CELLULOSE CHEMISTRY AND TECHNOLOGY

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

MELOUKI AZZEDINE,[*] BENYAHIA AZZEDINE,[**] DEGHFEL NADIR,[*] FARSI CHOUK,[***]
LAIB NOURI,[*] LEBID MAHMOUD[*] and ADDOUR YAKOUT[**]

[*]*Inorganics Materials Laboratory, University of M'sila, Algeria*
[**]*City, Environment, Society and Sustainable Development Laboratory, University of M'sila, Algeria*
[***]*Laboratory of Structural and Mechanical Materials (LMMS), M'sila, Algeria*
✉ *Corresponding author: B. Azzedine, azzedine.benyahia@univ-msila.dz*

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Researchers are showing increasing interest in plant fiber reinforced composites due to their eco-friendliness, low density, low cost and amazing mechanical properties. However, some challenges remain for researchers in this field, most notably poor adhesion between the polymer matrix and the plant fibers, which reduces the mechanical properties of composites reinforced with these fibers. This study aims to improve the adhesion between the matrix and the reinforcement by chemically treating Alfa fibers (*Stipa tenacissima*) with a 3 wt% NaOH solution at different times (1, 3, 5 and 24 h). FTIR, DRX, GTA and tensile tests were conducted. XRD tests showed that the crystallinity index of 3% alkali treated Alfa fibers for 5 h increased by 36.26%, compared to that of untreated fibers. The results also revealed that the mechanical properties of composites reinforced with treated fibers outperformed those reinforced with untreated fibers. These findings can contribute to the development of high mechanical performance composites, which can be competitive with those prepared with synthetic fibers.

Keywords: composite, *Stipa tenacissima*, adhesion, tensile strength, alkaline treatment

INTRODUCTION

Plant fibers are used in various light structural applications, such as automotive panels, thermal insulation, aerospace, packaging industry, windows and doors.[1-3] Natural fibers have attracted the attention of researchers due to their remarkable properties: easy availability, low density, biodegradability, renewability, cost-effectiveness and environmental friendliness. In addition, the processing of natural fibers does not pollute the environment or negatively impact workers and end users.[4-7] Natural fibers, when used as reinforcement in composite materials, are an attractive alternative to glass fibers due to their ease of processing and competitive mechanical and dynamic properties.[8-10]

The main issue that researchers face is poor interfacial adhesion between the polymer matrix and the fibers because the fibers are hydrophilic, while the polymer matrix is hydrophobic, resulting in poor adhesion.[11,12] To create better and more durable composites, limitations such as fiber hydrophobicity and wettability must be overcome.[13] Surface modification techniques, such as NaOH treatment, silane, potassium permanganate, benzoyl chloride, plasma treatment, laser treatment, corona treatment and others, have been reported in various studies as potential modifications to the surface of the fiber to improve its properties.[14–16]

The effect of alkali treatment on fiber-reinforced unsaturated polyester composites was investigated by Benyahia *et al.*[17] To improve the mechanical properties of the studied composite, the authors treated the Alfa fibers with different concentrations of NaOH (1, 3, 5, and 7%) for 24 hours (resistance to bending). The results show that the flexural strength is significantly improved, particularly in the composite reinforced with Alfa fibers treated with NaOH (7%). Raharjo *et al.*[18] used an alkaline treatment to improve the fiber-matrix interface of cantala fiber. The alkaline treatment was carried out for 0, 4, 8, 12, 16, 20, and 24 hours using a 2% NaOH solution. The composites' tensile properties were examined to determine the effect of alkaline treatment. The results show that adding alkali-treated fibers to recycled high-density polyethylene composites improved the composites' tensile performance by 15.5%.

Mouissa *et al.*[19] chemically treated wood sawdust with NaOH (5 wt%) and $KMnO_4$ (0.01 wt%) solutions. Mechanical tests on a composite reinforced with alkali-treated sawdust revealed increased flexural and compressive strength, with maximum values of 0.89 and 4.85 MPa, respectively. Borchani *et al.*[20] used a NaOH solution at two different concentrations (1 and 5% by weight) to treat Alfa fibers in a similar study. The authors reported that the treated Alfa fibers' crystallinity index and thermal stability improved. Boukoulda *et al.*[21] also used different concentrations of sodium hydroxide on Alfa fibers (9, 10, 11 and 12%). The composite reinforced with 9% NaOH-treated fiber had the highest tensile strength, according to their results. Adour *et al.*[22] prepared composites of an unsaturated polyester resin matrix, reinforced with treated and untreated Alfa fibers. Fiber treatment was performed by 5% NaOH solution at different times (1, 3, 5 and 24 hours). The analytical results showed that the curing time of the fibers had a positive effect on the mechanical properties of the composites. In this work, we prepared composite materials reinforced with Alfa fibers that have been treated with a 3% soda solution at different times (1, 3, 5 and 24 hours), with the objective of determining the extent of the effect of the treatment time on the mechanical properties of the composites studied (tensile strength).

