

## MECHANICAL PROPERTIES OF ALFA, SISAL, AND HYBRID ALFA/SISAL FIBER SATIN CLOTH REINFORCED EPOXY

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*The mechanical behavior of composites, made of an epoxy resin matrix reinforced by 30 and 40% of a satin cloth from long Alfa, sisal and hybrid Alfa/sisal fibers was studied. The fibers are obtained by extraction with elimination of binders such as pectins and lignin. For each type of fibers, appropriate and optimal chemical and thermal treatments were conducted within NaOH solution, to enhance both the fiber surface quality and the interfacial bonding between fibers and matrix. Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and chemical decomposition of treated and untreated fibers lead to prove the treatment efficiency. The thermogravimetric (TGA) and differential thermogravimetric (DTG) analyses showed better thermal stability. Differential scanning calorimetry (DSC) made it possible to quantify the enthalpy changes which showed an increase in the amount of heat as a function of the increase in weight fraction of natural fibers. The endothermic reaction of the composites studied containing 30 wt% fiber reinforcement was less than that containing 40 wt% fiber reinforcement. The composite materials were produced by vacuum assisted resin transfer molding (VARTM) method due to hydrophilic nature of the fibers. The results of static tests were compared to those of pure epoxy resin. It showed a significant increase for 40 wt% woven Alfa/epoxy of about 333, 113, and 81% in tension, 3-points bending and compression tests respectively. SEM morphology analysis revealed good interfacial adhesion between the treated fibers and the matrix.*

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## 1. Introduction

Numerous industrial applications and several studies have been developed using composites with natural fibers and biopolymer matrices. Antich et al. [1] studied the mechanical behavior of high impact polystyrene (HIPS) reinforced by short sisal fibers. The effect of chemical treatment of short sisal fiber-reinforced polyethylene composites has improved the tensile properties, particularly the Young's modulus up to 3.09 GPa [2]. The biocomposite materials based on Alfa cellulose fibers (esparto grass plant) as reinforcing elements and starch-based biopolymer matrix were molded by injection process [3]. The Young's modulus obtained was 4.16 MPa. By a molding technique, Helaili et al. fabricated a composite using epoxy resin reinforced by randomly distributed short Alfa fibers [4]. Its mechanical properties were validated by numerical homogenization and experiments. At 17 wt% of Alfa fibers reinforcement, Young's modulus was 6.26 GPa. Kumar et al. concluded that the selection of weave architecture is essential for material optimization and in all the cases twill-woven composites showed up to 10% higher mechanical properties than plain-woven composites [5]. Murugan et al. studied a non-woven reinforcement composite made of banana fibers. By modifying the fiber lengths and the volume fractions of reinforcement, they obtain the Young's modulus of 3.4 GPa [6]. Boominathan et al. studied the alkali-treated Acacia concinna fiber/polyester matrix composite. The composites reinforced by 30 wt% fibers demonstrated the best results: tensile modulus of 1.99 GPa, flexural strength of 137.31 MPa, flexural modulus of 2.86 GPa, impact property of 18.61 J/cm<sup>2</sup>, hardness property of 95 HRRW, and water absorption characteristics of 4.9% [7]. Composites made of Flax fibers with thermoplastic, thermoset, and biodegradable matrices were reviewed by Yan et al. [8]. It was found that Young's modulus of Flax fiber and its composites equal to 2.4 GPa. The composite based on a biodegradable polymer (MateBi) and fibers extracted from the Alfa plant was treated by Borchani et al. [9]. The Young's modulus was 505 MPa. Mechakra et al. [10] optimized the Alfa fibers treatment during 24 h with NaOH prior to mixing with resin using an extruder to produce filaments. The Young's modulus was 3.6 GPa. Nour et al. [11] studied a composite made of a polypropylene matrix reinforced by short Alfa fibers untreated and chemically treated. The fibers homogenization has been performed using a modified Mori-Tanaka model and by the inverse method which led to the determination of mechanical properties. A totally cellulosic-ingot lignocellulosic material prepared from Alfa fibers reinforcement and two types of matrices were developed by Labidi et al. [12]. The Young's modulus values were 3.8 and 4.2 GPa, respectively.

Motivated by the above-described results, the present study treated the thermochemical and mechanical behavior of a new composite made of an epoxy resin matrix reinforced by a satin cloth from natural fibers, which was not studied until now.

## 2. Materials and Method

The epoxy resin used is of the DGEBA type marketed by LORN CHIMICAL and manufactured by IMEXFA SA (Brussels, Belgium). It is unfilled, undiluted and is intended for the formulation of solvent-free epoxy systems, for molding composites, and etc. Its technical properties are given by the manufacturer: epoxy equivalent (182-190), viscosity at 25°C (800-1200 MPa), density at 20°C (1.17 g/cm<sup>3</sup>) and flash point (> 150°C). It is a pre-dosed kit of two components: resin (65 wt%) and hardener (35 wt%). The Alfa and Sisal plants employed in this study originate from Algeria (El Bayadh and Boumerdes) (Figs. 1a and 1b).

### 2.1. Alfa fibers extraction

It consists of two steps, first immersing fibers in a salty water to eliminate the wax, the sand, and the dust from the stem surfaces [10]. This process renders the stems more amenable to the next treatments. A 35 g·L<sup>-1</sup> of sodium chloride concentration is used in distilled water. The duration of the fiber immersions is 24 h at 60°C in an oven (MEMERT UN110) in order to allow the waxes of the outer wall to dissolve in the salty water. Second, brushing is performed in the longitudinal direction of the stems to obtain fibers of regular diameters.



Fig. 1. Raw stem; Alfa (a) and agave Sisalana plant (b).



Fig. 2. Untreated and treated samples fibers: untreated (a) and treated (b) Alfa; (c) untreated (c) and treated (d) Sisal.

## 2.2. Sisal fibers extraction

Agave plant leaves were crushed manually to soften them, and then scraped until the appearance of long fibers of white or greenish color. These fibers are then carefully extracted and cleaned in distilled water during 24 h [13].

## 2.3. Alfa fibers treatment

Alfa fibers were treated with an alkaline sodium hydroxide. Prior to starting the process, the fibers were rinsed and put into a  $1.6 \text{ mol}\cdot\text{L}^{-1}$  NaOH solution during 24 h at room temperature [10]. Then, they are completely neutralized into a 1.6 wt% concentrated acetic acid solution and dried at ambient air temperature for 24 h. After that, the fibers are maintained at  $60^\circ\text{C}$  for 12 h in an oven, then put into desiccators containing silica gel during 24 h to avoid the humidity impact.

## 2.4. Sisal fibers treatment

The extracted sisal fibers were immersed into a 10 wt% NaOH solution for 5 h and kept at  $65^\circ\text{C}$  during 6 h in an oven, then rinsed with distilled water and neutralized in a 2 wt% acetic acid solution to remove the residual NaOH solution. A final rinse was necessary prior to drying them at room temperature for 4 to 5 days. Thus, the fibers obtained by different treatments were used for the elaboration of the specimens [14].

TABLE 1. Chemical Composition of Alfa and Sisal Fibers

Material	Cellulose, wt%	Hemicellulose, wt%	Lignin, wt%
Untreated	43,08	21.35	28,67
NaOH treated	64.9	6.15	25.05
Untreated	63.7	20.48	10.13
NaOH treated	75.1	5.51	8.29

## 2.5. Determination of the different constituents of fibers

*2.5.1. Fiber sampling.* Sisal and Alfa fibers were first cleaned to remove dust and other particles and cut into pieces of 150-mm length to form the fibers samples. These latter were freeze-dried for 72 h and milled using a microfine grinding mill equipped with a 1-mm sieve. The grounded samples were freeze-dried once more for 24 h to ensure effective drying. The samples (Fig. 2a-d) were then stored into airtight plastic bags in a freezer set at  $-18^{\circ}\text{C}$  [15]. *2.5.2. Extractives.* About 10 g of each raw dried fiber samples were extracted. The milled samples were accurately weighed and extracted, using a Soxhlet apparatus. The extraction was first performed with 190 mL acetone and 10 mL water for 8 h, then with 200 mL n-hexane for 24 h at  $40^{\circ}\text{C}$ . The solvent extracts were evaporated to dryness at  $40^{\circ}\text{C}$  to determine the resin ratios [15].

