STUDIUL PROPRIETĂŢILOR MECANICE ALE UNUI MATERIAL COMPOZIT: POLIESTER NESATURAT/FIBRE ALFA
STUDY OF THE MECHANICAL PROPERTIES OF A COMPOSITE MATERIAL: ALFA FIBERS/ UNSATURATED POLYESTER

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Composite materials based on woven Alfa and unsaturated polyester resins were manufactured by hand layup technique. The Alfa fibers were collected from the M'Sila region (Algeria). Different chemical treatments such as mercerization, permanganate, acetylating and dichromate have been used in order to modulate the mechanical properties of these composites. According to tensile and flexural tests, the treated composites showed better mechanical performances compared to their untreated counterparts. Actually, the permanganate treated fiber-reinforced composites showed 43.02 % of increase in tensile strength and 31.59 % in Young's modulus, while the acetylated fiber-reinforced composites showed maximum improvement in flexural strength of about 23.80 %. Furthermore, the flexural modulus was also improved by 35.7 % compared to the untreated Alfa fiber composites. FTIR characterization of Alfa fibers showed that the quantity of hydroxyl groups (-OH) decreased and the crystallinity index increase by 20.53% after alkali treatment. SEM observations on fibers surface showed that the different chemical treatments modified the fiber which improved the fiber–matrix adhesion.

Keywords: *. Alfa fibers, composites, chemical treatment, tensile test, mechanical properties.*

1. Introduction

The use of plant fibers as reinforcing fillers for both thermoset and thermoplastic composites has attracted an increasing interest over the last few years (composites preparation and paper production)[1,2]. Natural fibers such as flax, hemp, bamboo and sisal are low-cost, environmentally friendly, biodegradable, nontoxic, and have low density as well as good thermal and acoustic insulating properties. Those advantages make them a sustainable and reliable alternative to synthetic fibers (e.g., carbon and glass fibers) in many industrial areas such as plastics, packaging, automotive, and building materials. However, the main drawback of the natural fibers is their hydrophilic nature, which makes them incompatible with the hydrophobic polymeric matrix (poor adhesion), leading to poor mechanical properties of the resulting composites [3]. Besides, natural fibers exhibit poor resistance to moisture, which lead to high water absorption, subsequently resulting in poor durability, mechanical properties and dimensional stability of the natural fiber reinforced composites [4, 5]. Chemical treatments on natural fibers, polymer matrix, or on both materials provide an alternative solution to address these issues [6, 7]. The chemical treatments aim to improve the hydrophobic character of the natural fiber, wettability and surface roughness, which improves the interfacial bonding between matrix and fibers. Chemical treatments can also decrease moisture absorption, leading to an enhancement of the mechanical properties and durability of the natural fiber reinforced composites [8, 9].

The modification of the fibers surface by chemical treatments and their effect on the mechanical properties of natural fiber reinforced polymer composites has been extensively investigated over the last decade [10]. Actually, Joseph et al. [11,12] observed that permanganate treated sisal fibers reinforced low density polyethylene (LDPE) composites, showing an enhancement in tensile properties thanks to the permanganate induced grafting. Researchers [13-15] worked on jute fibers/polyolefin composites and reported improved properties of the composites.

Different chemical modifications including permanganate treatment on natural fibers for reinforcing composites were reviewed by Li et al[6] and they reported that mechanical properties of the composites were improved significantly. Most

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permanganate treatments are conducted by using potassium permanganate ($KMnO_4$) solution (in acetone) in different concentrations with soaking duration from 1 to 3 min after alkaline pre-treatment [11,16-19]. As a result of permanganate treatment, the hydrophilic tendency of the fibers was reduced, and thus the water absorption of fiber-reinforced composite decreased. The hydrophilic tendency of fiber decreased as the $KMnO_4$ concentrations increased. But at higher $KMnO_4$ concentrations of 1%, degradation of cellulosic fiber occurred which resulted in the formation of polar groups between fiber and matrix.

Oxidation of cellulose with potassium dichromate-sulfuric acid, potassium dichromate-oxalic acid, potassium permanganate, sodium hypochlorite and periodate were performed by different researchers [20-22]. Hill and Abdul Khalil investigated the effect of chemical modifications such as acetylation, silane treatment and titanate coupling on the mechanical properties of coir and oil palm fiber-reinforced polyester composites [23]. A small increase in tensile strength, tensile modulus, and impact strength of composites reinforced with modified fibers was noted. Brahim and Cheikh extracted Alfa fibers with soda and studied the influence of fibers orientation of their unidirectional in the composite with unsaturated polyester matrix [24]. Bledzki et al[25] studied different concentrations of acetyl treatment on flax fibre and reported 50% higher thermal properties.

