

STUDY OF THE MECHANICAL BEHAVIOR OF A COMPOSITE MATERIAL
REINFORCED WITH STIPA TENACISSIMA FIBERS

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ARTICLE INFO	ABSTRACT
Article history: Received: Nov, 18th 2024 Accepted: Jan, 17th 2025	Objectives: This research investigates methods to enhance the adhesion between polymer matrices and plant fiber reinforcements, specifically Alfa fibers (Stipa tenacissima), using a 2 wt % NaOH solution for varying durations (1, 3, 5, and 24 h). Theoretical Framework: Plant fiber-reinforced composites are increasingly favored for their eco-friendliness, low density, cost-effectiveness, and promising mechanical properties. However, challenges persist due to inadequate adhesion between the polymer matrix and plant fibers, which can compromise composite performance. Method: Alfa fibers were chemically treated with a 2 wt % NaOH solution for different durations. A comprehensive suite of tests, including FTIR, DRX, GTA, SEM, and tensile tests, were conducted to evaluate the effects of the treatment. Results and Discussion: XRD analyses revealed a significant increase (36.26%) in the crystallinity index of Alfa fibers treated with a 2 wt % NaOH solution for 5 hours compared to untreated fibers. Moreover, mechanical testing demonstrated that composites reinforced with treated fibers exhibited superior mechanical properties compared to those reinforced with untreated fibers. Research Implications: These findings highlight the potential for enhancing the performance of plant fiber-reinforced composites through chemical treatments, thereby enabling them to compete effectively with composites utilizing synthetic fibers. Originality/Value: This study contributes to advancing high-performance composite materials by addressing the critical issue of interfacial adhesion between polymer matrices and plant fibers, thus expanding the application potential of eco-friendly composites in various industries.
Keywords: Composite; Stipa Tenacissima; Crystalline Index; Tensile Strength; Alkaline Treatment; Adhesion.	
	
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ESTUDO DO COMPORTAMENTO MECÂNICO DE UM MATERIAL COMPOSTO REFORÇADO
COM FIBRAS DE STIPA TENACISSIMA

RESUMO

Objetivos: Esta pesquisa investiga métodos para melhorar a adesão entre matrizes de polímeros e reforços de fibras vegetais, especificamente fibras Alfa (Stipa tenacissima), usando uma solução de NaOH a 2% em peso por diferentes períodos (1, 3, 5 e 24 h).

Estrutura Teórica: Os compósitos reforçados com fibras vegetais são cada vez mais favorecidos por sua compatibilidade com o meio ambiente, baixa densidade, custo-benefício e propriedades mecânicas promissoras. No entanto, ainda existem desafios devido à adesão inadequada entre a matriz polimérica e as fibras vegetais, o que pode comprometer o desempenho do composto.

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Método: As fibras Alfa foram tratadas quimicamente com uma solução de NaOH a 2% em peso por diferentes períodos. Um conjunto abrangente de testes, incluindo FTIR, DRX, GTA, SEM e testes de tração, foi realizado para avaliar os efeitos do tratamento.

Resultados e Discussão: As análises de XRD revelaram um aumento significativo (36,26%) no índice de cristalinidade das fibras Alfa tratadas com uma solução de NaOH a 2% em peso por 5 horas em comparação com as fibras não tratadas. Além disso, os testes mecânicos demonstraram que os compósitos reforçados com fibras tratadas apresentaram propriedades mecânicas superiores em comparação com os reforçados com fibras não tratadas.

Implicações para a Pesquisa: Essas descobertas destacam o potencial de aprimoramento do desempenho de compósitos reforçados com fibras vegetais por meio de tratamentos químicos, permitindo que eles concorram efetivamente com os compósitos que utilizam fibras sintéticas.

Originalidade/Valor: Este estudo contribui para o avanço dos materiais compostos de alto desempenho ao abordar a questão crítica da adesão interfacial entre matrizes de polímero e fibras vegetais, expandindo assim o potencial de aplicação de compostos ecologicamente corretos em vários setores.

Palavras-chave: Compósito, Stipa Tenacissima, Índice Cristalino, Resistência à Tração, Tratamento Alcalino, Adesão.

ESTUDIO DEL COMPORTAMIENTO MECÁNICO DE UN MATERIAL COMPUESTO REFORZADO CON FIBRAS DE STIPA TENACISSIMA

RESUMEN

Objetivos: Esta investigación investiga métodos para mejorar la adhesión entre matrices poliméricas y refuerzos de fibras vegetales, específicamente fibras Alfa (Stipa tenacissima), utilizando una solución de NaOH al 2 % en peso durante diferentes duraciones (1, 3, 5 y 24 h).