*2.5.3. Holocellulose.* 3 g of each fiber sample was immersed into 96 mL of distilled water with 1.2 g of sodium chlorite ( $\text{NaClO}_2$ ) and 0.6 mL of acetic acid. The mixture obtained was placed in a water bath at  $75^{\circ}\text{C}$  for 1 h and the process was repeated until a whitish appearance was obtained. Then, the precipitates were washed with distilled water and dried in an oven at  $100^{\circ}\text{C}$  for 12 h.

*2.5.4. Cellulose.* The cellulose was determined from holocellulose by basic hydrolysis of hemicelluloses. 25 mL of 17.5 wt% NaOH was added to 2 g of holocellulose and maintained for 50 min. The samples were filtered and 15 mL of 10 wt% acetic acid were added. The samples were filtered off, washed from acid, dried, and weighed. To obtain cellulose, each sample was put into a large test tube in an oven at  $103^{\circ}\text{C}$  for 12 h.

*2.5.5. Lignin.* The lignin was determined using extraction residues from the complete hydrolysis of the polysaccharides. The polysaccharides in the fiber samples were hydrolyzed and solubilized by 72 wt% sulfuric acid for 2 h. The samples were diluted to obtain a 3 wt% sulfuric acid solution, then filtered and dried in an oven for 12 h.

## 3. Results and Discussion

During chemical treatments, constituents such as hemicelluloses were hydrolyzed by the action of alkaline solutions. The results obtained showed that untreated Alfa and Sisal fibers had the amounts of lignin (28.67 and 10.13 wt%) and hemicelluloses (21.35 and 20.48 wt%), respectively, greater than those in the treated fibers (Table 1). At that, treated Alfa and Sisal fibers had the amounts of cellulose (64.9 and 75.1 wt%), respectively, greater than those in untreated fibers (see Table 1).

## 4. ATR-FTIR Analysis

Fourier transform infrared coupled with attenuated total reflectance (ATR-FTIR) is a very powerful technique for qualitative analysis to study the changes in the chemical composition during different stages of treatment. By means of FTIR spectroscopy and chemical decomposition, the efficacy of alkaline treatment on each type of fiber was demonstrated. The chemical analysis of the fibers revealed a reduction in the levels of hemicellulose, lignin, whereas amorphous cellulose is increased, leading to an increase in crystalline cellulose content. The ATR-FTIR measurement was obtained using the Thermoscientific Nicolet IS 10 instrument equipped with ATR Thermoscientific Smart ITR module at room temperature driven by computer

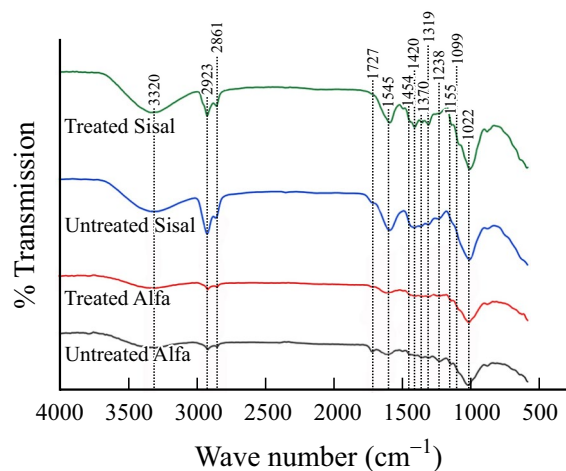


Fig. 3. FTIR spectra of untreated and treated Alfa, untreated and treated Sisal.

software, with spectral resolution of  $2\text{ cm}^{-1}$  and over wave number range of  $4000\text{--}400\text{ cm}^{-1}$  (wave lengths of  $2.5\text{ to }25\text{ }\mu\text{m}$ ). The fiber samples were cut and grinded into a fine powder and then carefully placed in the analysis chamber.

#### 4.1. Fourier transforms infrared analysis result

A comparison between the FTIR spectra (Fig. 3) of the treated and untreated Alfa and Sisal fibers shows a large absorption band, observed around  $3320\text{ cm}^{-1}$ , attributed to the ( $\text{--OH}$ ) group. After treatment, the band appeared weaker in intensity as a result of the arrangement of more hydroxyl groups, induced by the crack of bonds created from lignin and hemicelluloses with cellulose [15]. Both peaks observed at  $2923$  and  $2861\text{ cm}^{-1}$  are associated to ( $\text{CH}_2$  and  $\text{CH}$ ) groups of cellulose and hemicelluloses. Their decrease is due to the removal of hemicelluloses. The peak at wave number  $1727\text{ cm}^{-1}$ , which corresponds to the carbonyl groups ( $\text{C=O}$ ), can be attributed to the vibrations of the acetyl group of hemicellulose or to the carboxylic group (of the ferulicring and p-coumaric acid of lignin and/or hemicelluloses). Such a peak appears only in the case of untreated fibers [16] while the band at  $1545\text{ cm}^{-1}$  indicates the presence of ( $\text{C=C}$ ) groups of cellulose. The  $1454\text{ cm}^{-1}$  peak reflects the stretching vibrations of  $\text{CH}_2$  bonds which occur for treated sisal fibers because of the formation of cellulose while that around  $1420\text{ cm}^{-1}$  corresponds to the ( $\text{CH}_2$ ) groups of cellulose. Alcohol group of cellulose ( $\text{OH}$  deformation) are characterized by peaks located at  $1389\text{ cm}^{-1}$ . Peaks at  $1370\text{ cm}^{-1}$  contributed to the  $\text{C-H}$  asymmetric deformation, and that located at  $1319\text{ cm}^{-1}$  is assigned to the  $\text{CH}_2$  wagging of lignin. The intensity, at this peak, decreases after treatment for both types of fibers; indicating that the lignin component in fibers is removed by alkalization. The  $1238\text{ cm}^{-1}$  peak, attributed to the stretching frequency of the  $\text{COO}$  groups, indicates the presence of hemicelluloses and appears only for untreated fibers and not for alkali treated. This is due to the removal of the hemicelluloses by chemical treatment (sodium hydroxide reacts with hemicelluloses constituents which are removed from the natural fibers). Besides, the peaks at  $1155$  and  $1099\text{ cm}^{-1}$  are a characteristic of the stretching frequency of the  $\text{C--O--C}$  groups which indicates the presence of the cellulose for treated fibers. The occurrence of the peak at  $1022\text{ cm}^{-1}$  is attributed to the stretching vibrations of  $\text{C--O}$  bonds of cellulose [15], while the  $896$  and  $609\text{ cm}^{-1}$  peaks are associated to ( $\text{C--O--C}$ ) groups related, respectively, to the  $\beta$ -glycosidic linkages between glucose in the cellulose and the deformation vibration of the  $\text{C--OH}$  bond.