EXPERIMENTAL

Raw material

Alfa fibers were harvested in the Hodna region (M'sila-Algeria) in August 2022. The fibers were rinsed with tap water to remove dust and pollutants, then air-dried for 3 days at room temperature.

Alkaline treatment of Alfa fibers

The treatment consisted of 98% pure sodium hydroxide (NaOH), acetic acid and analytical grade glycerol (99.5% purity). Chopped Alfa fibers were treated in a 3 wt% sodium hydroxide solution for different periods (1, 3, 5 and 24 hours) at a solution ratio of 15:1 (w/v). After treatment, the fibers were soaked in an acidified solution to remove excess soda, rinsed with water to pH 7, and dried in an oven at 105 °C for 6 h. The fibers used in this preparation were 1 cm in size.[22]

Composite preparation

The matrix used for this study was an unsaturated orthophthalic polyester cured with 2% methyl ethyl ketone peroxide. Table 1 summarizes the characteristics of the unsaturated polyester matrix. The specimens were prepared using a wooden mold of dimensions 200×20×4 mm^3. The fiber content of the composite was set at 30% (v/v). The composite was prepared by the lay-up method. The fibers were introduced into the matrix, and the mixture was poured into the mold to obtain the samples. Table 2 lists the codes of the samples studied.

Fourier transform infrared spectroscopy (FTIR)

The spectra of the samples were recorded by an FTIR Affinity-1S SHIMADZU (Japan), installed at the Laboratory of Chemistry of the University of Setif, Algeria. The samples were prepared in tablet form: 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)

The treated and untreated Alfa fibers' crystallinity index (CrI) values were determined on a D8 diffractometer (Bruker-AXS, Germany) with copper radiation. The Cu Kα (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.*:[23]

$$CrI\% = \frac{I_{002} - I_{am}}{I_{002}} \quad (1)$$

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°.

Thermogravimetric analysis

Thermal properties were investigated using thermogravimetric analysis (TGA) to track the evolution of thermal stability in fibers. The measurements were made in a nitrogen atmosphere using thermal analysis equipment (SDT Q600 TA, USA) from 20 °C to 800 °C at a heating rate of 10 °C/min.

Scanning electron microscopy (SEM)

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

Mechanical testing

Tensile tests were conducted on the composite specimens using a Universal Testing Machine (Zwick Z50, Zwick-Roell, Germany), with a crosshead speed of 1 mm.min^{-1}. Rectangular-sized specimens (250 x 22 x 21 mm^3) were used for testing. The tensile testing of the behavior of Alfa fibers composite samples was carried out according to the ASTM D3039 standard.

Table 1
Physical and mechanical properties of unsaturated polyester[22]

Characteristics	
Viscosity at 25 °C	1000-1800Cps
Styrene content	39-41%
Acid value max	30 KOH/g
Density	1.09-1.11 Kg/dm^3
Gel time at 25 °C	20-40 min
Curing time	50-70 min
Exothermic peak	100-120 min
Tensile strength	40-60 MPa
Bending strength	80-100 MPa

Table 2
Coding of different composite samples prepared

Code	Formulation
UTAF/UP	Untreated Alfa fiber/unsaturated polyester
ATAF31/UP	Alfa fiber alkaline treated with 3% for 1 h/unsaturated polyester
ATAF33/UP	Alfa fiber alkaline treated with 3% for 3 h/unsaturated polyester
ATAF35/UP	Alfa fiber alkaline treated with 3% for 5 h/unsaturated polyester
ATAF324/UP	Alfa fiber alkaline treated with 3% for 24 h/unsaturated polyester

RESULTS AND DISCUSSION

FT-IR analysis

The spectra recorded from the FT-IR analysis of the untreated and treated Alfa fibers are shown in Figure 1, demonstrating that nearly identical patterns were obtained, with minor differences in the case of the treated sample. The peak response found at specific wavelengths between 400 and 4000 cm^{-1} determined the presence of cellulose, hemicelluloses, and lignin components in the fiber structure. The hydroxyl group (OH) bound to cellulose and hemicelluloses is responsible for the broadband absorption at around 3390 cm^{-1}.[11] A prominent absorption band (between 2840 and 2945 cm^{-1}) for treated and untreated Alfa fibers, respectively, may be related to the alkyl C-H stretching vibration (both symmetrical and asymmetrical) in natural fiber cellulose and hemicellulose components.[24] Another peek at 1735 cm^{-1} is associated with carbonyl groups C=O due to carbonyl (C=O) stretching vibrations of carboxyl and acetyl groups in hemicelluloses.[25]

Furthermore, Figure 1 shows that the peak of the samples ATAF31 and ATAF24 is lower when compared to that of untreated Alfa fibers. This finding may be due to the partial hydrolysis of cellulosic transcripts in alkaline media. The absorption bands at 1643 and 1242 cm^{-1} indicated that the acetyl groups in the lignin components were C=C and C-O stretched, respectively.[26] The intensity of two peaks (1257 cm^{-1} and 1735 cm^{-1}) on the spectra of treated Alfa fibers (ATAF31, ATAF33, ATAF324) decreased when compared to untreated Alfa fibers. This is due to the removal of some lignin and hemicelluloses. The peak at 1030 cm^{-1} is associated with hemicellulose C-O bond vibration.[27] In addition, it can be noted that removing hemicellulose components reduced the two peaks at 3390 cm^{-1} and 1030 cm^{-1}.[8] As a result of the alkaline treatment and processing time of Alfa fibers, some fiber components, such as lignin and hemicelluloses, dissolved.