In this work, we focused on the effect of various chemical treatments as a surface modifier on the mechanical properties of the woven Alfa reinforced plastic composites. The Alfa (StipaTenacissima L.) covers an area of 2 million hectares of the Algerian semi-arid region, which make it readily available, very low-cost, and potentially key for the sustainable development in Algeria and other countries [26]. After a pre-treatment with 2.5% wt NaOH, woven Alfa fibers were subjected to various chemical treatments namely: 0.2% wt permanganate, 0.2% w.t dichromate and acetic anhydride. The effect of these treatments on the fibers properties (surface modification) was explored. The influence on the mechanical properties was investigated to determine the best treatment. Furthermore, interface studies were made with SEM to ascertain the effects of the surface treatment of the fibers.

2. Materials and methods

2.1. Raw materials

Alfa (*StipaTenacissima L.*) is a tussock grass constituted of cylindrical stems with a height up to 1mand is rich with fibers [27]. The structure of Alfa fibers consist of three main constituents, namely; cellulose, hemicellulose and lignin. These components of the fibers contribute to determining the mechanical properties of the fibers. According to our previous work[28], the chemical composition of Alfa grass is: 40.81% cellulose, 30.8% hemicellulose, 22% lignin, 1.8% wax, and 1.7% of ash. Alfa fiber bundles are characterized by a mean diameter of 113 mm (ranging from 90 to 120 mm) and a density of =0.89 $g.cm^{-3}$[29].

The Alfa fibers used in this work were collected from a semi-arid region M'Sila, Algeria on May 2014. The Alfa fibers were first scoured with hot detergent solution 2% to remove the contaminants and adhering dirt and washed with distilled water. Thereafter, they were air dried for 72h at room temperature, and then Alfa stems were placed in packets, preserved in polyethylene bags and stored away from light for 60 days at ambient temperature. Alfa stems were cut into 35 cm length pieces [26]. During the next stage, the fibers were sieved to remove volatile compounds, and finally carded to make them soft and separated. The Alfa fibers were then washed several times with acetic acid. Then these fibers were washed again with distilled water and dried in an oven at 80°C for 5h. The resulting fibers were denoted as untreated Alfa fibers

2.2. Preparation of the woven Alfa fibers

Woven Alfa fabric used as reinforcement was obtained from sud region of M'sila and received in form of plain weaves [30] as shown in Figure 1. The fabric presents 5 mm of thickness.

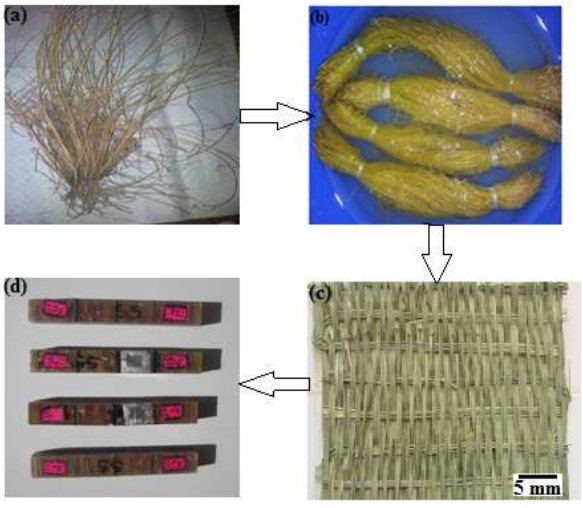

Fig.1 - Plain weaves Alfa fabric.

2.2.1. Chemical treatments

Alfa fibers were subjected to the following surface treatments in order to improve interfacial adhesion between Alfa fibers and unsaturated polyester matrix.

2.2.2. Alkali Treatment

The woven fibers were mercerized in a 2.5 % wt. sodium hydroxide (NaOH) solution at room temperature for 24h.The fiber/ solution

ratio used was 1:20 (w/v), washed with water to remove any traces of alkali from the fiber surface and neutralized with 1 % acetic acid solution and then dried in oven at 80 °C for 48h. The above concentration of sodium hydroxide has been frequently used on leaf-extracted plant fibers such as sisal in order to provide a fast and effective treatment in removing non-structural matter without causing degradation of fiber cellulose [31,32].

