EFFECT OF ALKALINE TREATMENT TIME ON FLEXURAL PROPERTIES OF ALFA FIBER/UNSATURATED POLYESTER COMPOSITE

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Environmental and energy conservation pressure has led to a dramatic increase in the need for economically feasible lightweight materials that can be better substituted for non-biodegradable materials in reinforced composites. To this end, this study examines composite materials prepared from unsaturated polyester resins reinforced with treated and untreated Alfa fibers. Fiber treatment was carried out by NaOH solution of 5% concentration at different times (1, 3, 5 and 24 h). The strength and flexural modulus of the composites were evaluated according to the ASTM D790 test method. The analytical results indicate that the fibers' alkaline pretreatment time had a positive influence on the mechanical properties of the composites.

Keywords: composite, *Stipa tenacissima*, adhesion, flexion, hydrophilic, alkaline treatment

INTRODUCTION

Natural fibers have been used as reinforcement in the manufacture of composite materials,[1-3] particularly in packaging, healthcare, pharmaceuticals, automotive, and aerospace fields.[4-7] They have many advantages, such as availability, low cost, low density, rigidity, biodegradability, and high mechanical behavior.[5,8,9] However, the main problem they face in this field is the fiber–matrix interface, as natural fibers have a hydrophilic character, while polymers have a hydrophobic character, resulting in poor adhesion.[10,11] Many factors can improve the adhesion between fibers and polymers to overcome this problem, such as alkali treatment, permanganate, dichromate and silane treatment. The most commonly used treatment is the alkaline treatment:[12]

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

Many researchers have observed that preparing such natural fiber-reinforced composites can improve the mechanical efficiency of these composites.[13] Laib *et al.*[12] prepared a composite material from an unsaturated polyester matrix with Luffa fibers, which underwent different chemical treatments (NaOH, permanganate, dichromate, silane and bleaching) to improve the fiber–matrix interfacial adhesion. The results revealed a maximum improvement in flexural strength of about 23.8% for the bleach-treated composites. Mishra *et al.*[14] treated sisal fiber with different soda concentrations (5 and 10%). They showed that the tensile strength of the composite treated with 5% NaOH is higher than that of the composites treated with 10% NaOH.

According to Ajouguim *et al.*,[15] who treated Alfa fiber with 6% sodium hydroxide at different times (1, 2, 4, 6 and 24 h), the alkaline treatment for 6 h and the hydrothermal treatment for 1 h were more favorable, due to the removal of organic matter, which increased the roughness and crystallinity index. Rokbi *et al.*[16] prepared composites based on unsaturated polyester reinforced with NaOH-treated Alfa fibers. The treated fiber composites provided higher flexural strength, compared to the untreated Alfa fiber composites; 10% NaOH resulted in a 60% increase in flexural strength and a 62% increase in modulus. Boukhoulda *et al.*[17] also treated Alfa fibers with sodium hydroxide at different concentrations (9, 10, 11 and 12%).

The results show that the composite reinforced by the 9% NaOH treated fiber presents the highest tensile strength.

Benyahia *et al*.[18] studied the effect of alkali treatment on fiber-reinforced unsaturated polyester composites. The authors treated Alfa fibers with different concentrations of NaOH (1, 3, 5 and 7%) for 24 h to improve the studied composite's mechanical properties (flexural strength). The results indicate that the flexural strength is significantly improved, especially in the composite reinforced with Alfa fibers treated with NaOH (7%). The present study investigates the effect of alkaline treatment time on the mechanical properties of a composite material based on a thermosetting matrix (unsaturated polyester) reinforced with Alfa fibers.

EXPERIMENTAL

Raw material

Alfa fibers were harvested in the Hodna region (M'sila, Algeria) in November 2019. The fibers were rinsed with tap water to remove dust and pollutants, and then air-dried for 3 days at room temperature.