X-ray diffraction

Figure 2 shows the diffraction spectra of treated and untreated Alfa fibers. The crystallinity index values of the fibers (Table 1) improved after the treatment. The sample's highest crystallinity index is 43.46% (ATAF35), with an increase of 36.26 % compared to that of the untreated fibers. These results also show that the treatment time has a positive effect, increasing the crystallinity index (CrI), because, with a treatment time of 5 h, the crystallinity index reaches a maximum value. Pickering *et al.*[28] also reported that alkaline treatment resulted in a higher crystallinity index and better thermal stability for harakeke and hemp fibers. This increase may be attributed to the removal of amorphous hemicelluloses from the fibers, or there is a possibility of rearrangement of the crystalline regions, so that the fiber has a more crystalline nature.[29,30] On the other hand, beyond 5 h, the CrI decreases. Thus, with a longer treatment time, the alkaline treatment can damage the fibers and lead to a decrease in the crystallinity index.[31,32]

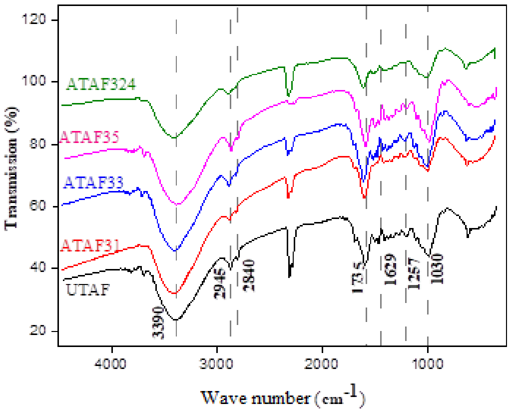

Figure 1: Infrared spectra of untreated Alfa fibers and those treated for 1, 3, 5 and 24 h

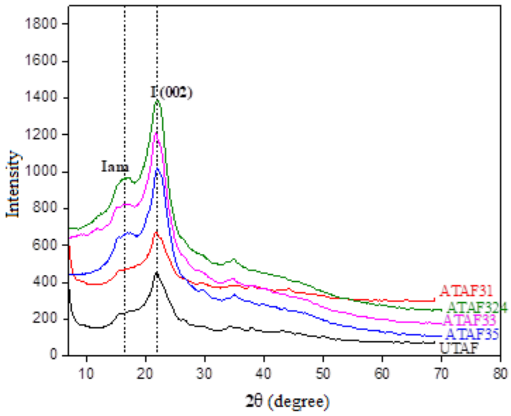

Figure 2: X-ray diffraction patterns of untreated Alfa fibers and those treated for 1, 3, 5 and 24 h

Table 3
Crystallinity index of untreated Alfa fibers and those treated at different times (1, 3, 5 and 24 h)

Material	$I_{(002)}$	I_{am}	CrI%
UTAF	430	312	27.7
ATAF31	1145	752	34.3
ATAF33	420	268	36.19
ATAF35	1026	580	43.46
ATAF324	1259	749	40.5

Thermogravimetric analysis (TGA)

Thermogravimetric analyses were performed to compare the effects of the treatments on the thermal stability of the fibers. Figure 3 depicts the thermal stability curves of untreated Alfa fibers and those treated with NaOH (3%) at various times (1, 3, 5, 24 h). It depicts three areas of mass loss. The treated and untreated Alfa fibers lost a small amount of weight in the first region between 25 and 100 °C, corresponding to water molecule evaporation. Several authors[33,34] have made the same observation. Untreated Alfa fibers begin to decompose around 205 °C in the second region, between 200-290 °C, while treated Alfa fibers begin to decompose between 220 °C and 230 °C, respectively. Hemicellulose decomposition may cause fiber decomposition.[6,31,35] Decomposition in the third region, 290-400 °C, is related to cellulose and lignin degradation.[35]

Izani *et al.*[36] demonstrated that temperatures ranging from 200 °C to 400 °C correspond to the decomposition of cellulose and hemicelluloses. Meanwhile, lignin decomposition begins above 400 °C, it is the most difficult component to degrade; the mass stabilizes at 700 °C, or 30% for treated fiber; however, the loss remains stable above 550 °C or 37% for untreated fiber. As a result, the treated fiber's lost mass is less than that of the untreated fiber, as shown in Figure 3. As a result, we can conclude that fiber treatments enhanced thermal stability. We highlight a shift toward higher temperatures in the samples ATATF33 and ATAF 324. Mohanty *et al.*[36] made the same observation. These results show that Alfa fibers treated with NaOH improved their thermal behavior over time. As a result, as suggested by FTIR measurements and chemical analysis,[37,38] the evolution of the degradation temperatures of the treated Alfa fibers can be attributed to the efficient removal of hemicelluloses.