Marco Teórico: Los materiales compuestos reforzados con fibras vegetales son cada vez más apreciados por su respeto al medio ambiente, su baja densidad, su rentabilidad y sus prometedoras propiedades mecánicas. Sin embargo, persisten problemas debidos a una adhesión inadecuada entre la matriz polimérica y las fibras vegetales, que puede comprometer el rendimiento del material compuesto.

Método: Las fibras de alfa se trataron químicamente con una solución de NaOH al 2 % en peso durante diferentes períodos de tiempo. Para evaluar los efectos del tratamiento se llevó a cabo un amplio conjunto de pruebas, incluyendo FTIR, DRX, GTA, SEM y ensayos de tracción.

Resultados y Discusión: Los análisis de DRX revelaron un aumento significativo (36,26%) del índice de cristalinidad de las fibras Alfa tratadas con una solución de NaOH al 2% en peso durante 5 horas en comparación con las fibras no tratadas. Además, las pruebas mecánicas demostraron que los materiales compuestos reforzados con fibras tratadas presentaban propiedades mecánicas superiores a las de los reforzados con fibras no tratadas.

Implicaciones para la Investigación: Estos resultados ponen de relieve la posibilidad de mejorar el rendimiento de los materiales compuestos reforzados con fibras vegetales mediante tratamientos químicos, lo que les permitiría competir eficazmente con los materiales compuestos que utilizan fibras sintéticas.

Originalidad/Valor: Este estudio contribuye al avance de los materiales compuestos de alto rendimiento al abordar la cuestión crítica de la adhesión interfacial entre las matrices poliméricas y las fibras vegetales, ampliando así el potencial de aplicación de los compuestos ecológicos en diversas industrias.

Palabras clave: Composite, Stipa Tenacissima, Índice Cristalino, Resistencia a la Tracción, Tratamiento Alcalino, Adhesión.

1 INTRODUCTION

Over the past few decades, composite materials have attracted considerable attention from researchers in materials science and engineering [1,2]. This is chiefly attributable to their various benefits, including a diminished volume-to-weight ratio, enhanced specific strength-to-weight ratio, malleability in shaping and sizing, corrosion resistance, uncomplicated

manufacturing process, recyclability, and economic efficiency [3,4]. Researchers are increasingly drawn to plant fiber-reinforced composites because of their environmentally friendly attributes, low density, cost efficiency, and remarkable mechanical properties. Nonetheless, challenges remain in this domain, especially concerning the inadequate adhesion between the polymer matrix and plant fibers, resulting in diminished mechanical properties of the resultant composites [5-13].

To improve the quality and durability of composites, it is essential to tackle the limitations posed by the hydrophobicity and wettability of the fibers [14]. A multitude of surface modification techniques has been investigated in the literature to address these challenges, including NaOH treatment, silane, potassium permanganate, benzoyl chloride, plasma treatment, laser treatment, and corona treatment, among others. Multiple studies have indicated these methods as viable means to alter the fiber surface, consequently enhancing its properties [15,16]. Benyahia et al. [17] examined the effects of alkali treatment on fiber-reinforced unsaturated polyester composites. The authors subjected Alfa fibers to different NaOH concentrations (1 %, 3 %, 5 %, and 7 %) for 24 h to improve bending resistance. The results demonstrated a notable enhancement in flexural strength, especially in composites reinforced with Alfa (*Stipa tenacissima*) fibers subjected to 7 % NaOH treatment.

Raharjo et al. [18] utilized an alkaline treatment to improve the fiber-matrix interface of cantala fiber in their study. The alkali treatment was performed for different durations (0, 3, 6, 9, and 12 h) utilizing a 6 % NaOH solution. The tensile properties, elastic modulus, and impact strength of unsaturated polyester resin composites reinforced with cantala fibers (UPR-CFs) were evaluated to determine the effects of the alkaline treatment. The findings indicated that a 6-h alkaline treatment produced the maximum density, tensile strength, and elastic modulus of the composites. Significant precedents in the literature further substantiate the effectiveness of these treatments. Mouissa et al. [19] conducted a chemical treatment on wood sawdust using a 5 wt % NaOH solution for 24 h at ambient temperature. Their extensive mechanical evaluations of composites reinforced with alkali-treated sawdust revealed increased flexural and compressive strength, attaining peak values of 0.89 and 4.85 MPa, respectively. Benamrane et al. [20] investigated the optimization of the mechanical properties of Alfa fibers utilizing the Taguchi method (L9), which encompasses three principal factors, each linked to three variables: gauge length (20 mm, 40 mm, 60 mm), NaOH treatment concentration (0.5 %, 2 %, 5 %), and treatment duration (48 h, 24 h, 12 h). The findings indicated enhancements in tensile stress and Young's modulus, with optimal properties identified at a length scale of 60 mm, a NaOH