2.2.3. Permanganate treatment

For the permanganate treatment, alkaline treated woven fibers were dipped in 0.02 % w/v potassium permanganate acetone solution for 5 min (the fiber/ solution ratio used was 1:20 (w/v)). Fibers were dried at 80 °C for 48h to remove excessive solvent and moisture in hot air oven.

2.2.4. Dichromate treatment

Woven fibers were pre-treated with alkali and then dipped in potassium dichromate solution in acetone for about 5 minutes (the fiber/ solution ratio used was 1:20 (w/v)). The dichromate solution of concentration 0.02 % w/v was used. Woven fibers were washed in distilled water and finally dried in oven at 80°C for 48h.

2.2.5. Acetylating treatment

The alkali pre-treated woven fibers were subjected to acetylation with acetic anhydride in acetic acid medium (Concentrated H_2SO_4 acts as a catalyst in this reaction). Fibers were washed in distilled water and then dried in oven at 80°C for 48h. The coding of the different sets of fibers is reported in Table 1.

Table 1

Coding of the different fiber categories studied

Material code	Chemical treatment
UAF	Untreated
ATAF	NaOH
PTAF	$KMnO_4$
DTAF	$K_2Cr_2O_7$
AATAF	$CH_3COOCOCH_3$

2.3. Preparation of composites

Composites were made using a mould with dimensions 300x300x50 mm^3. The resin selected for this study was an isophthalic polyester resin, which was cured using 1% of methyl ethyl ketone peroxide as catalyst and 0.5% of cobalt naphthenate accelerator. The manufacturing of the composite structures consisted in soaking one plat of woven fibers with polyester resin in the mould (air bubbles were removed carefully with roller). Five plates of composites were manufactured (Table 2) using hand lay-up technique [24]. The post curing of the samples was done at 50 °C for 24 h. The filler content was fixed at 40% for all samples. After curing the composite, the specimens were cut for test according to the ASTM standards.

3. Chemical and physical characterizations of the fibers

FTIR spectra of untreated and treated fibers are obtained by mixing the samples with KBr. Conventional wafers have been prepared using a laboratory hydraulic press. The IR absorption study (4000–400 cm^{-1}) was conducted using a FTIR spectrometer (Shimadzu, series 8300, Japan).

X-ray diffraction (XRD) experiments were performed by using a PANanlytical X'Pert (Philips) PRO PW3209 powder X-ray diffractometer, operating at 40 kV and 20 mA in the Bragg–Brentano geometry and using Cu Kα radiation (Kα = 0.154059 nm). Crystallinity Index (CrI) was used to evaluate the physical structures changes (estimating the degree of crystallinity) of the fibers according to the empirical method developed by Segal et al [33]. According to this formula, the Crystallinity Index can be written as:

$$\mathrm{CrI}\ \% = \frac{I_{002} - I_{am}}{I_{002}} X100 \qquad (1)$$

Where I_{002} is the intensity of the principal cellulose I peak at around 2θ angle (22.7°); I_{am} is the intensity attributed to amorphous cellulose given at around 2θ angle 18°.

The treated and untreated Alfa fibers morphology were observed by scanning electron microscopy (SEM) using a Cambridge S200 Scanning Electron Microscope (UK).

Table 2

Composition of the composites manufactured in this study

Composites	Designation	Fibers loading % (w/w)	Fibers loading % (v/v)
Untreated tissue Alfa Fibers / Unsaturated Polyester	UTAF/UP	11.66	
Alkali treated tissue Alfa Fibers / unsaturated Polyester	ATAF/UP	10.56	
Permanganate treated tissue Alfa Fibers / unsaturated Polyester	PTAF/UP	11.26	40
Dichromate treated tissue Alfa Fibers / unsaturated Polyester	DTAF/UP	10.5	
Acetic anhydride treated tissue Alfa Fibers / unsaturated Polyester	AATAF/UP	9.46	

3.1. Mechanical Tests

Tensile and flexural tests of the composite specimens were carried out using a Universal Testing Machine (Zwick Z50, Zwick/Roell, Germany) at a cross head speed of 1mm/min. Rectangular specimens of size (250x22x21mm^3) were used for testing. For the flexural test, specimens were submitted to the three-point bending test according to ASTMD790 standard. The load displacement curves were obtained after evaluating the flexural strength and Young's modulus of the composites using the following equations:

Flexural strength :

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

Flexural modulus :

$$E_f = \frac{L^3 m}{4bd^2} \quad (3)$$

Where *L* (mm) is the support span; *b* (mm) is the width of the specimen; *d* (mm) is the thickness; *P* (N) is the maximum load; and m is the slope of the initial straight line portion of the load–displacement curve. The tensile testing of the behavior of Alfa fibers composite samples was carried out according to the ASTM D3039 standard.