SEM analysis of Alfa fibers

Figure 4 presents the micrographs of Alfa fibers obtained by SEM. The outer surface and longitudinal section of treated and untreated Alfa stems were examined. Alfa rods, like all natural fibers, are similar to a composite material reinforced by several elementary cellulose fibers (fibrils) bound together lengthwise, lignin, and other non-cellulosic materials; the whole is regularly covered with waxes and impurities (Fig. 4a).[34,39] Longitudinal section observations (Fig. 4b) show that inside the stems there are many small elements placed regularly and surrounding the bundles of fibers. These elements represent the comma-shaped cells that are still abundant in Alfa stems. A comparison of different micrographs of treated and untreated Alfa stems reveals morphological changes in the Alfa stem after chemical treatment with soda (Fig. 4c and 4d). Untreated Alfa rods have a smooth surface, whereas treated rods have exposed fibrils. The surface roughness increases as the alkaline treatment time increases (from 1 h to 24 h). As a result, removing non-cellulosic materials, such as lignin, hemicelluloses, and wax increases the roughness of the treated fiber surface.[40] Surface roughness increases the interfacial adhesion between the polymer matrix and the natural fibers, contributing to the fibers' reinforcing role.[41]

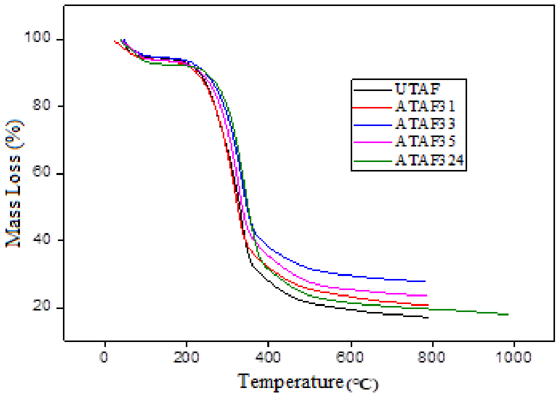

Figure 3: TGA of untreated and NaOH-treated Alfa fibers at different times (1, 3, 5 and 24 h)

Mechanical testing of composites

Tensile strength

Figure 5 shows that alkaline treatment of Alfa fibers at various times improves the tensile strength of the composites. When the Alfa fibers are alkaline treated at different times, the tensile strength of the composites (ATAF31/UP, ATAF33/UP, ATAF35/UP, and ATAF324/UP) improves compared to that of the composites reinforced with untreated fibers (UTAF/UP). This result is attributed to the treated fibers' good adhesion to the polymer matrix. The maximum tensile strength value for composite ATAF35/UP was 15.73 MPa, representing an 11% increase over that of the untreated composite. It is important to note that higher alkali treatment time improves the mechanical efficiency of the composites under consideration. Alkaline treatment reduces fiber size, which has been observed in other natural fibers, such as hemp,[42] flax[43] and coconut,[44] and improves fiber dispersion in the matrix. The alkaline treatment removes impurities and parietal components, such as lignin, pectin, and waxy substances, which coat the outer surface of fiber cell walls. In addition, it results in fiber formation and provides a rough surface for physical and chemical reactions at the filler-polymer interface.[45]