Alkaline treatment of Alfa fibers

Alfa fibers were immersed in sodium hydroxide solution at a concentration of 5% (w/v) for (1, 3, 5 and 24 h). The ratio (fibers/solution) used was 2:3 (w/v). After the treatment, the fibers were rinsed with acidified water (containing 2% acetic acid) for 3 min. Then, the rinsing with tap water was repeated several times to eliminate the excess traces of soda. Finally, the fibers were dried for 5 days at room temperature and then, in an oven at 100 °C for 6 h. The fibers used in this preparation were 1 cm in size.

Composite preparation

The matrix used for this study was unsaturated ortho-phthalic polyester, cured using 2% methyl ethyl ketone peroxide. Table 1 summarizes the characteristics of the unsaturated polyester matrix. The composites were made using a wooden mold with 200 x 20 x 4 mm^3 dimensions. The fiber content in the composite was set at 30% (v/v). The fiber size was 1 cm. The composite was prepared by the lay-up method. The fibers were imprinted in the matrix, and the mixture was poured into the mold to obtain the samples (Fig. 1). Table 2 lists the codes used to denote the different samples.

Fourier transform infrared spectroscopy (FTIR)

The spectra of the samples were acquired by a SHIMADZU IR Affinity-1S Fourier Transform Infrared Spectrophotometer (Japan), installed at the Laboratory of Chemistry of the University of Setif, Algeria. The samples were prepared in the form of tablets: a mixture of a small amount (~5% by mass) of previously ground Alfa fiber (treated or untreated) with 95% by mass of potassium bromide (KBr). The scanning range was between 400 and 4000 cm^{-1}, with a resolution of 2 cm^{-1}.

X-ray diffraction (XRD) analysis

The crystallinity index and fibril orientation can directly affect Young's modulus and mechanical properties, so the evaluation of the fibers used in this study was necessary. Crystallinity index (CrI) values of treated and untreated Alfa fibers were determined on a D8 diffractometer (Bruker-AXS, Germany) with copper radiation. The CuKα (λ = 1.54 Å) was operated at 40 kW and 20 mA with a velocity of 5°/min over a 2Θ range from 5° to 70°. The crystallinity index (CrI) of the samples was calculated based on the diffraction intensities given by Segal *et al*.:[19]

$$CrI\% = \frac{I_{002} - I_{am}}{I_{002}} \quad (2)$$

where CrI is the crystallinity index, $I_{(002)}$ is the intensity of the (002) plane crystal phase at 2θ = 22°, and I_{am} is the intensity of the amorphous phase at 2θ = 17°.

Table 1
Physical and mechanical properties of unsaturated polyester

Unsaturated polyester	Characteristics
Viscosity 25 °C	1000-1800 Cps
Styrene content	39-41%
Acid value	Max. 30 KOH/g
Density	0.09-1.11 Kg/dm^3
Gel time at 25 °C	20-40 minutes
Curing time	50-70 minutes
Exothermic peak	100-120 minutes
Tensile strength	40-60 MPa
Bending strength	80-100 MPa

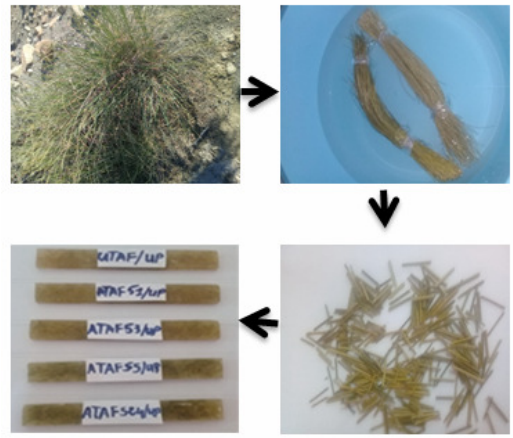

Figure 1: Schematic representation of different stages of treatment

Table 2
Coding of the different composite samples studied

Composites	Codes
UTAF/UP	Untreated Alfa fiber/unsaturated polyester
ATAF51/UP	Alfa fiber treated with 5% alkaline (for 1 h)/polyester
ATAF53/UP	Alfa fiber treated with 5% alkaline (for 3 h)/polyester
ATAF55/UP	Alfa fiber treated with 5% alkaline (for 5 h)/polyester
ATAF524/UP	Alfa fiber treated with 5% alkaline (for 24 h)/polyester

Thermogravimetric test

Thermal properties were studied by thermogravimetric analysis (TGA) to observe the fibers' thermal stability evolution. The measurements were carried out from 20 °C to 800 °C, at a heating rate of 10 °C/min, in a nitrogen atmosphere, using an SDT Q600 TA device (USA).