It is observed from Fig. 3 that the spectra of fibers have the same appearance, and all samples show closely similar spectra despite the decrease or disappearance of the intensity of the bands, characterizing the non-cellular constituents (lignin, hemicelluloses and wax), with the alteration of the treatment concentration; making fibers cleaner and increasing the interfacial adhesion between the fiber and the matrix. On the other hand, it is observed that some peaks appear, and others increase in the treated fibers; indicating the presence of cellulose. This component ensures a stiffness for reinforcing composite material in

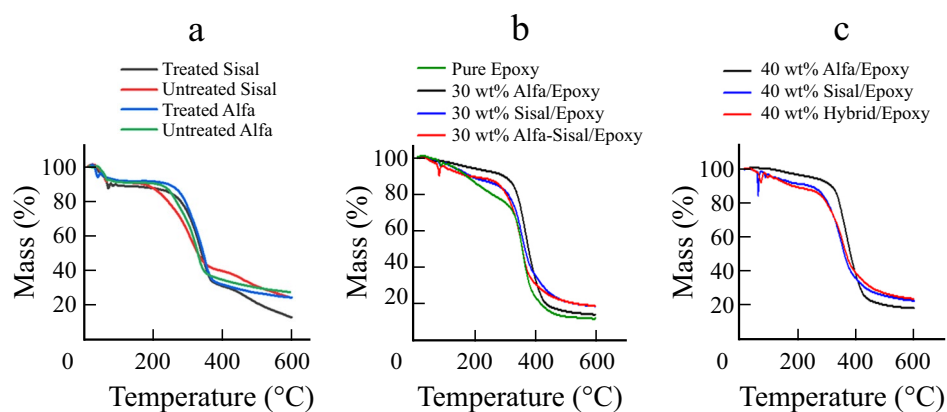


Fig. 4. TGA curves: fibers (a), 30 wt% fibers reinforcement (b) and 40 wt% fibers/epoxy reinforcement/epoxy (c).

agreement with previous findings. Consequently, the present FTIR results are in good accord with the literature; suggesting that the alkali treatments studied in this work was successful.

## 5. Thermal Analyses

The thermogravimetric analysis (TGA) is an essential analysis to determine the thermal properties of fibers throughout their life cycle and for evaluating their thermal stability to avoid the degradation of their physical-mechanical properties. The analysis was performed using the NETZSCH STA 409PC/PG instrument in a nitrogen atmosphere at a heating rate of 10°C/min. The spectrum was recorded in a temperature range of 20 to 600°C. The samples analyzed weighed between 3 and 6 mg.

### 5.1. TGA results

From TGA and DTG results, the analysis revealed that the chemical treatment gave good thermal stability to the fibers. The thermal stability temperatures of untreated and treated Alfa fibers are 192.59 and 216.12°C, respectively, and untreated and treated Sisal fibers are 156.08 and 218.11°C, respectively. These values fall in the order of thermoplastic polymer manufacturing cross-linking temperature. It can be concluded that these fibers are suitable as good reinforcing elements for application in polymer composites at high processing temperatures. At that, the thermal degradation temperatures of untreated and treated Alfa fibers are 399.33 and 451.12°C, respectively, and untreated and treated Sisal fibers are 498.58 and 495.61°C, respectively. The natural reinforcing fibers allow better thermal stability behavior of the composite materials. The results showed that increasing the weight fraction of the fibers increases the composite thermal stability.

In the graphical representation of the TGA results (Fig. 4a), the first phase represents the evaporation of water which is important for the treated fibers. This indicates that the treated fibers contain less moisture (allowing better adhesion of the fibers with the hydrophobic matrix). In the second phase, the mass loss is significant for the treated fiber; this phase corresponds to the degradation of the cellulose (the treated fibers contain more cellulose). In the third phase, the mass loss is greater for untreated fibers; this phase corresponds to the degradation of lignin. The loss of residual mass is greater for untreated fibers; it contains more residual deposit and impurity. TGA curves of the various samples of treated and untreated Alfa and Sisal fibers are presented in Fig. 4b. The degradation curves are similar due to their identical chemical compositions.

Different stages of thermal degradation are observed. The first mass loss was observed below 100°C. It is evaluated at 9.73 and 9.09 wt% for the untreated and treated Alfa fibers, respectively, and 9.79 and 13.25 wt% for untreated and treated Sisal fibers, respectively. This loss of mass represents the evaporation of water available in the fibers [17]. At this stage, the fibers

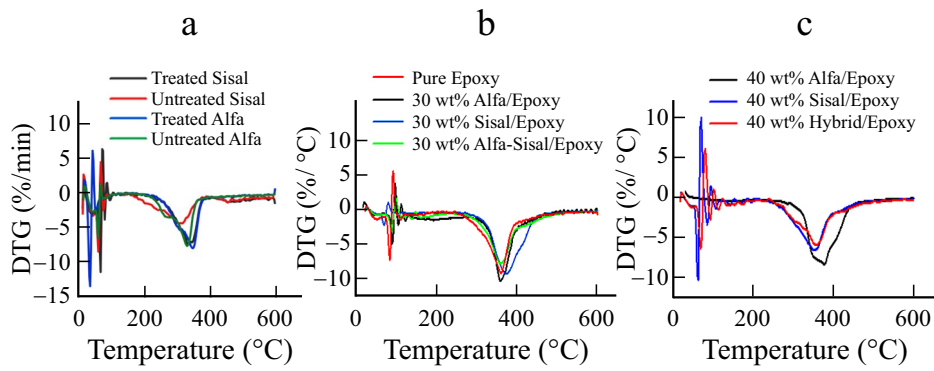


Fig. 5. DTG curves: fibers (a), 30 (b) and 40 wt% fibers/epoxy (c).

TABLE 2. Initial ( $T_d^i$ ) and Final ( $T_d^f$ ) Temperatures of Degradation, Maximum Temperature of Stability ( $T_s^{\max}$ ), Maximum Temperature of Decomposition ( $T_d^{\max}$ ), and Mass loss ( $\Delta m$ ) of Different Fibers and Composites from TGA and DTGA analyses

Specimen	$T_d^i$ , °C	$\Delta m$ , %	$T_s^{\max}$ , °C	$\Delta m$ , %	$T_d^{\max}$ , °C	$\Delta m$ , %	$T_d^f$ , °C	$\Delta m$ , %	Residue
Fibers									
untreated Alfa	60.93	93.89	192.59	90.27	330.93	51.95	399.33	34.87	27.56
treated Alfa	53.62	95.22	216.12	90.91	348.62	46.59	451.12	29.05	24.48
untreated Sisal	61.08	93.11	156.08	90.21	311.08	75.23	498.58	30.86	24.35
treated Sisal	68.11	89.39	218.11	86.75	333.11	54.77	495.61	21.56	13.06
Pure Epoxy	81.78	98.17	292.25	76.26	358.02	48.93	496.68	12.67	11.44
Epoxy composites reinforced with Alfa, Sisal, or Hybrid fibers									
30 wt% Alfa	87.84	97.95	272.00	91.26	357.84	53.11	478.23	16.34	13.56
40 wt% Alfa	72.96	98.88	277.96	93.54	377.96	52.51	477.96	21.52	18.79
30 wt% Sisal	92.13	95.11	243.83	85.43	373.61	46.04	519.93	20.05	18.15
40 wt% Sisal	64.06	84.24	247.39	90.24	352.11	55.24	496.61	26.07	23.13
30 wt% Alfa-Sisal	86.38	89.87	239.08	87.88	356.87	50.13	490.39	21.38	18.19
40 wt %Alfa-Sisal	67.50	96.10	247.50	87.44	375.50	56.90	482.44	26.49	24.07

have thermal stability. In the first stage, the mass loss of the treated fibers is less than that of untreated fibers, indicating less moisture in the treated fibers. Note that the fibers studied have thermal stability up to temperatures 192, 216, 156, and 218°C. Then, a second mass loss testified the maximum of thermal degradation process. We observed an increase of the decomposition temperatures of the treated fibers in comparison with the untreated fibers. As previously described, this behavior suggests that celluloses with higher crystallinity have higher thermal stability. In addition, it is interesting to note that at 400°C, the mass loss of the untreated samples is greater than the fibers treated with NaOH since their lower purity [17], which is attributed to the degradation of lignin and other remaining fiber components [16]. The mass loss continues until 600°C. The heating up to 600°C revealed that the mass loss and the residual weight of the untreated samples were greater than those of alkali treated and corresponds to 27.56 and 24.48 wt% for untreated and treated Alfa fibers, respectively, and 24.35 and 13.06 wt% for untreated and treated Sisal fibers, respectively.