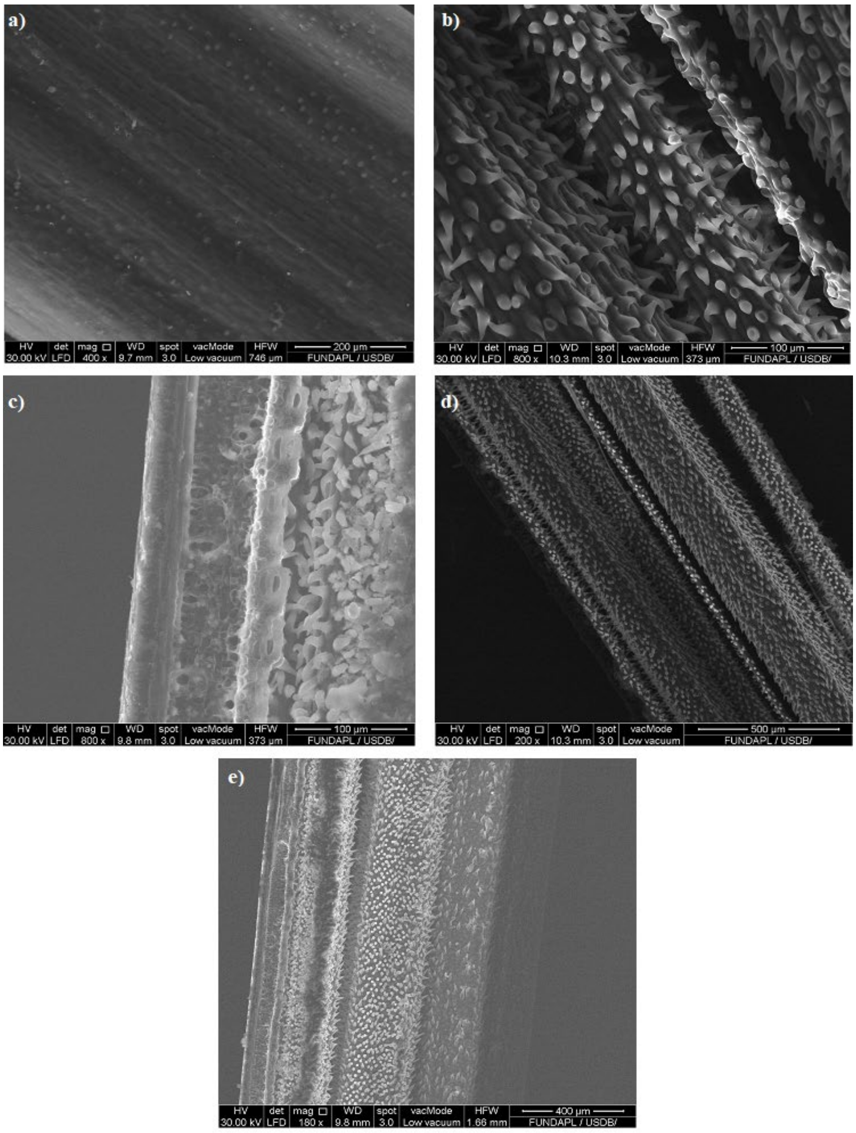

Figure 4: SEM images of Alfa stems: (a) untreated, outer surface, (b) untreated, longitudinal section, (c) 3% NaOH treated for 3 h, longitudinal section, (d) 3% NaOH treated for 5 h, longitudinal section, (e) 3% NaOH treated for 5 h, longitudinal section

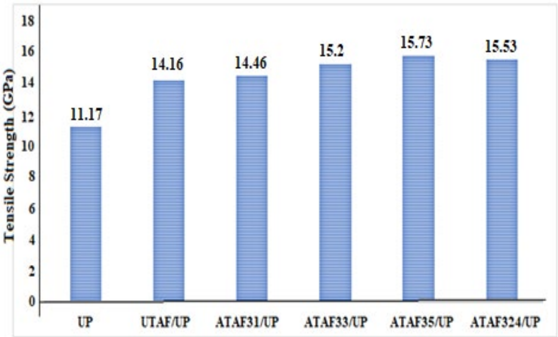

Figure 5: Effect of alkaline treatment time on the tensile strength of the studied composite

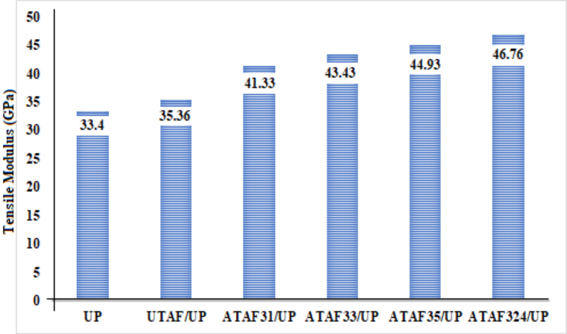

Figure 6: Effect of alkaline treatment time on the tensile modulus of the studied composite

Tensile modulus

The evolution of the tensile modulus of composites reinforced with treated and untreated Alfa fibers is depicted in Figure 6. In general, the composites reinforced with treated Alfa fibers exhibit higher tensile moduli than the composites reinforced with untreated Alfa fibers. It can be noted that higher fiber treatment time increases the stiffness of composites. These findings are consistent with those of Rocha *et al.*[46] and Arrakhiz *et al.*[47] On the other hand, composites reinforced with fibers treated for one hour show an increase in the modulus of elasticity (ATAF324/UP). The maximum tensile modulus was 46.76 GPa, 32% higher than that of the untreated fiber-reinforced composites.

Morphological characterization of composites by scanning electron microscopy

Figure 7 (a-d) shows the micrographs of the fractured surfaces of the composites based on unsaturated polyester reinforced with Alfa fibers, observed under the scanning electron microscope. SEM micrographs of the UTAF/UP, ATAF31/UP, ATAF33/UP, and ATAF35/UP formulations were examined to determine the effect of alkaline treatment of Alfa fibers on the interface of the composites (Fig. 7). Figure 7a and 7b show the presence of microvoids and cavities on the surface of samples UTAF/UP and ATAF31/UP. These microvoids are accentuated by the alkaline treatment effect, due to the loosening of the charge of the UP matrix during the fracture, highlighting the incompatibility of the two phases due to poor interfacial adhesion and the difference in energies (or polarities) of the free surface of the hydrophilic filler and the hydrophobic polymer.[48,49] On the other hand, the micrographs of the fractured surfaces of the treated (Alfa/UP) composites (Fig. 7c and 7d) show good adhesion and compatibility between the Alfa fibers and the UP matrix, with fewer voids and cavities after all of the modifications mentioned.