concentration of 5%, and a curing duration of up to 24 h. According to M. Werchefani's et al.[21] study, enzymatic treatment of alfa fibers emerges as the most effective method for reducing their moisture absorption compared to chemical treatment. Another investigation conducted by Addour et al. [22] focused on the fabrication of composites utilizing an unsaturated polyester resin matrix reinforced with both treated and untreated Alfa fibers. The fiber treatment conducted with a 5% NaOH solution for varying durations (1, 3, 5, and 24 h) produced beneficial effects on the mechanical properties of the composites. Consistent with this body of research, our current study concentrates on the formulation of composite materials reinforced with Alfa fibers subjected to a 2 % soda solution for varying durations (1, 3, 5, and 24 h) at room temperature.

The main goal is to determine the impact of treatment duration on the mechanical properties, particularly tensile strength, of the examined composites.

2 MATERIAL AND METHODS

Alfa fibers were collected in the Hodna region (M'Sila, Algeria) in August 2024. The fibers were washed with tap water to eliminate dust and contaminants and then air-dried for three days at ambient temperature.

2.1 ALKALINE TREATMENT OF ALFA FIBERS

The treatment comprised 98 % pure sodium hydroxide (NaOH), acetic acid, and analytical grade glycerol (99.5 % purity). Chopped Alfa fibers underwent treatment in a 2 wt % sodium hydroxide solution for varying durations (1, 3, 5, and 24 h) at a solution ratio of 15:1 (w/v). Subsequent to treatment, the fibers were immersed in an acidified solution to eliminate surplus soda, rinsed with water to achieve a pH of 7, and dried in an oven at 105 °C for 6 h. The fibers utilized in this preparation measured 1 cm in length [22].

2.2 COMPOSITE PREPARATION

The research employed an unsaturated orthophthalic polyester matrix cured with 2 % methyl ethyl ketone peroxide. Specimens were created utilizing a wooden mold with dimensions of 200×20×4 mm³. The composite targeted a fiber content of 30 % (v/v) and was

produced utilizing the lay-up technique. Fibers were integrated into the matrix, and the resultant mixture was cast into the mold to produce the samples. Table 1 presents the sample codes.

2.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The spectra of the samples were obtained using an FTIR Affinity-1S SHIMADZU (Japan) located at the Laboratory of Chemistry at the University of Sétif, Algeria. The samples were prepared as tablets, consisting of approximately 5 % by mass of previously ground Alfa fiber (either treated or untreated) combined with 95 % by mass of potassium bromide (KBr). The scanning range extended from 400 to 4000 cm^{-1} , with a resolution of 2 cm^{-1} .

2.4 X-RAY DIFFRACTION (XRD)

The crystallinity index (CrI) values of the treated and untreated Alfa fibers were measured using a D8 diffractometer (Bruker-AXS, Germany) with copper radiation. The Cu $K\alpha$ ($\lambda = 1.54 \text{ \AA}$) was operated at 40 kW and 20 mA, with a scan rate of 5 $^{\circ}/\text{min}$ across a 2θ range of 5° to 70° . The crystallinity index (CrI) of the samples was determined using the diffraction intensities provided 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 $2\theta = 22^{\circ}$, and I_{am} is the intensity of the amorphous phase at $2\theta = 17^{\circ}$.

2.5 THERMO GRAVIMETRIC ANALYSIS

Thermal properties were examined through thermogravimetric analysis (TGA) to monitor the progression of thermal stability in fibers. Measurements were conducted in a nitrogen atmosphere utilizing thermal analysis equipment (SDT Q600 TA, USA) from 20 $^{\circ}\text{C}$ to 800 $^{\circ}\text{C}$ at a heating rate of 10 $^{\circ}\text{C}/\text{min}$.

2.6 SCANNING ELECTRON MICROSCOPY (SEM)

The SEM test was conducted post-fracture of the samples to evaluate the adhesion between Alfa fibers and the matrix. A JOEL JSM 7001F scanning electron microscope was utilized in this examination (Japan). The SEM images were acquired utilizing secondary electron imaging at a beam acceleration voltage of 15 kV.

2.7 MECHANICAL TESTING

2.7.1 Tensile test

This test was conducted to ascertain the tensile strength and elastic modulus of the composite, in accordance with ASTM D3039 standards, utilizing a Universal Testing Machine (Zwick Z50, Zwick-Roell, Germany) at a crosshead speed of 1 mm/min. Rectangular specimens measuring 250 x 22 x 21 mm³ were utilized for testing.