4. Results and discussion

4.1. Infrared spectroscopy analysis

The FTIR spectra of untreated and treated fibers are shown in Figure 2. We can see that the spectra are similar to each other. The broad absorption band observed in the 3422 cm^{-1} is related to the hydrogen bonding (OH) stretching vibration. This figure shows a decrease of the band corresponding to hydroxyl group located at around 3422 cm^{-1}. This can be explained by the fact that the sodium hydroxide treatment dissolved hemicelluloses [34], certain extractable constituents such as pectins [35], and some amount of lignin. The first fact is reinforced by the decline that occurred in the region of the carbonyls at around 1737 cm^{-1} related to the stretching of the C=O group showing a probable dissolution of the hemicelluloses [36]. It can also be due to the dissolution of pectin and lignin in Alfa [37]. This band disappears for alkali and acetylation treated samples. This means that the hemicelluloses were removed from the fiber surface. For the acetylated fibers, this band is shifted to 1726 cm^{-1} indicating that some amount of hemicelluloses was removed [36]. The second one reveals that the alkaline treatment breaks the bond between carbohydrates and lignin, the latter is therefore solubilized, which is supported by the decrease of the band at around 2937 cm^{-1} associated with the stretching of the CH group of the methoxyl groups of aromatic ring [38], and the band characteristic of lignin at about 1650 cm^{-1}, It appears weaker in intensity in the spectrum after the alkali treatment. The vibration band at 2916 cm^{-1}, corresponding to the stretching of the C–H aliphatic group, is included in almost all natural fibers. This band decreased due to the removal of hemicelluloses [39]. The band at 1250 cm^{-1} for untreated fiber is corresponded to the C=O stretching in the aromatic ring of lignin [40]. For alkali, permanganate, dichromate and acetylation treated fibers this band was decreased, from these results; it proves that lignin attached on the surface of fibers was removed by the designated chemical treatments [41]. The spectrum of acetylated fiber can be easily distinguished from that of untreated by the increased intensity of the C=O (carbonyl) peak at 1741 cm^{-1} and the significant reduction in the hydroxyl signal [42].The peaks observed between 1100-1600 cm^{-1} shows the presence of hemicelluloses in untreated Alfa fibers and the reduced intensity of these peaks in alkali treated, permanganate treated and anhydride acid treated Alfa fibers indicates the slight removal of hemicelluloses from the fiber surface [43]. The reduced of peak due to the presence of alcohol group of cellulose at 1377 cm^{-1} in all chemically treated Alfa fibers confirmed the chemical modification [44-47].

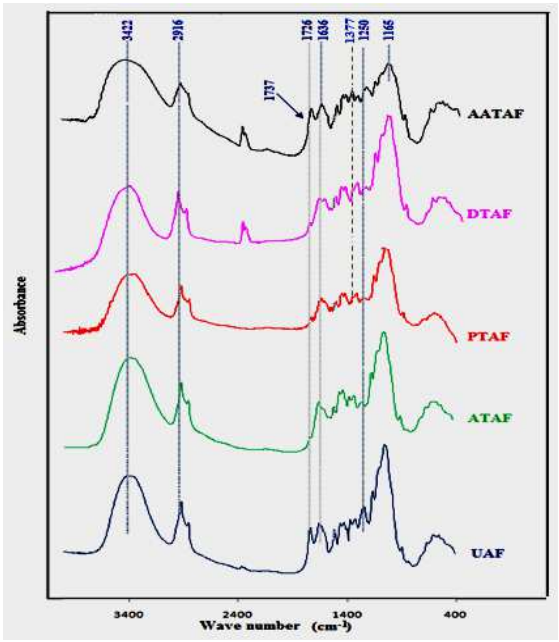

Fig. 2 - FTIR Spectra of the treated and untreated Alfa fibers.