This result is due to the greater amount of cellulose available in the treated fibers. The results showed that with the NaOH treatment, the fiber becomes more heat resistant. These observations indicate that the improved thermal stability is mainly affected with the chemical treatment, where hemicellulose and lignin are eliminated [16-18]. It is important to note that the treated fibers show a stable behavior at 180°C, which corresponds to the crosslinking temperature of composites, which

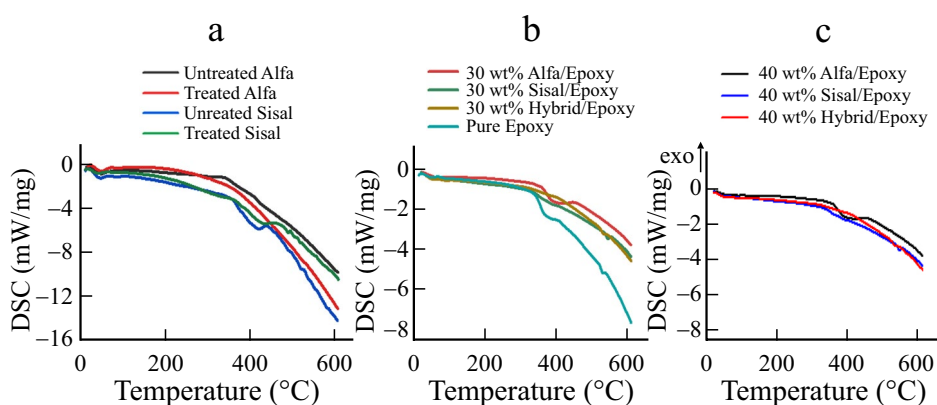


Fig. 6. DSC curves: fibers (a), 30 (b) and 40 wt% (c) fibers /epoxy.

allows their use as a reinforcing material for the composites materials. Figure 2b represents the curves of the different types of composites. The TGA at 600°C revealed that the mass loss and the residual mass of the samples with 40 wt% reinforcement were greater than those with 30 wt% reinforcement. The residual mass corresponding to each type of material is represented in Table 2. It is deduced that the increase in the volume fraction increases the thermal resistance of the materials.

## 5.2. DTG analysis

DTG curves of fibers presented in Fig. 5a indicate the first peaks below 100°C corresponding to the evaporation of water for the treated and untreated Alfa and Sisal fibers. A series of second wider peaks were observed at 330 and 348°C for untreated and treated Alfa fibers, respectively, and 300, and 333°C for untreated and treated Sisal fibers, respectively. This loss of mass is associated with chemical decomposition of the hemicellulose and glycosidic bonds of cellulose. Note that the thermal decomposition of untreated samples is more advanced indicating their low thermal stability in comparison with the treated samples. The greater final decomposition temperatures of treated samples are due to their high molecular arrangement, because some components were eliminated by the treatment. In the second stage of degradation, untreated fibers degrade slightly more and speedily than treated ones; this allows us to say that thermal decomposition requires high energy. The DTG for the different types of materials (Figs. 5b and 5c) indicates a first peak below 100°C related to water evaporation. As for the fibers, a second wider peak appears indicating chemical decomposition. Note that the decomposition temperature of composites reinforced by 40 wt% of fibers is greater than at 30 wt%. The maximum decomposition temperatures corresponding to the peaks are presented in Table 2 [18].

## 5.3. Differential scanning calorimetry

Differential scanning calorimetry (DSC) is able to quantify the thermal changes in both fibers and composites as a function of temperature. The endothermic reaction of the composites with 40 wt% fiber reinforcement is greater than that with 30 wt%. In fact, the enthalpy of pure resin is 100.44 J/g which decreases to 21.71 J/g for 30 wt% Alfa/epoxy and increases to 42.51 J/g for 40 wt% Alfa/epoxy; the same growth sequence was observed for Sisal/epoxy and Alpha-Sisal/epoxy hybrid materials. Bessa et al. [18] reported that the reinforcement of Alfa-treated fibers affects the amount of endothermic enthalpy. It reduces from -305.27 to -181.63 J/g when the fiber content increased from 0 to 30 wt%.

For all types of fibers, the curves in Fig. 6a show an endothermic peak around 49.04 and 49.06°C for untreated and treated Alfa fibers, respectively, and 48.58 and 43.11°C for untreated and treated Sisal fibers, respectively, indicating the start of water evaporation.



TABLE 3. Onset ( $T_{onset}$ ) and Peak ( $T_{peak}$ ) Temperatures of Decomposition and Heat of Decomposition ( $Q_d$ ) of Different Types of Fibers and Composites

Specimen	$T_{onset}$ , °C	$T_{peak}$ , °C	$Q_d$ , J/g
Fibers			
Untreated Alfa	49.04	–	131.28
Treated Alfa	49.06	–	185.20
Untreated Sisal	48.58	413.58	171.52
Treated Sisal	43.11	428.11	108.45
Pure Epoxy	48.69	372.53	100.44
Epoxy composites with Alpha, Sisal or Hybrid fibers			
30 wt% Alfa	60.46	370.46	21.71
40 wt %Alfa	58.10	405.33	42.51
30 wt % Sisal	49.49	–	40.10
40 wt% Sisal	49.50	534.32	57.23
30 wt % Hybrid Alpha-Sisal	50.00	375.00	53.37
40 wt% Hybrid Alpha-Sisal	51.08	523.96	73.72

TABLE 4. Mechanical Characteristics of Untreated and Treated Alfa and Sisal Fibers in Tension

Specimen	$\varnothing$ , $\mu\text{m}$	$\sigma_{max}$ , MPa	$\varepsilon_{max}$ , %	$E$ , GPa
Untreated Alfa	218.66±57.12	6882.94±245.19	2.07±0.68	18.58±0.41
Treated Alfa	106.95±32.30	9220.54±276.81	2.49±0.79	41.25±0.63
Untreated Sisal	163.14±35.33	2762.21±122.00	13.30±3.75	14.69±0.39
Treated Sisal	108.15±28.01	5539.62±133.45	3.03±0.68	17.67±0.64

For Sisal fibers a second endothermic peak appears around 413.58 and 428.11°C. It is attributed to the decomposition of cellulose. No other exothermic or endothermic peaks were observed between the first and second peaks, indicating that both fibers remained stable and retained their mass. The enthalpy of the different samples can be calculated by integrating over time (in seconds), i.e., the graph area corresponds to the enthalpy of the sample transformation. The different values taken from the curves are presented in Table 3 [18].

For all types of materials, the curves (Figs. 6b and 6c) represent an endothermic peak at the start of heating indicating the beginning of water evaporation. The different values obtained from the curves are presented in Table 4. According to these values, it is noted that the quantity of enthalpy of materials reinforced by 40 wt% fibers is greater than that reinforced by 30 wt%.