Table 1
Coding of different composite samples prepared

Symbol of composites	Annotation
UTAF/UP	Untreated Alfa fiber/unsaturated polyester
ATAF21/UP	Alfa fiber alkaline treated with 2 % for 1 h/ unsaturated polyester
ATAF23/UP	Alfa fiber alkaline treated with 2 % for 3 h/ unsaturated polyester
ATAF25/UP	Alfa fiber alkaline treated with 2 % for 5 h/ unsaturated polyester
ATAF224/UP	Alfa fiber alkaline treated with 2 % for 24 h/ unsaturated polyester

3 RESULTS AND DISCUSSION

3.1 FT-IR ANALYSIS

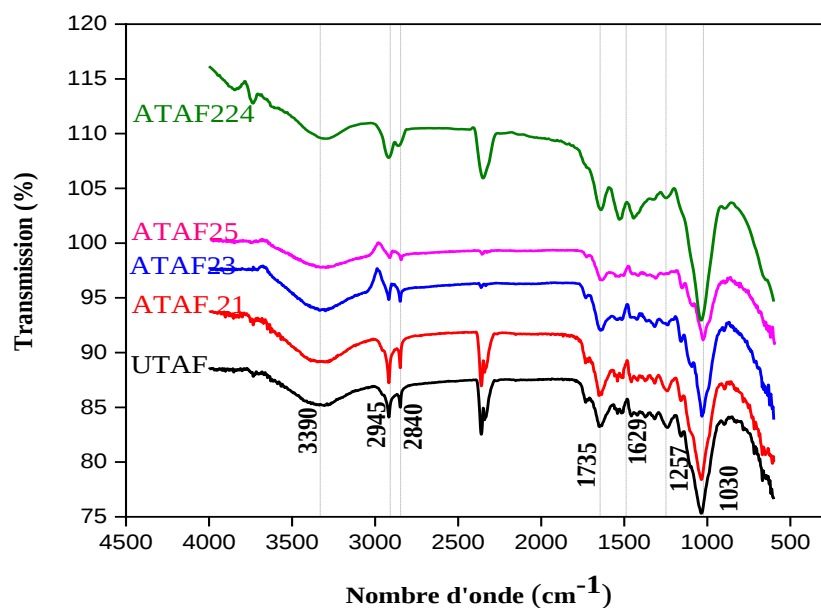
The spectra recorded from the FT-IR analysis of the untreated and treated Alfa fibers are presented in Figure 1, revealing nearly identical patterns. Minor differences were observed in the treated sample. The peak responses at specific wavelengths between 400 and 4000 cm⁻¹ indicate the presence of cellulose, hemicelluloses, and lignin components in the fiber structure. The broadband absorption around 3390 cm⁻¹ is attributed to the hydroxyl group (OH) bound to cellulose and hemicelluloses [12]. A significant absorption band (between 2840 and 2945 cm⁻¹) in both treated and untreated Alfa fibers may be associated with the alkyl C-H stretching

vibration (symmetrical and asymmetrical) present in the cellulose and hemicellulose components of natural fibers [24]. We notice a decrease in peak at 2945 cm^{-1} and 2840 cm^{-1} for samples ATAF25 and ATAF224.

Another peak at 1735 cm^{-1} is associated with carbonyl groups ($\text{C}=\text{O}$) resulting from the carbonyl ($\text{C}=\text{O}$) stretching vibrations of carboxyl and acetyl groups in hemicelluloses [25]. Furthermore, Figure 1 demonstrates that the maxima of samples ATAF23, ATAF25, and ATAF224 are inferior to those of samples ATAF21 and untreated Alfa fibers. This observation may be ascribed to the partial hydrolysis of cellulosic transcripts in alkaline conditions, in contrast to untreated Alfa fibers. The absorption bands at 1629 and 1257 cm^{-1} signify the stretching of acetyl groups in the lignin components, namely $\text{C}=\text{C}$ and $\text{C}-\text{O}$, respectively [26]. The intensity of two peaks (1257 and 1735 cm^{-1}) in the spectra of treated Alfa fibers (ATAF21, ATAF23, ATAF25, ATAF224) diminished relative to untreated Alfa fibers, signifying the elimination of certain lignin and hemicelluloses. The peak at 1030 cm^{-1} corresponds to the vibration of the $\text{C}-\text{O}$ bond in hemicellulose [27]. The elimination of hemicellulose components is observed to diminish the two peaks at 3390 and 1030 cm^{-1} [28]. As a result of the alkaline treatment and processing duration of Alfa fibers, specific fiber constituents, including lignin and hemicelluloses, have been dissolved.

