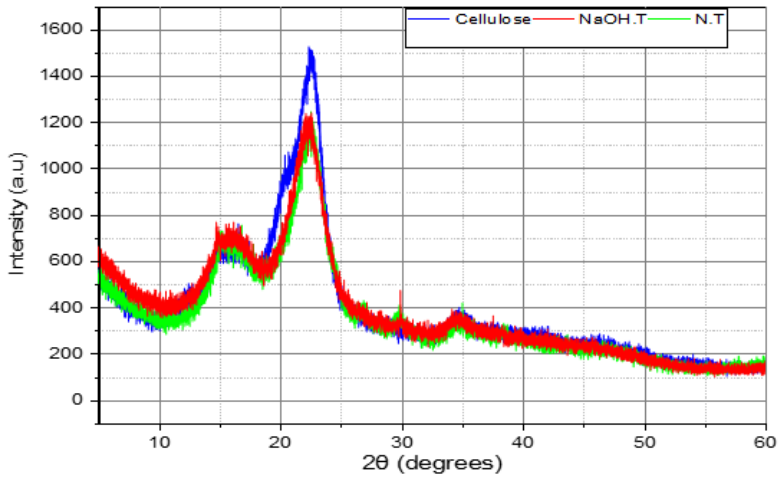

Figure 8

XRD spectra of natural sisal fibers; NaOH treated sisal fibers and cellulose

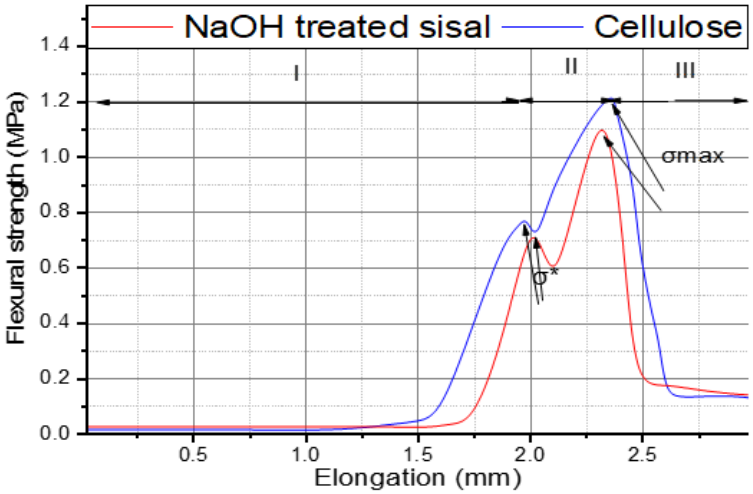

Figure 9

Mechanical behaviour of plaster composite with alkali-treated sisal fibers and cellulose reinforcement

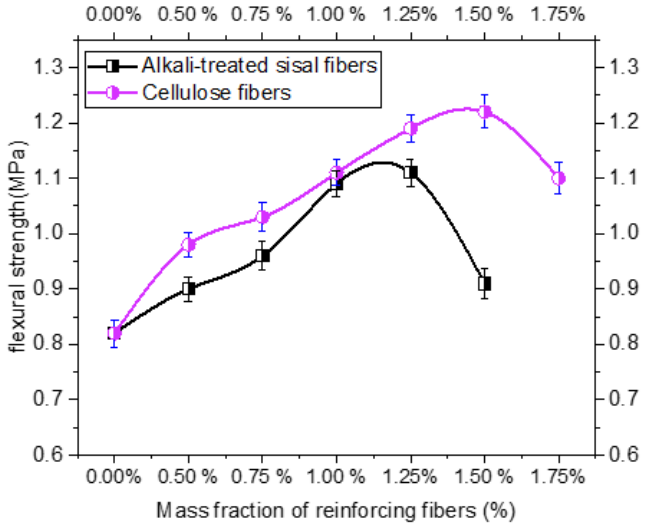

Figure 10

mass fraction effect on the flexural strength of plaster composite with alkali-treated sisal fibers and cellulose reinforcement

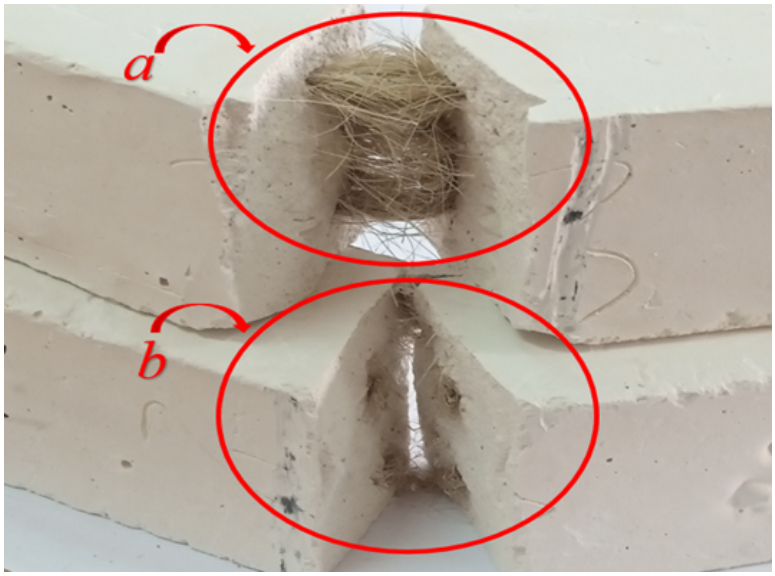

Figure 11

Cracking behaviour of plaster composite with alkali-treated sisal fibers(a) and with Cellulose reinforcement(b)

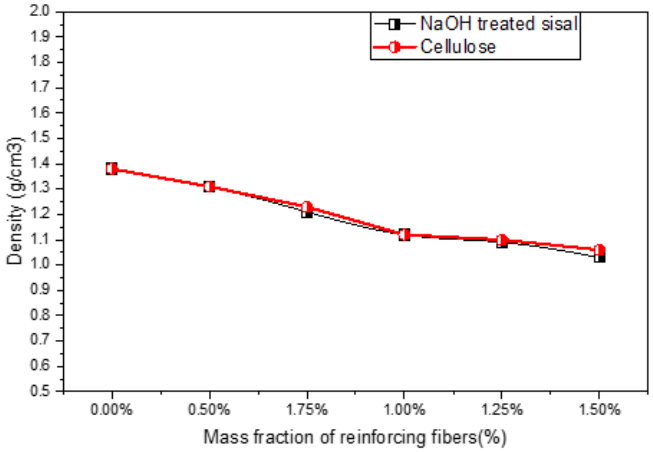

Figure 12

Density of comosite

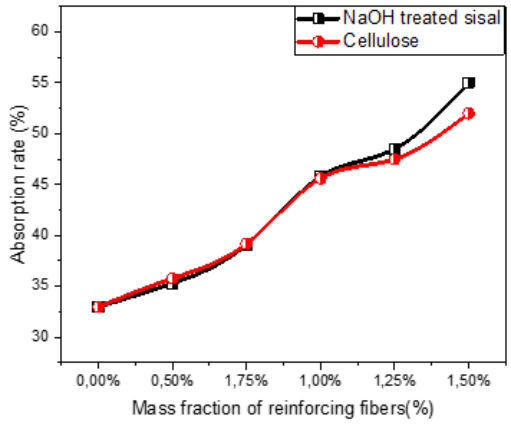

Figure 13

Absorption rate of composite