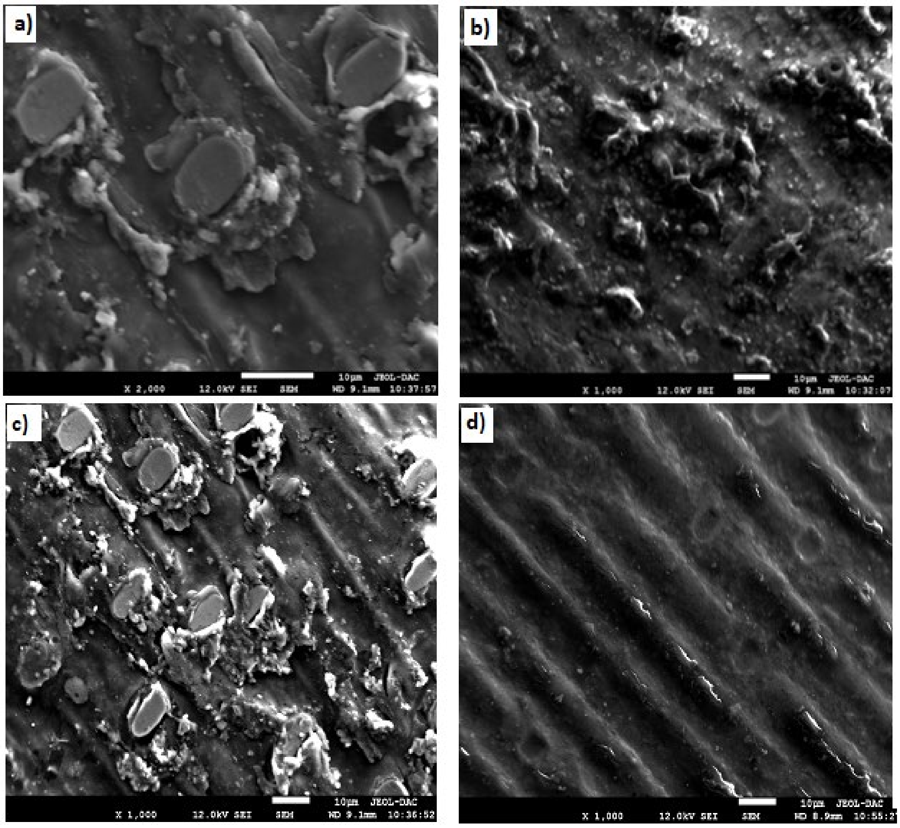

Figure 7: SEM micrographs of untreated and treated composites; (a) UTAF, (b) ATAF31, (c) ATAF33, (d) ATAF35

CONCLUSION

Alfa-reinforced polyester-based composites were successfully manufactured and characterized in this study. The mechanical properties of the composite material were designed to improve with the treatment applied to the fibers before manufacturing the composites. The mechanical testing of the material incorporating Alfa fiber treated with 3 wt% NaOH solution at different times (1, 3, 5, 24 h) revealed higher mechanical strength values – of 15.73 MPa for tensile strength and 46.76 GPa for tensile modulus, respectively. This finding suggests good fiber-matrix adhesion, due to which stress is effectively transferred from the matrix to the fiber. Given the morphological structure of the treated fiber, the longitudinal surface study revealed that the surface became rough after being exposed to high-time alkali treatment.

Furthermore, the alkaline treatment increased the Alfa fibers' crystallinity index. The ATAF33 sample achieved the highest value, with the crystallinity index increasing by 56.89%, compared to that of untreated fibers. The treated ATAF31/UP, ATAF33/UP, ATAF35/UP, and ATAF324/UP composites showed an increasing trend in crystallinity index values, consistent with their relatively good mechanical properties. The removal of hemicelluloses, lignin, and other surface contaminants from the fiber surface was demonstrated by FT-IR analysis of the composites. These findings may broaden the potential applications Alfa fiber reinforced polyester composites as promising materials.