Figure 1

Infrared spectra of untreated Alfa fibers and those treated with 2 % NaOH for 1, 3, 5 and 24 h



3.2 X-RAY DIFFRACTION

Figure 2 illustrates the diffraction spectra of untreated Alfa fibers and those subjected to a 2 % NaOH solution for varying durations (1, 3, 5, and 24 h) at room temperature. Table 2 demonstrates the enhancement of the crystallinity index (CrI) of treated fibers with prolonged treatment duration. The findings indicate that Alfa fibers subjected to a 2 % NaOH concentration for 5 h exhibit superior morphological structure and an elevated degree of crystallinity. It attained 36.5 %, reflecting a 17 % increase relative to the untreated Alfa fibers. The alkali treatment eliminates specific cementing agents, including hemicellulose, lignin, and waxes. The elimination of these materials results in an enhancement of the crystallinity index and cellulose content of the fibers [29]. Alkali treatment can specifically induce the cleavage of ester linkages between lignin and polyuronic, leading to the partial dissolution of the binding material.

As a result, the quantity of exposed cellulose increases, facilitating the crystallization of the treated fibers. This consequently enhances the crystallinity index in treated fibers[30,31]. The alkali treatment enhances the crystallinity index (CI) by alleviating the cellulose chains and eliminating surplus amorphous components, lignin, and waxes [32,33]. This may result from the reorganization of molecular chains, leading to superior fiber orientation compared to other concentrations. Addour et al. [22], Pickering et al. [34], and Maghlaoui et al. [35] have reported that alkaline treatment enhances the crystallinity index and thermal stability of fibers, including Alfa, harakeke, and hemp fibers. The increase may be ascribed to the elimination of amorphous hemicelluloses from the fibers [36,37,24], or it may result from the reorganization of the crystalline regions, leading to a more crystalline fiber structure [26,38,39]. After 24 h of treatment, the CrI of the ATAF224 sample diminishes, presumably due to extended alkalization. Prolonged soda treatment can harm the fibers and lead to a reduction in the crystallinity index [35,40].

Figure 2

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

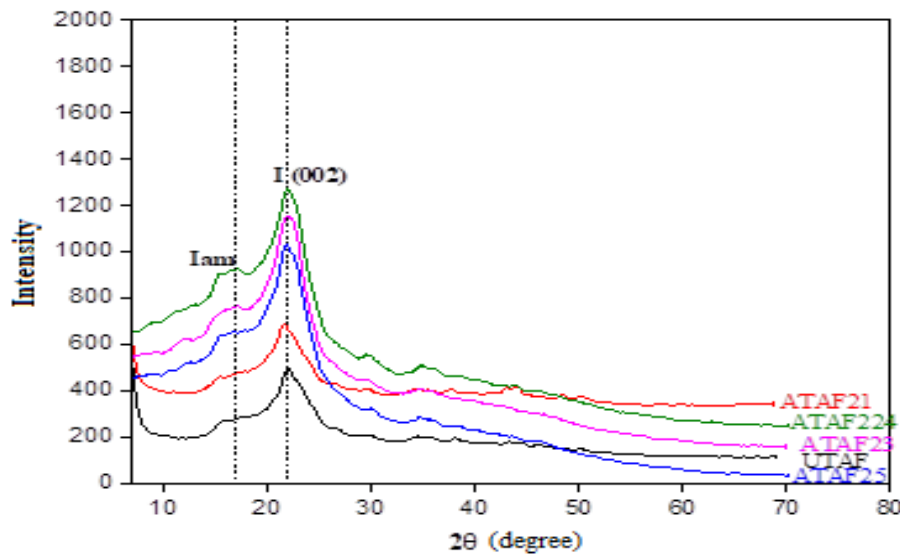


Table 2

The crystallinity index of untreated and treated Alfa fibers at different times (1, 3, 5 and 24 h)

Material	I (002)	I _{am}	CrI%
UTAF	430	312	27.7
ATAF21	450	310	31
ATAF23	1127	730	35.5
ATAF25	1126	714	36.5
ATAF224	1138	788	30.4