Scanning electron microscopy (SEM)

After the samples were broken, the SEM test was performed to check the adhesion between Alfa fibers and the matrix. The SEM apparatus used in this test was JOEL JSM 7001F (Japan). The SEM images were obtained by the secondary electronic imaging method, with a beam acceleration voltage of 15 kV.

Mechanical evaluation: three-point bending test

The three-point bending test of the composite was performed using a ZWICK/Roell Z100 type apparatus. The initial load applied was 1N, the maximum load capacity was 100 kN, and the traverse speed was 2 mm/min. The samples were prepared for the test with dimensions of 80 mm × 12.7 mm × 3 mm, with a recommended span to depth ratio of 16:1 according to ASTM D790. The calculation of flexural strength and flexural modulus was performed using Equations (3) and (4):

$$\sigma_f = \frac{3PL}{2bd^2} \quad (3)$$

$$E_f = \frac{PL^3}{4bd^3w} \quad (4)$$

where L is the length of the sample (mm), P is the load applied to the sample (N), b and d are, respectively, the width and the thickness of the sample (mm), and W is the deviation.

RESULTS AND DISCUSSION

FTIR analysis

Figure 2 shows a broad absorption band around 3321 cm^{-1}, corresponding to the hydroxyl group (OH) bound to cellulose and hemicelluloses.[10] The peaks at 2913 cm^{-1} and 2840 cm^{-1} correspond to the vibrations of the aliphatic group of CH and CH_2 present in cellulose and hemicelluloses, respectively.[18,20] Another peak at 1735 cm^{-1} is related to the carbonyl groups C=O due to the partial hydrolysis of hemicelluloses in an alkaline medium.[9] The band at 1640 cm^{-1} is attributed to the stretching vibration of the H-OH bond of water.[21] In addition, a small peak around 1257 cm^{-1} is related to the lignin's vibration of the acetyl group (C=O).

The peak at 1033 cm^{-1} is associated with the vibration of the C-O bond of hemicelluloses.[18] The intensity of the two peaks (1257 cm^{-1} and 1735 cm^{-1}), observed on the spectra of the treated Alfa fibers (ATAF51, ATAF53, ATAF524), decreased compared to those of untreated Alfa fibers. This is due to the partial removal of lignin

and hemicelluloses. Moreover, the two peaks at 3321 cm^{-1} and 1033 cm^{-1} were reduced due to the removal of hemicelluloses components.[16,22,23] Thus, the alkaline treatment and processing time of Alfa fibers caused the dissolution of some fiber components, including lignin and hemicelluloses.[9,15,16]

X-ray diffraction

Figure 3 shows the diffraction spectra of treated and untreated Alfa fibers. The crystallinity index values of the fibers listed in Table 3 show an improvement after the treatment. The highest crystallinity index is 41.3% for the sample (ATAF55), with an increase of 33% compared to the untreated fibers. These results also show that the treatment time has a positive effect and increases the crystallinity index (CrI), since, with a treatment time of 5 h, the crystallinity index reaches the maximum value. Aruan Efendy *et al*.[24] observed the same result: alkali treatment results in a higher crystallinity index and better thermal stability for harakeke and hemp fibers. This increase may be attributed to the removal of amorphous hemicellulose from the fibers,[9,15,24] or there is a possibility of rearrangement of the crystalline regions so that the fiber has a more crystalline nature.[25,26] On the other hand, beyond 5 h, the (CrI) decreases. This can be explained by the fact that a long-duration alkaline treatment can damage the fibers and lead to a decrease in the crystallinity index.[9,15,24]