REFERENCES

[1] G. T. Yashas, S. Girijappa, R. Mavinkere, P. Jyotishkumar and S. Suchart, *Front. Mater.*, **6**, 27 (2019), https://doi.org/10.3389/fmats.2019.00226

[2] A. Amrane, Z. Sereir, C. Poilâne and A. Vivet, *Rev. Compos. Mater. Avan.*, **29**, 185 (2019), https://doi.org/10.18280/rcma.290401

[3] R. Boujmal, C. A. Kakou, S. Nekhlaoui, H. Essabir, M. O. Bensalah *et al.*, *J. Thermoplast. Compos. Mater.*, **31**, 974 (2018), https://doi.org/10.1177/0892705717729197

[4] M. R. Sanjay, P. Madhu and M. Jawaid, *J. Clean Prod.*, **172**, 566 (2018), https://doi.org/10.1016/0006-291x(75)90498-2

[5] V. K. Thakur and M. K. Thakur, *Carbohyd. Polym.*, **109**, 102 (2014), https://doi.org/10.1016/j.carbpol 2014.03.039

[6] Z. Kassab, A. Boujemaoui, H. B. Youcef, A. Hajlane, H. Hannache *et al.*, *Cellulose*, **26**, 9567 (2019), https://doi.org/10.1007/s10570-019-02767-5

[7] S. Mouhoubi, M. Bourahli, H. Osmani and S. Abdeslam, *J. Nat. Fiber.*, **14**, 239 (2017), https://doi.org/10.1080/15440478.2016.1193088

[8] F. Arrakhiz, M. Elachaby, R. Bouhfid, S. Vaudreuil, M. Essassi *et al.*, *Mater. Design*, **35**, 318 (2012), https://doi.org/10.1016/j.matdes.2011.09.023

[9] A. Bessadok, S. Marais, F. Gouanvé, L. Colasse, I. Zimmerlin *et al.*, *Compos. Sci. Technol.*, **67**, 685 (2007), https://doi.org/10.1016/j.compscitech.2006.04.013

[10] M. Paiva, I. Ammar, A. R. Campos, R. Cheikh and A. Cunha, *Compos. Sci. Technol.*, **67**, 1132 (2007), https://doi.org/10.1016/J.COMPSCITECH.2006.05.019

[11] D. Hammiche, A. Boukerrou, Y. Grohens, N. Guermazi and F. E. Arrakhiz, *J. Polym. Res.*, **27**, 308 (2020), https://doi.org/10.1007/s10965-020-02284-1

[12] M. A. Sayeed and A. Paharia, *J. Text. Inst.*, **110**, 1588 (2019), https://doi.org/10.1080/00405000.2019.1610998

[13] M. H. Alaaeddin, S. M. Sapuan, M. Y. M. Zuhri, E. S. Zainudin and M. Al-Oqla Faris, *J. Clean Prod.*, **235**, 473 (2019), https://doi.org/10.1016/j.jclepro.2019.06.341

[14] R. Vijay, D. Lenin Singaravelu, A. Vinod, I. D. F. P. Raj and M. R. Sanjay, *J. Nat. Fiber.*, **17**, 1739 (2019), https://doi.org/10.1080/15440478.2019.1598914

[15] R. Vijay, D. Lenin Singaravelu, A. Vinod and M. R. Sanjay, *J. Nat. Fiber.*, **18**, 80 (2019), https://doi.org/10.1080/15440478.2019.1612308

[16] M. R. Sanjay, S. Siengchin and J. A. Parameswaranpillai, *Carbohyd. Polym.*, **207**, 108 (2019), https://doi.org/10.1016/j.carbpol.2018.11.083

[17] A. Benyahia and A. Merrouche, *Polym.-Plast. Technol. Eng.*, **53**, 403 (2014), https://doi.org/10.1080/03602559.2013.844828

[18] W. R. Wijang, K. Bambang and T. Teguh, *Journal of Southwest Jiaotong University*, **55**, 1 (2020), https://doi.org/10.35741/issn.0258-2724.55.3.25

[19] F. Mouissa, A. Benyahia, M. Djehiche, K. Belmokre, N. Deghfel *et al.*, *Mater. Tech.*, **109**, 101 (2021), https://doi.org/10.1051/mattech/2021013

[20] K. E. Borchani, C. Carrot and M. Jaziri, *Cellulose*, **22**, 1577 (2015), https://doi.org/10.1007/s10570-015-0583-5

[21] A. Boukhoulda, F. Boukhoulda, H. Makich, M. Nouari and B. Haddag, *J. Compos. Mater.*, **53**, 3993 (2019), https://doi.org/10.1177/0021998319853025

[22] A. Yakout Addour, A. Benyahia, N. Laib and N. Deghfel, *Cellulose Chem. Technol.*, **56**, 1081 (2022), https://doi.org/10.35812/CelluloseChemTechnol.2022.56.96

[23] L. Segal, J. J. Creely, A. Martin and C. Conrad, *Text. Res. J.*, **29**, (1959), https://doi.org/10.1177/004051755902901003

[24] B. S. Yew, M. Martini, B. M. Saiful and H. W. Fwen, *Polym. Compos.*, **48**, 653 (2019), http://dx.doi.org/10.17576/jsm-2019-4803-19

[25] J. S. N. Raju, M. V. Depoures and P. Kumaran, *Int. J. Biol. Macromol.*, **186**, 886 (2021), https://doi.org/10.1016/j.ijbiomac.2021.07.061

[26] M. M. Kabir, H. Wang, K. T. Lau, F. Cardona and T. Aravinthan, *Compos. Part B Eng.*, **43**, 159 (2012), https://doi.org/10.1016/j.compositesb.2011.06.003