4.2. Scanning Electron Microscopy (SEM)

Figure 3 shows SEM micrographs of the surface of Alfa fibers subjected to different chemical treatments. It is shown that the surface of an untreated Alfa fiber is covered with a layer of different components such as: pectin, lignin and impurities (Fig. 3a). After NaOH treatment (Fig. 3b), most of the lignin and pectin are removed resulting in a rougher surface. Other authors suggested that this roughness may be due to the removal of hemicelluloses, lignin and cellulosic constituents after treatment [48, 49]. This rougher surface facilitates both mechanical

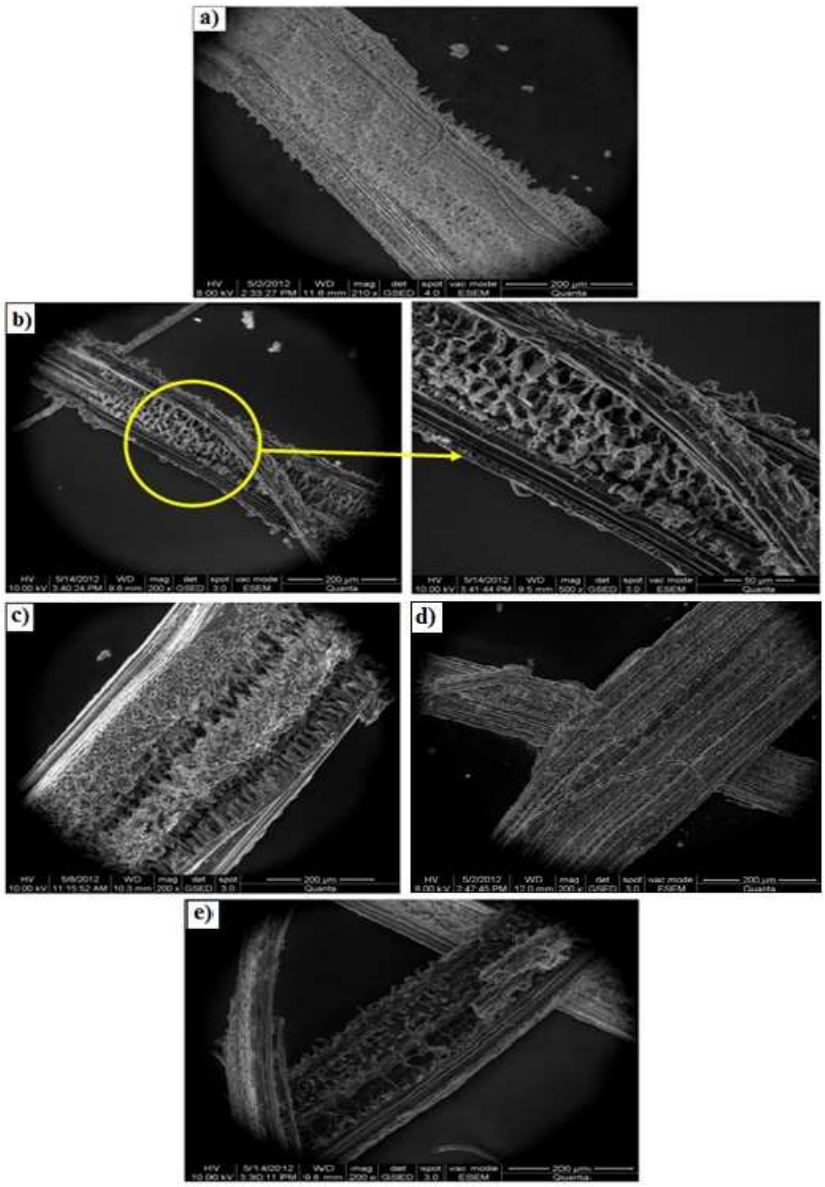

Fig.3- SEM micrographs of: (a) UAF; (b) ATAF; (c) PTAF; (d) DTAF; (e) AATAF.

interlocking and bonding reaction due to the exposure of the hydroxyl groups to the matrix. On other hand, the dissolution of hemicelluloses may increase the interfibrillar region when treated with alkali for longer durations as noticed by Sudhakara et al [50], which increases the available surface area and consequently improves adhesion at the fiber–matrix interface in composites. SEM image of permanganate treated Alfa fiber is shown in Figure 3(c). A comparison between the untreated and permanganate treated Alfa fibers reveals topographical changes because of the permanganate ions react with the lignin constituents and carve the fiber surface, leading to rougher Alfa fiber surface. This can reduce hydrophilic nature of the Alfa fiber (improving chemical interlocking at the interface and providing better adhesion with the matrix) [44,51,52]. The SEM image of dichromate treated Alfa fiber is shown in Figure 3d which clearly shows that this treatment provides rough surface, which give better mechanical interlocking with the matrix.