3.3 THERMO GRAVIMETRIC ANALYSIS (TGA)

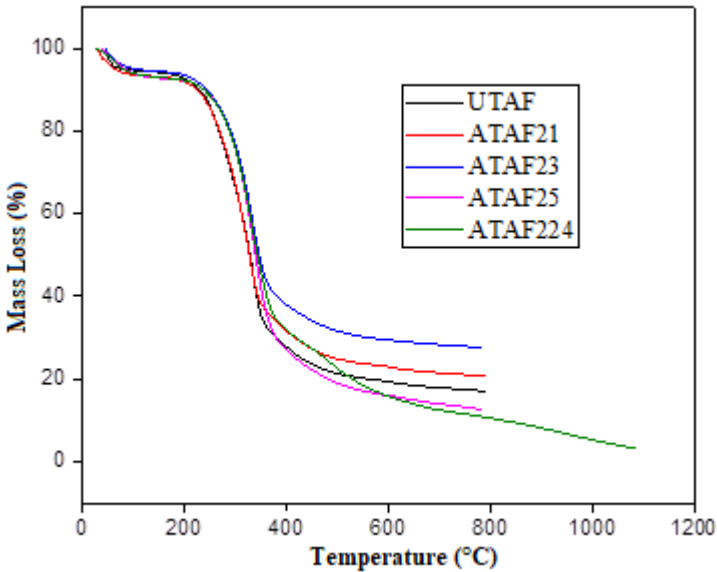
Thermogravimetric analyses were conducted to evaluate the impact of the treatments on the thermal stability of the fibers. Figure 3 illustrates the thermal stability curves of untreated Alfa fibers and those subjected to NaOH treatment (2 %) over different durations (1, 3, 5, and 24 h). It illustrates three regions of mass depletion. The treated and untreated Alfa fibers experienced a minor weight loss in the initial temperature range of 25 to 100 °C, attributed to the evaporation of water molecules. Multiple authors 33,34 have noted the same observation. Untreated Alfa fibers commence decomposition at approximately 205°C in the second region, spanning 200-290°C, whereas treated Alfa fibers initiate decomposition between 220°C and 230°C. The decomposition of hemicellulose may lead to the degradation of fiber [40,41]. Decomposition in the third region, 290-400 °C, pertains to the degradation of cellulose and lignin [42].

Izani et al. [43] established that temperatures between 200 °C and 400 °C are associated with the decomposition of cellulose and hemicelluloses. Lignin decomposition commences at temperatures exceeding 400 °C. The most challenging component to decompose stabilizes at 700 °C, or 30%, for treated fiber; conversely, the loss remains constant above 550 °C, or 37%, for untreated fiber. The mass loss of the treated fiber is inferior to that of the untreated fiber, as illustrated in Figure 3. Consequently, we can ascertain that fiber treatments improved thermal stability. We emphasize a transition towards elevated temperatures in the samples ATATF23, ATAF25, and ATAF224. Mohanty et al. [44] noted a similar finding. The results indicate that Alfa fibers subjected to NaOH treatment exhibited enhanced thermal properties over time (Table 3). Consequently, as indicated by FTIR measurements and chemical analysis, the variation in degradation temperatures of the treated Alfa fibers can be ascribed to the effective elimination of hemicelluloses [44,45].

Table 3
Thermal degradation parameters of untreated and treated Alfa fibers

Matériel	b UTAF	ATAF21	ATAF23	ATAF25	ATAF224
Td(°C)	205	220	223	223	221
Tf(°C)	359 369	366 376	380		
Mass loss at Tdmax (%)	60,1	54,7	50,2	61,3	57,1

Figure3
TGA of untreated and NaOH-treated Alfa fibers at different times(1,3,5 and 24 h)