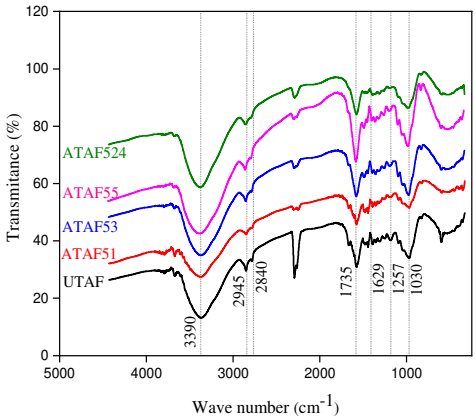

Figure 2: FTIR spectra of treated and untreated Alfa fibers

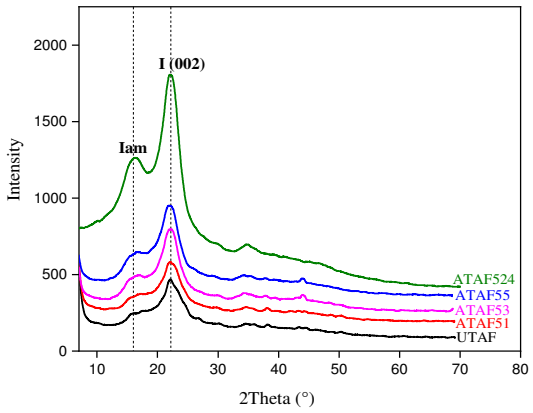

Figure 3: X-ray diffractograms of treated and untreated Alfa fibers

Table 3
Crystallinity index of treated and untreated Alfa fibers

Sample	UTAF	ATAF51	ATAF53	ATAF55	ATAF524
$I_{(002)}$	430	473	684	658	1505
I_{am}	312	276	402	386	900
CrI%	27.7	34	38	41.3	40.2

Thermogravimetric analysis (TGA)

Thermogravimetric analyses were performed to compare the influence of treatments on the thermal stability of the fibers.[27] Figure 4 shows the thermal stability curve of treated and untreated Alfa fiber. Three regions of mass loss can be noted. In the first region, between 25-100 °C, the treated and untreated Alfa fibers have lost a small amount of weight, corresponding to the water molecules' evaporation. Several authors have observed the same result.[5,28] In the second region, between 200-290 °C, untreated Alfa fibers started to decompose around 205 °C, while ATAF55 and ATAF524 started to decompose at about 220 °C and 230 °C, respectively. The decomposition of fibers may be due to the decomposition of hemicelluloses.[15,29] The decomposition in the third region, 290-400 °C, is related to cellulose and lignin decomposition.[28]

It is important to highlight a shift to higher temperatures for samples ATATF51, ATAF55 and ATAF524. The same result was reported by Mohanty *et al*.[30] These results clearly show the improvement of the thermal behavior of NaOH-treated Alfa fibers at different periods. Therefore, the evolution of degradation temperatures of

treated Alfa fibers can be attributed to the effective removal of hemicelluloses, as suggested by FTIR measurements and chemical analysis.[5,27]

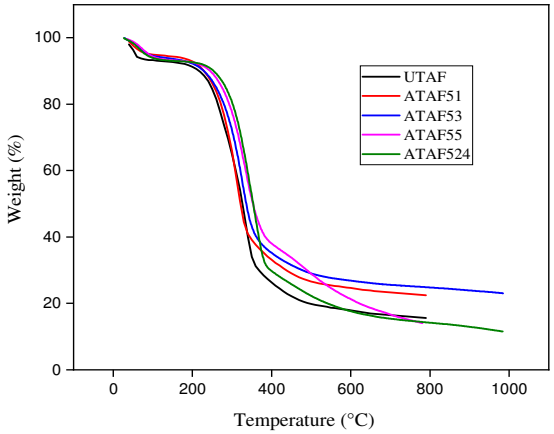

Figure 4: Thermogravimetric curves of treated and untreated Alfa fibers

Effect of alkaline treatment on mechanical properties

Flexural strength

Table 4 summarizes the effect of alkali treatment of the fibers and their incorporation into the unsaturated polyester composite material on the mechanical properties of the composites in flexure. Figure 5 shows the evolution of the flexural strength of the composites as a function of the treatment time. An increase in flexural strength is noticed for ATAF51/UP, ATAF53/UP, ATAF55/UP, and ATAF524/UP composites, respectively, compared to the untreated composite UTAF/UP. The same observation was made by Kabir *et al.*,[31] who studied the effect of alkalinization on hemp fiber reinforced polyester composites. Among all the treatments, the hemp fiber composites treated with 8% NaOH showed the best flexural strength and modulus compared to the untreated and other chemically treated composites.