## 6. SEM Observation

SEM observation was performed to observe the morphology and cell walls of untreated and treated fibers (Fig. 7). The long untreated Alfa (Fig. 7a) and Sisal (Fig.7 c) fibers are composed of bundles linked together by lignocellulosic constituents that act as the link element which protects the plant from external effects. They are covered with impurities made of cells and other constituents, such as waxes and pectin. The fibers walls are composed of microfibrils or fibrils of very small dimensions. After chemical treatments, the roughness of the fibers is attenuated and cleaned. As shown in (Fig. 7b) and (Fig. 7d), the roughness of the fibers reduced after treatment. A smoother surface morphology of treated fibers in comparison with untreated ones and the microfibrils are more visible because the treatment dissolved the cement link between them; the same remark was done in [10]. The chemical alkali treatment attacked the hemicellulose contents of the fibers, so they were

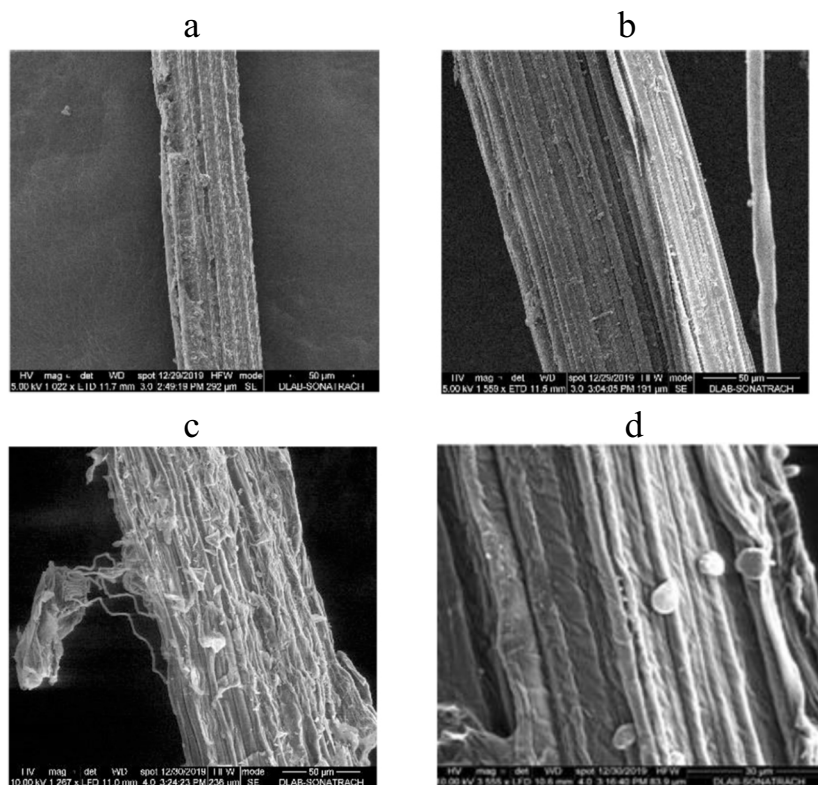


Fig. 7. Fiber SEM observations: untreated (a) and treated (b) Alfa, untreated (c) and treated (d) Sisal.

cleaned, the impurities were eliminated, and their diameter reduced. The treated fibers represent a residue rate lower than that of the untreated fibers. This has been proven by the SEM images which clearly show the reduction in residue deposits on the fiber (the microfibrils are clear and visible after the alkali treatment). The decrease in residues was also demonstrated by the decreasing diameter of the fibers after treatment Fig. 7.

## 7. Fiber Static Tensile Test

Prior to testing, the diameter of each fiber selected was measured. In general, fibers have complex morphology which depends on environmental conditions, age, season, and location. Each fiber is considered cylindrical, with an average diameter of three measurements taken at three different locations of the fiber (at both ends and in the middle). The diameter was determined by means of an OPTIKA optical microscope equipped with a camera and controlled by the Action-vision software. Tensile mechanical characterization tests of treated and untreated Sisal and Alfa fibers were performed according to ASTM D3822-07 on the Zwick/Roell Z010 universal test machine with 5 kN load at a constant speed of 2 mm/min and a gauge length of 80 mm. 20 samples of each type of untreated and treated fibers were used.

## 8. Results and Discussion

It can be seen that the fibers of Alfa have a better elastic behavior than that of Sisal. By comparing the values of the Young's modulus  $E$  of untreated ( $18.56 \pm 0.41$  GPa) and treated ( $41.25 \pm 0.63$  GPa) Alfa fibers and untreated ( $14.69 \pm 0.39$  GPa) and treated ( $17.67 \pm 0.64$  GPa) Sisal fibers (Table 4), we conclude that the chemical treatments used increase significantly

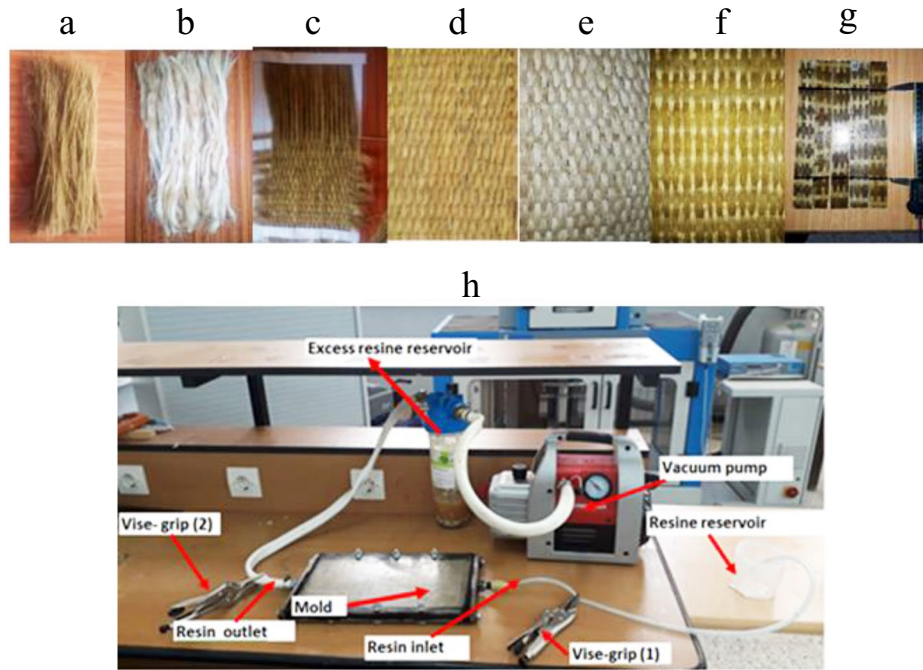


Fig.8. Composite material elaboration; Alfa (a) and Sisal (b) fibers; manufacturing cloth process (c); Alfa (d), Sisal (e) and woven, and hybrid (f) cloth; specimens (g) and VARTM (h).

the elastic property of the fibers. Arthanarieswaran et al. [19] and Khaldi et al. [20] determined the following values of tensile Young's modulus for Banana (3.50 GPa), Alfa (28.43 GPa) and Sisal (25.01 GPa) fibers.

The determination of the standard deviation allows us to evaluate the dispersion of the results relative to the mean. For the Young's modulus, the standard deviation of the treated fibers is greater than the untreated ones. The calibration of the moduli determined for the 20 samples is close to the average value.

## 9. Preparation of the Alfa/Epoxy Resin Composites

### 9.1. Weaving of material

Using treated long Alfa and Sisal fibers (Figs. 8a and 8b) of same length and equal weight strands were elaborated; giving rise to 480 g/m<sup>2</sup> and 2-mm thick Alfa, Sisal, and hybrid hand-made woven satin cloths. Hybrid Alfa/Sisal cloth is composed of 50% of each type of fiber ((Figs. 8d and 8f)). The strands (warp thread) of the fibers of each type of cloth to be manufactured are stretched, mounted, and kept parallel with 5-mm spacing by stitching their ends to fixed double-sided adhesive tapes. In the normal direction to these strands, a needle is used to insert individual strands (weft thread): the first (next) weft strand passes alternately over (under) and under (over) the warp strands. This process is repeated to achieve the desired dimensions of the cloth (Fig. 8c).