[27] A. Benyahia, A. Merrouche, Z. Rahmouni, M. Rokbi, W. Serge *et al.*, *Mechan. Ind.*, **15**, 69 (2013), https://doi.org/10.1051/MECA/2013082

[28] K. L. Pickering, M. A. Efendy and T. M. Le, *Compos. Part A: Appl. Sci. Manuf.*, **83**, 98 (2016), https://doi.org/10.1016/j.compositesa.2015.08.038

[29] S. Ouajai and R. Shanks, *Polym. Degrad. Stabil.*, **89**, 327 (2005), https://doi.org/10.1016/j.polymdegradstab.2005.01.016

[30] H. Abral, H. Putra, S. Sapuan and M. Ishak, *Polym.-Plast. Technol. Eng.*, **52**, 446 (2013), https://doi.org/10.1080/03602559.2012.748804

[31] S. Ajouguim, K. Abdelouahdi, M. Waqif, M. Stefanidou and L. Saâdi, *Cellulose*, **26**, 1503 (2019), https://doi.org/:10.1007/s10570-018-21

[32] B. Meghlaoui, M. O. Ouali and S. Hocine, *U.P.B. Sci. Bull. B*, **81**, 115 (2019)

[33] I. Rahal, W. Soua and R. B. Younes, *CEIR*-31, 37 (2017)

[34] S. Mishra, A. K. Mohanty, L. T. Drzal, M. Misra and G. Hinrichsen, *Macromol. Mater. Eng.*, **289**, 955 (2004), http://dx.doi.org/10.1002/mame.200400132

[35] K. Kharrati, M. Salhi, J. Sliman and R. Abdeljabar, *Mater. Sci. Appl.*, **13**, 519 (2022), https://doi.org/10.4236/msa.2022.1310032

[36] M. N. Izani, M. T. Paridah, U. M. K. Anwar, M. M. Nor and P. S. H'ng, *Compos. Part B: Eng.*, **45**, 1251 (2013), https://doi.org/10.1016/j.compositesb.2012.07.027

[37] A. Mohanty, M. A. Misra and G. Hinrichsen, *Macromol. Mater. Eng.*, **276**, 1 (2000), https://doi.org/10.1002/(SICI)1439-2054(20000301)276:1%3C1::AID-MAME1%3E3.0.CO;2-W

[38] N. Laib, A. Benyahia, A. Redjem and N. Deghfel, *Cellulose Chem. Technol.*, **55**, 159 (2021), https://doi.org/10.35812/CelluloseChemTechnol.2021.55.17

[39] V. Fiore, T. Scalici and A. Valenza, *Carbohyd. Polym.*, **77**, 106 (2014)

[40] L. Y. Mwaikambo and M. P. Ansell, *J. Appl. Polym. Sci.*, **84**, 2222 (2002), https://doi.org/10.1002/app.10460

[41] R. Narendar and K. P. Dasan, *Compos. Part B: Eng.*, **56**, 770 (2014), https://doi.org/10.1016/j.compositesb.2013.09.028

[42] V. Placet, F. Trivaudey, O. Cisse, V. Gucheret-Retel and M. L. Boubakar, *Compos. Part A: Appl. Sci. Manuf.*, **43**, 275 (2012), https://doi.org/10.1016/j.compositesa.2011.10.019

[43] J. Andersons, E. Sparnins, E. Joffe and L. R. Wallstrom, *Compos. Sci. Technol.*, **65**, 693 (2005), https://doi.org/10.1016/j.compscitech.2004.10.001

[44] R. Chollakup, W. Smitthipong, W. Kongtud and R. Tantatherdtam, *J. Adhes. Sci. Technol.*, **27**, 1290 (2013), https://doi.org/10.1080/01694243.2012.694275

[45] C. Albano, M. Ichazo, J. Gonzalez, M. Delgado and R. Poleo, *Mater. Res. Innov.*, **4**, (2001), https://doi.org/10.1007/s100190000108

[46] N. Islam, R. Rahman, M. Haque and M. Huque, *Compos. Part A: Appl. Sci. Manuf.*, **41**, 192 (2010), https://doi.org/10.1016/j.compositesa.2009.10.006

[47] F. Z. Arrakhiz, M. El-Achaby, A. C. Kakou, S. Vaudreuil, K. Benmoussa *et al.*, *Mater. Design*, **37**, 379 (2012), https://doi.org/10.1016/j.matdes.2012.01.020

[48] J. Rout, S. S. Tripathy, S. K. Nayak, M. Misra and A. K. Mohanty, *J. Appl. Polym. Sci.*, **79**, 1303 (2000), https://doi.org/10.1016/S0266-3538(01)00021

[49] M. A. Norul Izani, M. T. Paridah, U. M. K. Anwar, M. Y. Mohd Nor and P. S. H'Ng, *Compos. Part B: Eng.*, **45**, 1251 (2013), https://doi.org/10.1016/j.compositesb.2012.07.027