The ATAF55/UP sample of NaOH treated Alfa fiber composites shows higher flexural strength, *i.e.*, the maximum value is 64.30 MPa for the composite (ATAF55/UP), with a 32% increase rate, compared to the composite (UTAF/UP). This result indicates that longer alkaline treatment time of Alfa fibers ensures better removal of lignin, hemicelluloses and other impurities, allowing the detachment and reorganization of fibers along their main axis, giving rise to a more rigid structure. Thus, the alkaline treatment roughens the fiber's surface and improves the fiber's adhesion strength to the matrix.[18,32,33] However, it can be noted that higher alkaline treatment time can damage the fiber's crystalline structure, which can lead to counter effective results, as in the case of the ATAF524/UP composite.[9,24,34]

Flexural modulus

An improvement in Young's modulus is observed for all composites: ATAF51/UP, ATAF53/UP, ATAF55/UP, and ATAF524/UP, compared to the UTAF/UP composite, which has a value of 1.64 GPa. The maximum value is 5.21 GPa noted for the ATAF55/UP composite, with a 68% increase over the untreated UTAF/UP composite. The alkaline treatment improved the fiber–matrix interaction by removing lignin and hemicelluloses, which led to better incorporation of the fibers into the matrix.[14] These results are similar to those of Benyahia *et al.*,[22] NurAzua *et al.*,[35] Hashem *et al.*[36] and Williams *et al.*[37] This change in the flexural modulus is due to the increase in the bond strength between the fibers and the matrix.[18,38] The reduced mechanical properties in the case of fibers treated at 24 h, which corresponds to the composite ATAF524/UP, may be caused by the thickening of the cell wall, leading to weak adhesion between the matrix and the fiber.[17,35,36]

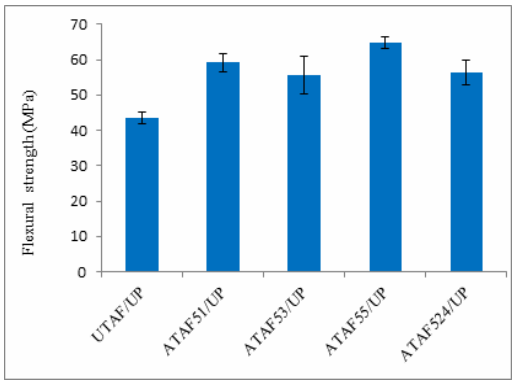

Figure 5: Effect of treatment time on flexural strength of composites

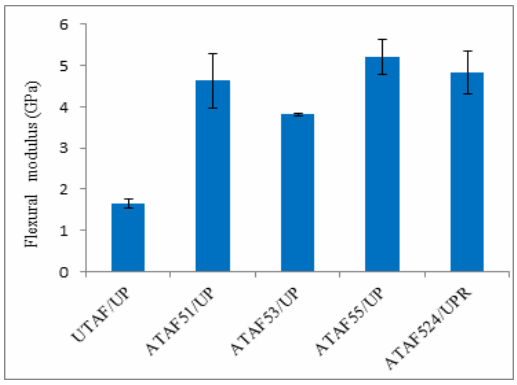

Figure 6: Flexural modulus of treated and untreated Alfa fiber-reinforced composites

Table 4
Mechanical properties of composites

Mechanical properties	UTAF	ATAF51	ATAF53	ATAF55	ATAF524
Flexural strength (MPa)	43.41±2.94	59.13±4.19	55.46±7.41	64.37±2.8	56.4±5.01
Flexural modulus (GPa)	1.64±0.2	4.62±1.1	3.81±1.06	5.21±0.72	4.82±0.91

SEM analysis

Figure 7 shows the SEM analysis of the fracture profiles of the composites reinforced with the treated and untreated Alfa fibers. The fibers' detachment and indentation in the matrix are visible for the composites loaded with untreated Alfa fibers (Fig. 7a-c).