### 9.2. Elaboration of composite materials by VARTM

The composite material was fabricated by VARTM (Fig. 8h) in an aluminum mold of 300-mm length, 170-mm width, and 4-mm thickness at room temperature. First, a mold release agent was used to facilitate the release of each type

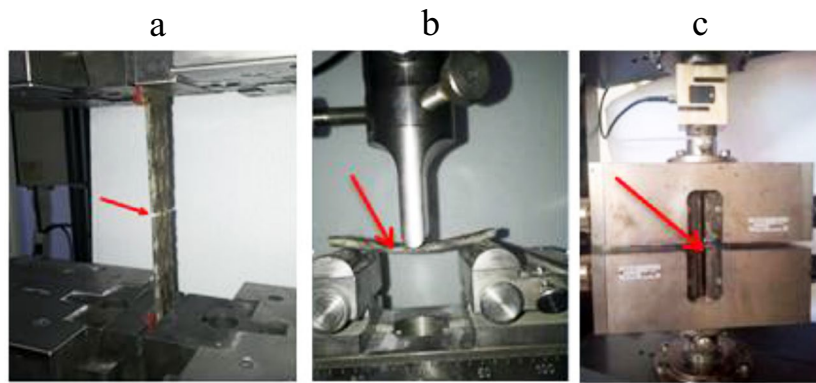


Fig. 9. Mechanical characterization: static tensile test (a), three-points bending test (b), and compression test (c).

TABLE 5. Breaking strain ( $\epsilon_{ult}$ ), Tensile strength ( $\sigma^m$ ), and Young's modulus ( $E$ ) of woven Alfa, Sisal and Hybrid reinforced Epoxy

Specimen	$\epsilon_{ult}$ , %	$\sigma^m$ , MPa	$E$ , GPa	Rise, %
Pure resin epoxy	3.94±0.05	57.55±1.12	2.85±0.007	–
Epoxy composites with Alpha, Sisal, and Hybrid fibers				
40 wt% Alfa	2.38±0.45	79.08±3.91	12.35±0.99	333
30 wt% Alfa	1.73±0.11	44.65±2.98	7.4±0.5	160
40 wt% Sisal	4.52±0.58	84.14±5.96	4.99±0.28	75
30 wt% Sisal	3.37±0.23	27.44±6.02	3.08±0.20	8
40 wt% Hybrid Alfa-Sisal	3.80±0.37	53.56±4.00	4.47±0.51	57
30 wt% Hybrid Alfa-Sisal	4.29±1.26	49.47±9.01	3.34±0.62	17

of composite. Each cloth of reinforcement is put into the mould. Prior to starting injection, care was taken to ensure that the pump-mold circuit is hermetic: the resin supply line is blocked by the vise-grip long nose (1). The vacuum pump dial indicates (–1000 mbar), which has to be maintained during operation and shutdown. This allowed the hydrophilic fibers to be dehumidified and air to be evacuated so as to ensure good fiber/resin adhesion. The resin injection starts and then stops first by the resin outlet vise-grip long nose (2), once the volume of the mould is completely filled, then by the second resin inlet vise-grip long nose (1). The excess resin reservoir prevents resin residues from reaching the vacuum pump and damaging it. The demolding process is done after 12 h, when the complete cross-linking of the resin is achieved. The material obtained is put in an oven (MEMERT UN110) at 80°C during 8 h, and then cooled to room temperature to ensure structure homogenization required by ISO 527 standard mechanical characterization tests (Fig. 8g).

## 10. Mechanical Characterization of Composites

### 10.1. Tensile Tests

The mechanical properties of the samples were measured by tensile tests (Fig. 9a). They were tested under uniaxial loading according to ISO-527-4 standard on a universal machine of the Zwick/Roell Z010 type with a speed of 2 mm/min and capacity charge of 10 kN. Three samples of pure epoxy resin and five samples (250×25×4 mm) according to the ISO 527 standard of each reinforced composite were tested. The Young's modulus, maximum stress and strain of each sample were determined (Table 5).

TABLE 6. Values of Bending strength ( $\sigma^{bu}$ ), Bending Modulus ( $E_b$ ), Force ( $F_{max}$ ), Ultimate Strain ( $\varepsilon_{max}$ ) of Materials Determined in Three-Point Bending Test

Material	$D_{max}$ , mm	$F_{max}$ , N	$\sigma^{bu}$ , MPa	$\varepsilon_{max}$ , %	$E_b$ , GPa	Rise, %
Pure epoxy	17.40±3.22	99.80±16.07	59.88±9.64	0.10±0.02	3.17±0.42	–
Epoxy composites						
40 wt% Alfa	4.60±0.84	127.49±16.68	76.49±10.01	0.03±0.00	6.7±0.81	113
30 wt% Alfa	3.85±0.31	75.60±9.24	45.36±5.54	0.02±0.00	5.03±0.05	60
40 wt% Sisal	10.89±4.73	132.06±20.08	79.24±12.05	0.06±0.03	5.64±0.44	79
30 wt% Sisal	7.80±1.24	119.07±12.14	71.44±7.28	0.05±0.01	5.35±0.61	69
40 wt% Hybrid	8.03±2.07	88.99±12.52	53.39±7.51	0.05±0.01	6.47±0.51	105
30 wt% Hybrid	6.19±1.19	103.05±17.69	61.83±10.61	0.04±0,01	6.45±0.66	104

TABLE 7. Displacement ( $w$ ), Force ( $F_{max}$ ), Strength ( $\sigma^{cu}$ ), and Modulus ( $E$ ) in Compression of Woven Alfa, Sisal and Hybrid Reinforced Epoxy

Material	$w$ , mm	$F_{max}$ , kN	$\sigma^{cu}$ , MPa	$E$ , GPa	Rise, %
Pure Epoxy	3.99±00	1.43±0.12	35.65	3.87±0.20	//
40% Woven Alfa /Epoxy	2.02±1.13	2.88±0.09	72.09	7.02±0.97	81
30% Woven Alfa /Epoxy	3.034±1.23	2.32±0.05	58.05	4.59±0.61	19
40% Woven Sisal /Epoxy	3.26±1.07	1.69±0.12	42.37	5.85±0.24	51
30% Woven Sisal /Epoxy	3.99±00	1.78±0.57	44.58	5.57±0.66	44
40% hybrid Woven /Epoxy	2.49±0.56	1.07±0.22	26.70	5.71±0.45	48
30% hybrid Woven /Epoxy	3.34±0.39	1.09±0.18	27.24	4.58±0.13	19

## 10.2. Three-point bending tests

Three-point bending tests were performed (Fig. 9b). The tested sample is placed on two supports separated by a distance of 16 times the sample thickness. Midway of the supports, a 10 kN force is applied with constant loading punch speed of 2 mm/min. Four specimens of 80×10×3 mm were tested on a Zwick/Roell Z010 machine type according to ISO 178 standard. The Young's modulus, maximum bending stress, and strain of each sample was determined (Table 6).

## 10.3. Compression tests

A series of compression tests of the composites fabricated were carried out in order to evaluate their mechanical properties (Fig. 9c). Four specimens of 10×120×4 mm with the gauge length of 10 mm were tested according to the EN ISO 14126 standard on a Zwick/Roell Z010 machine equipped with a 10 kN force sensor. The loading was incremented at a displacement speed of 2 mm/min. The axial displacement was measured and loading stopped after the displacement exceeded 4 mm. Four tests were performed for each specimen (Table 7).