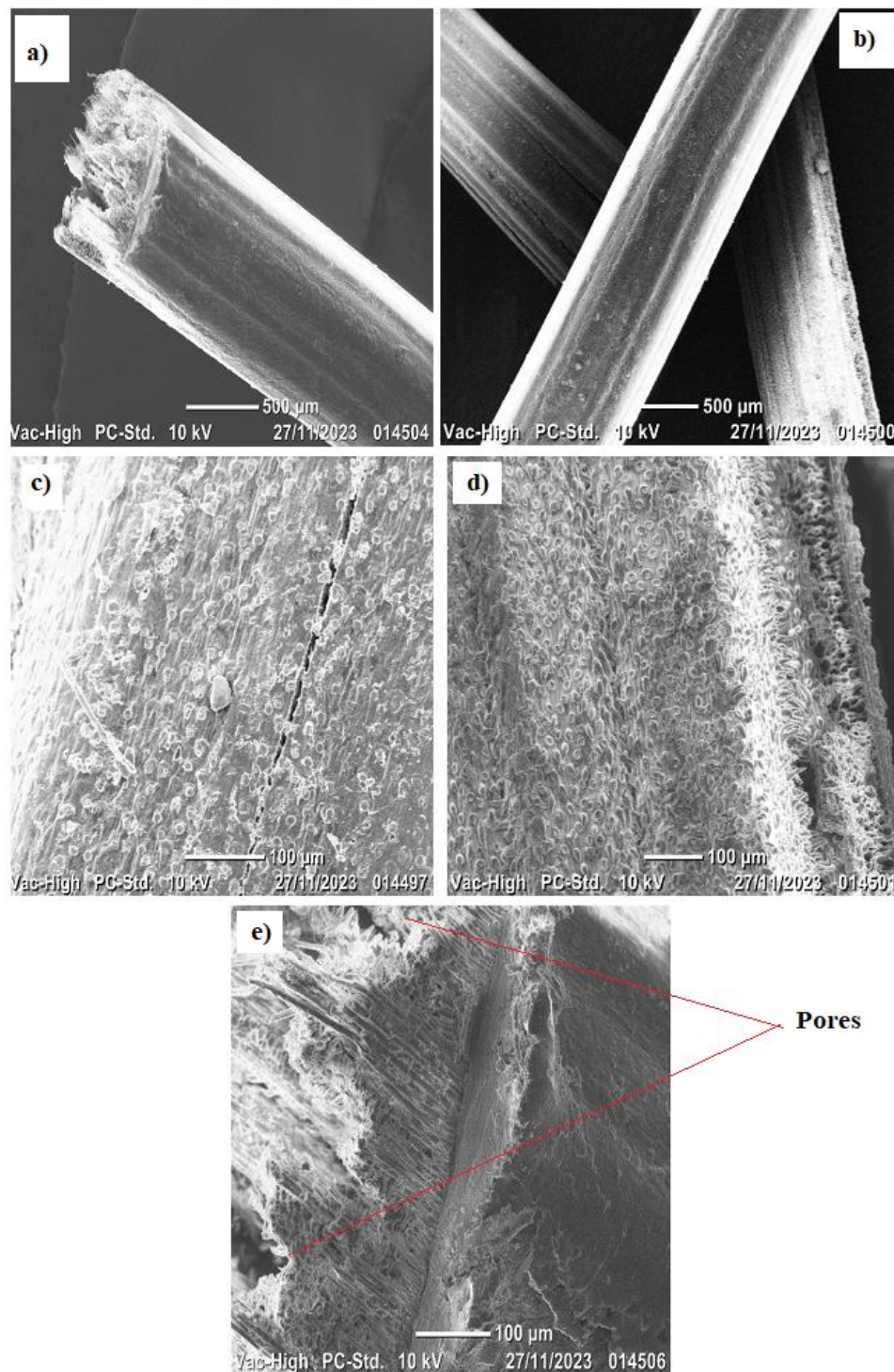
3.4 SEM ANALYSIS OF ALFA FIBERS

Figure 4 displays the scanning electron microscopy (SEM) micrographs of Alfa fibers. The external surface and longitudinal section of both treated and untreated Alfa stems were analyzed. Alfa rods, akin to all-natural fibers, resemble a composite material fortified by numerous elementary cellulose fibers (fibrils) aligned longitudinally, along with lignin and other non-cellulosic substances; the entirety is consistently coated with waxes and impurities (Fig. 4a) [46,47]. A comparison of various micrographs of treated and untreated Alfa stems demonstrates morphological alterations in the Alfa stem following chemical treatment with soda (Fig. 4b, 4c, 4d, and 4e). Untreated Alfa rods possess a smooth surface, while treated rods exhibit exposed fibrils. The surface roughness escalates with an increase in alkaline treatment duration, ranging from 1 h to 24 h.

Consequently, the elimination of non-cellulosic substances, including lignin, hemicelluloses, and wax, enhances the roughness of the treated fiber surface [48]. Surface roughness enhances the interfacial adhesion between the polymer matrix and the natural fibers, thereby augmenting the reinforcing function of the fibers [49]. The roughened surface promotes mechanical interlocking and bonding reactions through the exposure of hydroxyl groups to the matrix, alongside the dissolution of hemicelluloses. Prolonged alkali treatment, as observed by Sudhakara et al. [50], may enhance the interfibrillar region, thereby increasing the available surface area and subsequently improving adhesion at the fiber-matrix interface in composites.