The SEM image of acetylated Alfa fiber is shown in Figure 3e. The acetylation treatment took out certain portions of covering layers of hemicellulose and lignin from the cellulose microfibrils [53]. Hence, this acetylation treatment increases effective fiber surface area, leading to good adhesion with the matrix.

We conclude that the fibers become porous and fibrillated by the acetylation, permanganate, dichromate and sodium hydroxide treatments. Luz et al [54] observed similar results with sugarcane bagasse fiber-reinforced polypropylene composites.

4.3. X-Ray diffraction

The X-Ray diffractograms of the untreated and treated fibers is depicted in Figure 4. The crystallinity index of untreated and treated fibers (NaOH, permanganate, dichromate and

anhydride acetic) were calculated and found to be 49.80, 62.67, 60.69, 58.3, and 58.01% respectively. This can be attributed to the removal of amorphous hemicellulose from the fibers and the rearrangement of the crystalline regions in such a way that the fiber exhibits a more crystalline nature [55]. It was also seen that the crystallinity index of the Alfa fiber increased with anhydride acetic of about 14.15%. The increase in crystallinity is due to the loss of amorphous hemicelluloses [56]. The crystallinity index of fibers treated increases significantly to reach a maximum at a alkali treatment (62.67%), which corresponds to an increasing of the (CrI) by 20.53% (Table 3). This increasing indicated the improvement in the cellulose structure and finally contributed to enhancing the tensile strength of the fiber.

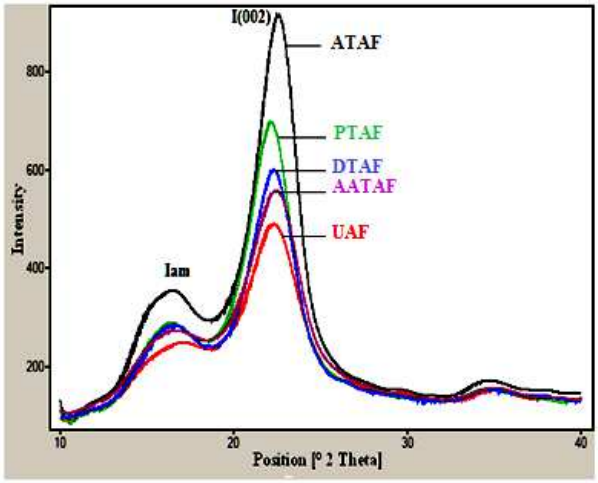

Fig.4 - X-ray diffraction patterns of untreated and treated Alfa fibers.

Table 3

Crystallinity index (CrI) on natural and treated fibers

Treated fibers	Iam	I002	CrI (%)
UAF	130	259	49.80
A TAF	209	560	62.67
PTAF	193	491	60.69
DTAF	211	506	58.3
AATAF	338	805	58.01

4.4. Mechanical tests

4.4.1. Tensile strength (TS) and modulus(TM)

The effect of different chemical treatments on tensile and flexural properties of woven Alfa fiber /unsaturated polyester composites is given in Table 4. It is clearly shown that the different treatments used has a positive impact on the TS and TM, which is higher for the permanganate treated woven Alfa fibers composite (Fig.5). The highest TS value reported was 46.95 MPa. This may be due to highly reactive permanganate ions (Mn^{3+}) with cellulose hydroxyl groups (forming cellulose manganate). Permanganate ions react with the lignin constituents and carve the fiber surface. As a result fiber surface becomes physically rough. This reduces hydrophilic nature of the fiber. Permanganate treatment of natural fibers enhances the chemical interlocking at the interface and provides better adhesion with the matrix [44,51,57]. The tensile modulus also increases with the PTAF/UP specimen and showed better tensile properties has a value of 2.69 GPa, which increased by 31.59 % compared to untreated Alfa fiber composites (Fig.6). This was due to the enhancement in bonding between fiber and matrix. Similar improvement or increase in the tensile properties of alkali treated natural fibers reinforced composites were reported by Lai et al, Rout et al and Ray et al [46,58,59].