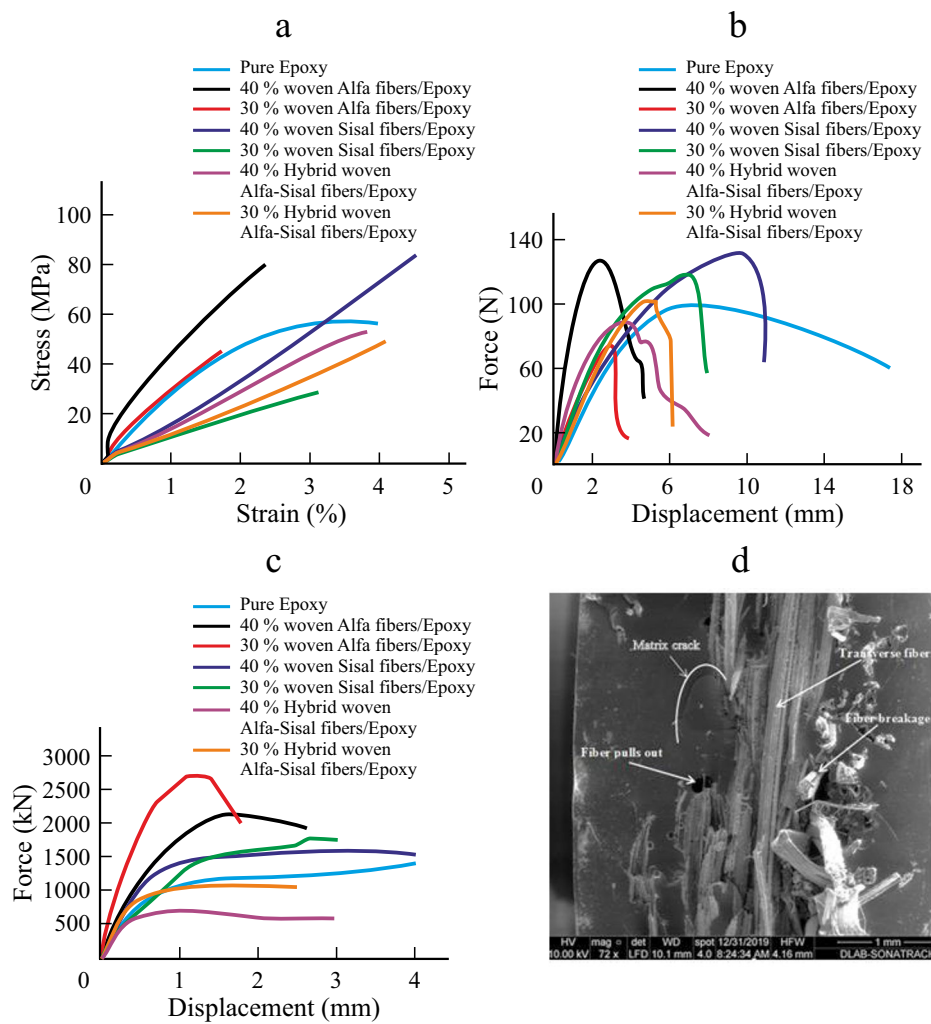


Fig.10. Composites mechanical properties in tension (a), three-point bending (b), and compression (c) and fracture mechanism (d).

## 10.4. Results and discussion

**10.4.1. The static tensile characterization.** The static tensile test results of the composites were compared with those of the pure epoxy resin. The Young's modulus of the Alfa/epoxy composites with 40 and 30 wt% Alfa fibers increased about 333 and 160%, respectively, in comparison with that of pure epoxy. The Sisal/epoxy composite with 40 and 30 wt% sisal fibers demonstrated Young's modulus increasing of 75 and 8%, respectively. Finally, 40 and 30 wt% hybrid woven Alfa-Sisal/epoxy composites have 57 and 17% increasing, respectively. The weight fraction of reinforcing long fibers and their chemical treatment clearly influenced on material resistance. Therefore, good interfacial adhesion due to the alkaline treatment, which makes the fiber surface waterproof and removes the non-cellulosic components (wax, pectins and hemi-cellulosic lignin). Whereas material made with Sisal fibers seems to be governed by the elastic behavior of the latter fiber. The values of ultimate strain of Sisal/epoxy composites with 40 and 30 wt% of fibers are  $4.52 \pm 0.58$  and  $3.37 \pm 0.23\%$ , respectively. Then, the fibers add elasticity to the material (Fig. 10a). Material reinforced by Alfa or Sisal fibers showed brittle behavior with displacement of  $3.128 \pm 0.67$  and  $6.34 \pm 0.9$  mm in the case of Alfa/epoxy and Sisal/epoxy composites with 40 wt%, respectively, compared to that of pure resin equaled to  $6 \pm 0.5$  mm. The hybridization of the fibers (Alfa-Sisal) confers to the matrix more rigidity and elastic behavior. The average values of 5 samples of the maximum stress and strain for each type of material are presented in Table 5.

Alfa fibers have a very irregular diameter (wide at the base and thin at the end of the fiber) resulting in a large standard deviation unlike Sisal fiber, which has a diameter considered to be regular; which explains the significant standard deviation of the woven Alfa/epoxy composite compared to that of woven Sisal/epoxy one [21]. The fracture mechanism in traction of the composite was analyzed by SEM. From the obtained images (Fig. 10d), it is seen that the reinforcement is well incorporated in the middle of the matrix and the transverse fibers have not been broken and adhered well to the matrix. Some cracks in the matrix and the fracture of the transverse fibers leaving some empty holes are also observed.

*10.4.2. Three-point bending characterization.* The experimental results obtained from six types of composites demonstrated the beneficial effect of adding Alfa and Sisal fibers as reinforcement to epoxy matrix composites. In comparison of pure epoxy, values of Young's modulus of epoxy composites reinforced by 40 and 30 wt% of Alpha fibers increased about 113 and 60%, respectively, and epoxy composites with 40 and 30 wt% of hybrid fibers increased about 105 and 104%, respectively. Sisal/epoxy composites with 40 and 30 wt% of reinforcement demonstrated 79 and 69 wt% increase, respectively. Composites reinforced Alfa fibers had brittle behavior; their displacement was  $4.60 \pm 0.84$  and  $3.85 \pm 0.31$  mm at 40 and 30 wt% of Alfa fibers, respectively, in comparison with that of pure resin equaled to  $17.40 \pm 3.22$  mm (Fig. 10b).

The bending strength  $\sigma^{bu}$  and the strain  $\varepsilon$  can be calculated as follows [22].

$$\sigma^{bu} = \frac{3Fd}{2bh^2}, \quad (1)$$

$$\varepsilon = \frac{6hD}{d^2}, \quad (2)$$

where  $F$  is the breaking force (N),  $d$  is the distance between supports (mm),  $b$  is the width of the specimen (mm),  $h$  is the thickness of the specimen (mm),  $D$  is the maximal deflection (mm),  $\varepsilon$  is the strain at the maximal deflection. The results obtained are presented in Table 6.

Moreover, as indicated in Table 6, the bending modulus determined for each tested epoxy composite is much greater than the value of 3.1 GPa, and 57 MPa corresponding to Flax yarn long/SPI [8] and Polyester/Alfa [22].

The bending strength values  $76.49 \pm 10.01$  and  $79.24 \pm 12.05$  MPa corresponding epoxy composites with 40 wt% of Alfa and Sisal fibers, respectively, (see Table 6) are much greater than the values 5.79 MPa of Plaster/Alfa [23], 61.1 MPa of Sisal/Polyester [14],  $50.86 \pm 3.79$  of Bagasse/Polyester [24],  $59.57 \pm 4.2$ MPa of Sisal /Polypropylene [16], 49 MPa of Flax yarn long/SPI [8], 57 MPa of Alfa/ Polyester [22], 28.3MPa of Biopolymer matrix/Alfa [3] and smaller than  $172.9 \pm 0.49$  MPa of Luffa/Glass /Polyester [25].