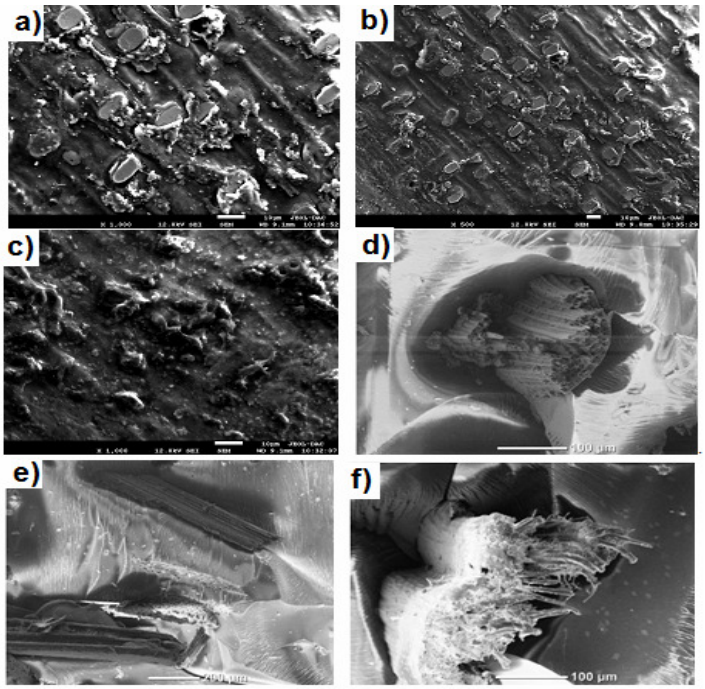

Figure 7: SEM micrographs of composites: (a), (b), (c) UTAF/UP (d) ATAF51/UP, (e) ATAF55/UP, (f) ATAF524/UP

The surface of the fibers is smooth, signifying low adhesion at the fiber–matrix interface, which is common for plant fiber and UP matrix biocomposites. For samples ATAF51/UP and

ATAF53/UP, it can be noted that the treated fiber composites (Fig. 7d-e) show less matrix cracking and fiber debonding, which may contribute to higher stress absorption, resulting in higher flexure. A reduction of holes in the fracture surface of treated fiber composites, compared to untreated fiber composites, is also noted. This result could be due to good interfacial bonding between the Alfa fibers and the matrix. Therefore, fiber extraction was minimized.[37]

Figure 7e shows microfibrils aligned parallel on the outer surface of Alfa fibers. This is due to the dissolution of cementation materials, such as impurities, waxes and pectins, enabled by the alkaline treatment. These results are consistent with the FTIR and XRD findings, which revealed that the crystallinity index increased for the treated Alfa fibers, which, consequently, enhanced the mechanical properties of the composite.[39] After 24 h of treatment, the fibers become a bit brittle (Fig. 7f), illustrating a decrease in the bending properties

CONCLUSION

The study revealed that the optimum value of flexural strength was obtained for the composites reinforced by fibers treated with NaOH solution (5%) for 5 h, compared to the composites reinforced by untreated fibers. The increase in flexural strength was of about 32%, due to better adhesion between the fiber and the matrix. Micrographic observation using SEM provided clear evidence of wax degradation and fatty substances on the surface of Alfa fibers. The changes in the structural content of the fiber after alkaline treatment decreased the contact angle of the fiber, improved the adhesion bonds between the matrix and the fiber, and resulted in better load transfer. This led to improved mechanical properties of the resulting composites. Thus, findings of the study recommend the treatment of Alfa fibers with NaOH at the optimal time, to produce composites with exceptional mechanical performance and competitive properties to those of synthetic fibers, but have the advantage of being manufactured from renewable and low cost raw materials.

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