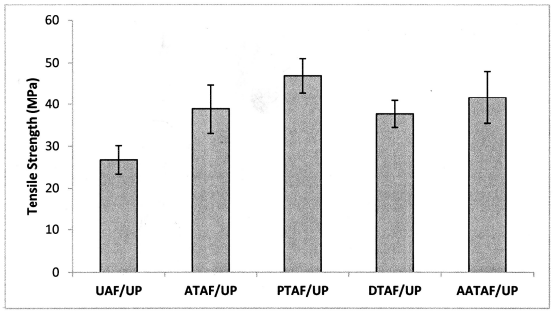

Fig.5 - Effect of fiber modification on tensile strength of woven Alfa fiber–unsaturated polyester composite.

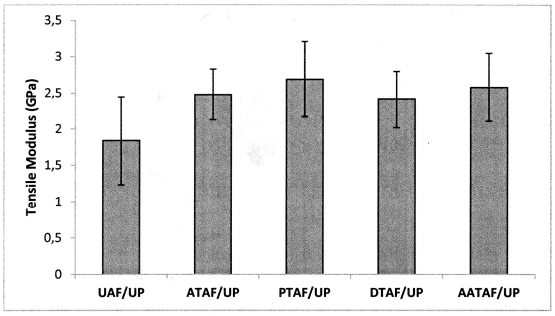

Fig.6- Effect of fiber modification on modulus strength of woven Alfa fiber–unsaturated polyester composite.

4.4.2. Flexural strength (FS) and modulus (FM)

The flexural strength and modulus of untreated and treated composites were shown in Figures 7 and 8. It was clearly seen that the AATAF/UP composite showed the highest improvement in terms of flexural strength (Fig.7); which increased by 23.80 % compared to UAF/UP composite. This addition remarkable in flexural strength may be an indication of better adhesion between the polyester and modified woven Alfa fibers. Flexural modulus on the other hand increased with modified the fibers by the treatments investigated (Fig.8). The flexural le modulus of AATAF/UP composite has a value of 6.19 GPa which increased by 35.7 % compared to UAF/UP composite. This result indicates that, acetyl groups could have effectively esterified

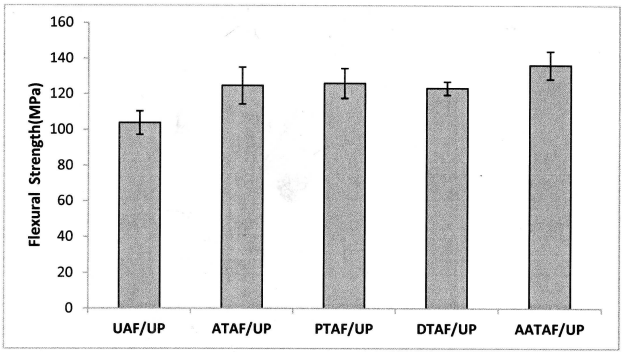

Fig.7- Effect of fiber modification on flexural strength of woven Alfa fiber–unsaturated polyester composite.

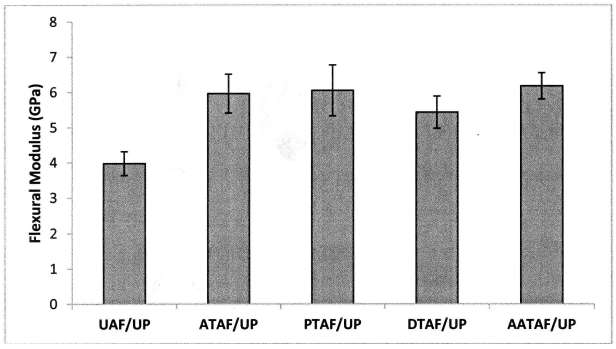

Fig.8 - Effect of fiber modification on flexural modulus of woven Alfa fiber–unsaturated polyester composite.

the hydroxyl groups existing within the pretreated fibers and facilitated strong fiber matrix interface bonding[57]. It may also be due to the incorporation of rigid Alfa filler, which offered a characteristic reduction in the ductility of unsaturated polyester resin matrix and hence resulted in the increase of flexural modulus[60]. We note that there is a significant improvement in mechanical properties of the composites investigated in this work compared to those reported in our previous study [26].