*10.4.3. Compression characterization.* Force-displacement dependences in compression of the materials tested are presented in Fig. 10c. Epoxy composites reinforced by 40 and 30 wt% of Alpha fibers had a greatest rigidity and were loaded up to the maximum force of  $2883.46 \pm 90.91$  and  $2322.09 \pm 50.29$  kN, respectively, at the ultimate displacements of  $2.02 \pm 1.13$  and  $3.034 \pm 1.23$  mm, respectively. Epoxy composites made from Sisal fibers demonstrated typical rubber-like plastic behavior. Epoxy composites reinforced by 40 and 30 wt% Sisal fibers failed at the maximum force of  $1694.61 \pm 124.83$  and  $1783.07 \pm 565.16$  kN, respectively, and ultimate displacement of  $3.26 \pm 1.07$  and  $3.99 \pm 00$  mm, respectively. Hybrid epoxy composites demonstrated the deformation behavior with initial elastic stage and subsequent plasticity stage. Hybrid epoxy composites reinforced by 40 and 30 wt% of Alpha and Sisal fibers failed at maximum force of  $1067.81 \pm 224.38$  and  $1089.62 \pm 179.11$  kN, respectively, at ultimate displacement of  $2.49 \pm 0.56$  and  $3.34 \pm 0.39$  mm, respectively. Hybrid epoxy composites reinforced by 40 and 30 wt% of Alpha and Sisal fibers had compressive strength of 72.09 and 58.05 MPa, respectively, which was much greater than the values of 30.53 MPa of Nano filler/epoxy [26], 19.9 MPa of Kenaf/Geopolymer-foam-fiber [27], 52.9 MPa of Kenaf/Cement, and 51.8 MPa of Jute/Cement [28] composites. Values of the maximum force at failure, ultimate displacement, and the compressive elastic modulus in compression are represented in Table 7.



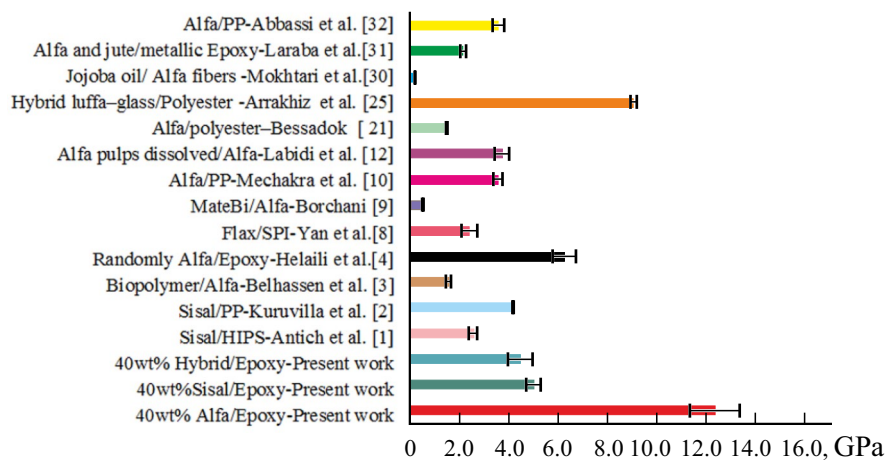


Fig. 11. Comparison of Young's moduli of composites reinforced with different natural fibers.

## 11. Discussion

Mechanical characteristics experimentally determined for epoxy composites made using natural fibers (see Fig. 8) were compared with available in literature. Bessadok et al. [21] used chemically treated Alfa fibers for unsaturated polyester composites reinforcement and showed that chemical treatments increased Young's modulus from 1.46 to 1.47 GPa. Ben Brahim and Ben Cheikh studied the influence of fiber orientation and volume fraction on the tensile properties of Alfa-polyester composite. The heterogeneity and non-woven nature of the raw material made it difficult to handle, so the fibers were soaked in an aqueous solution of starch at a concentration of  $20 \text{ g}\cdot\text{L}^{-1}$  to prevent dispersion [29]. The Young's modulus obtained at 44 wt% of Alfa fibers reinforcement equaled 9.0 GPa. However, the author's investigation did not take into account the hydrophilic character of fibers which was shown to be significant in the current work. In fact, the Alfa fibers contain quantities of humid air which induce defects of inclusions at the Polyester/Alfa interface. This drawback was obviated by using the VARTM in the current work. Mokhtari et al. [30] fabricated ten biocomposite materials by casting method using Alfa cellulose fibers as a reinforcement and biobased PUs as matrices prepared from Jojoba oil. The maximum value of Young's modulus obtained was 165.4 MPa. Laraba et al. [31] developed a sandwich panel composed of Alfa fiber-based core and a Hybrid polymer matrix composite (Jute and metallic mesh) as skin with Young's modulus 2.16 GPa. A review on Alfa fiber (*Stipa Tenacissima L.*): From the plant architecture to the reinforcement of polymer composites was made by El-Abbassi et al. [32] reviewed the polymer composites reinforced by Alfa fibers. The Young's modulus was 3.57 GPa.

The experimental results obtained were compared to those described in literature for composites with short or long reinforcing fibers, regardless of the reinforcement rate. The comparison showed that the value of Young's modulus of the epoxy composite fabricated in this work with 40 wt% Alfa fibers is the greatest one (Fig. 11).

## 12. Conclusion

Mechanical behavior of composites, consisting of epoxy resin matrix reinforced by 30 and 40 wt% of satin cloth made of long Alfa or Sisal fibers and Hybrid Alfa/Sisal epoxy composites was investigated. After extraction, an optimal alkaline sodium hydroxide treatment developed in our previous investigation (Mechakra et al. [10]). SEM, FTIR, and chemical decomposition of each type of fibers demonstrated the effectiveness of alkaline treatment. Chemical analyses performed on different types of fibers provided the cellulose, hemicellulose, and lignin contents which were applied to establish that the rate of cellulose is more important for the treated fibers than for the untreated ones.

TGA and DTG analyses allowed us to reveal that chemical treatment ensures good thermal stability to the fibers. Natural reinforcing fibers allow a better thermal stability behavior of composites. The results showed that increasing the



weight fraction of the fibers increases the thermal stability of the composite. DSC was able to quantify enthalpy changes in fibers and composite materials as a function of temperature. A similar study was conducted by Tesfay et al. [16] on treated and untreated Sisal fibers. It showed an increasing the amount of heat as a function of the increasing the volume fraction of natural fibers. The endothermic reaction of the composites studied containing 30 wt% of reinforcing fibers is less than that containing 40 wt% of fibers.

Indeed, the quantity of enthalpy of the pure resin equaled to 100.44 J/g decreases to 21.71 J/g for epoxy composite with 30 wt% of Alfa fibers and increases to 42.51 J/g for composite with 40 wt% of Alfa fibers; the same tendency was observed for Sisal/epoxy and the hybrid Alfa-Sisal/epoxy composites.

Six different composites were fabricated by VARTM. The results obtained revealed a significant increase of mechanical properties, in particular for epoxy composite with 40 wt% of Alfa fibers. This composite demonstrated the following values of elastic modulus: 12.35 GPa (in tension) 6.71 GPa (in three-point bending), and 7.02 GPa (in compression) which significantly greater the corresponding values for pure epoxy resin: 2.85 GPa (333% rise), 3.17 GPa (113% rise), and 3.87 GPa (81% rise), respectively. These findings were confirmed by extending the comparison in our previous work with PP/short Alfa fibers ( $E = 3.6$  GPa) [10], and the transverse Young's modulus ( $E_t = 3.6$  GPa) of the Alfa/Polyester composite [29].

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