4.5. Digital photo camera

Figure 9 shows optical micrographs of the tensile fracture surfaces of untreated and permanganate treated woven Alfa composite. UAF/UP composites showed a poor interfacial adhesion as a result of low affinity between the polymer matrix and woven Alfa fibers (see Fig.9a). The poor adhesion appears to facilitate debonding of the fibers (The interfacial structure of UAF/UP composites was unable to transfer stress effectively). The composite with permanganate-treated fibers present more homogeneous surfaces and fewer cavities and voids, and consequently have better tensile strength compared with the unmodified composites. This finding suggests that fiber treatment has a positive effect on interfacial adhesion between fibers and matrix (see Fig. 9b).

In summary, we report significant improvements in the mechanical properties of composites containing modified woven Alfa fibers. Permanganate proved an effective adhesion promoter for Alfa fibers/ UP composites, a finding evidenced by SEM images of the fracture surfaces of the materials.

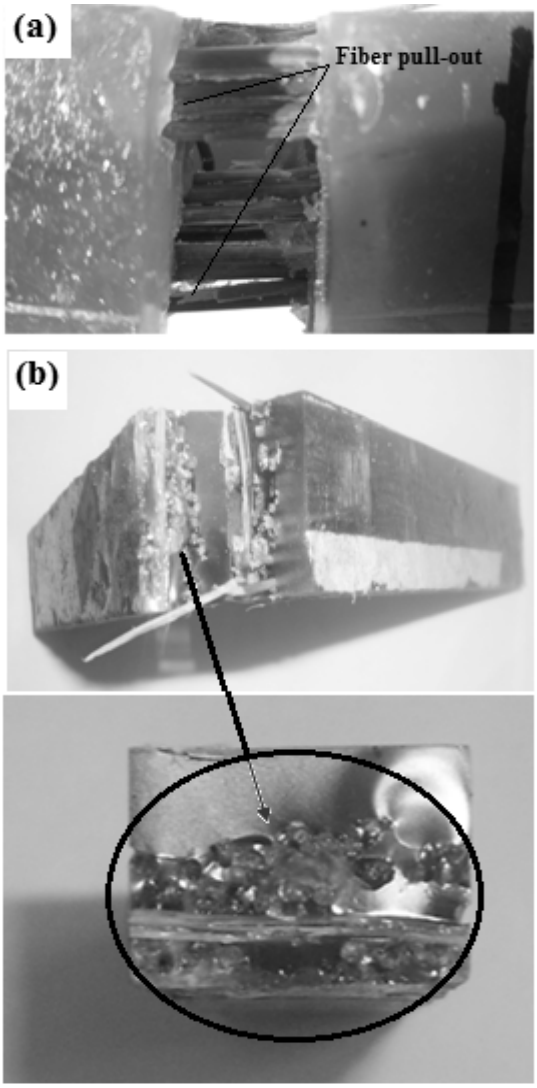

Fig.9 - Optical micrographs of the tensile fracture surfaces of (a) untreated woven Alfa fiber composite and (b) permanganate treated woven Alfa fiber composite.

5. Conclusion

The influences of chemical treatments of the Alfa fibers on mechanical properties of the woven Alfa/unsaturated polyester composites have been experimentally evaluated and the following conclusions are drawn:

(i) The performance of the woven Alfa/composites is very influenced by the surface properties of the fibers.

ii) Alkali treatments, permanganate, dichromate and acetic anhydride have improved the mechanical properties, i.e. tensile and flexural properties of the composites in comparison to the untreated ones thanks to interfacial bonding improvement;

(iii) The value tensile strength and modulus of permanganate treated composite were respectively of about 43.02% and 31.59 %, which were due to high enhancement in the interfacial adhesion of the fiber with the polyester resin. (thanks to an enhancement in the interfacial adhesion of the fiber with the polyester resin);

(iiii) The flexural properties of their composites were increased after these different treatments and showed maximum improvement in flexural strength of about 23.80 % concerning the acetylated fiber-reinforced composites (thanks to an enhancement in the interfacial adhesion of the fiber with the polyester resin).

(iiiii) SEM analysis revealed that different treatments modified the fiber morphology. Alfa fiber can be obtained at a relatively low cost compared to glass fiber reinforcements.

Finally, these fibers have a very promising future in composite material.

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