Figure 4

SEM micrographs of (a) UAF; (b) ATAF21; (c) ATAF23; (d) ATAF25; (e) ATAF224

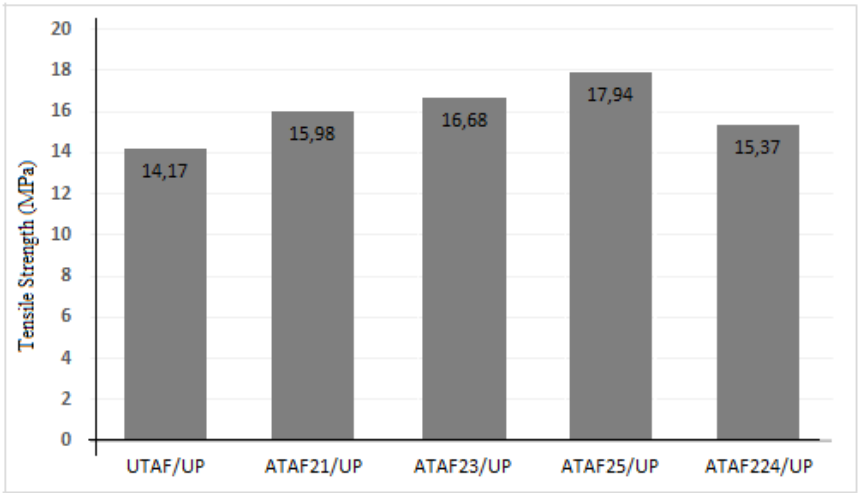


3.5 MECHANICAL TESTING OF COMPOSITES

3.5.1 Tensile strength and elastic modulus of unsaturated polyester-alfa fibers composites

The tensile strength of the unsaturated polyester/Alfa fiber composites is illustrated in Figure 5. The figure indicates that alkaline treatment of Alfa fibers for varying durations enhances the tensile strength of the composites in comparison to those reinforced with untreated fibers (UTAF/UP). Sakuri et al. [51] made a similar observation while investigating the impact of alkali treatment duration on the tensile strength of cantala fibers, utilizing the micromechanical reference for the tensile strength of unsaturated polyester-cantala fibers. The variance analysis conducted on the experimental results indicates that the duration of alkali treatment significantly influences the tensile strength of the composites. This outcome is ascribed to the effective adhesion of the treated fiber to the polymer matrix. The maximum tensile strength of composite ATAF25/UP was 17.94 MPa, indicating a 21 % enhancement compared to the untreated composite. The duration of alkali treatment for the fibers enhances the mechanical efficiency of the examined composites. Alkaline treatment diminishes fiber size, a phenomenon noted in other natural fibers like hemp [52], flax [53], and coconut [54], and enhances fiber dispersion within the matrix. The alkaline treatment eliminates impurities and structural components, including lignin, pectin, and waxy substances, that encase the outer surface of fiber cell walls. Moreover, it leads to fiber formation and creates a coarse surface for physical and chemical interactions at the filler-polymer interface [55].

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

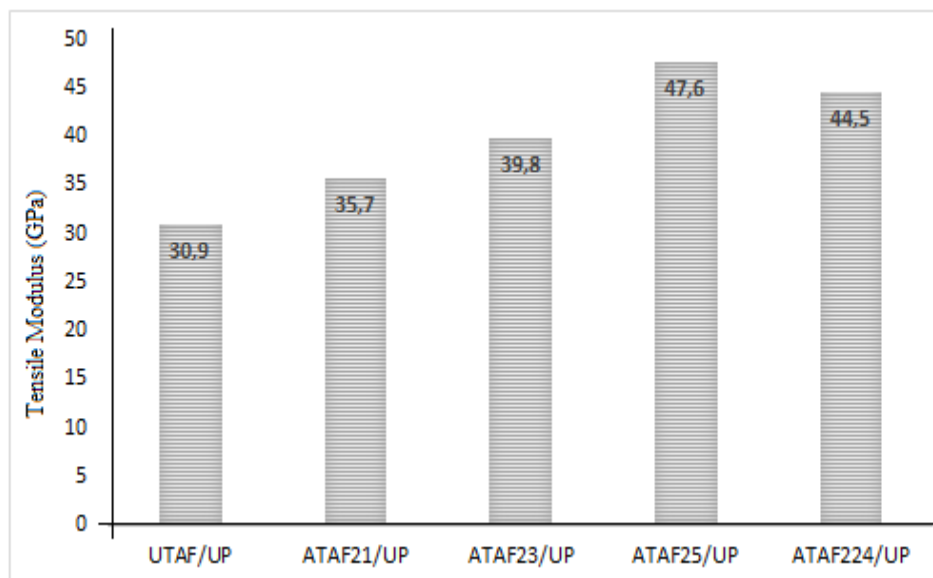


3.5.2 Tensile modulus

Figure 6 illustrates the progression of the tensile modulus in composites reinforced with both treated and untreated Alfa fibers. Composites reinforced with treated Alfa fibers demonstrate superior tensile moduli compared to those reinforced with untreated Alfa fibers. Extended fiber treatment duration enhances the stiffness of composites. These results align with those of Rocha et al. [56] and Arrakhiz et al. [57]. Conversely, composites reinforced with fibers subjected to 1 h of treatment exhibit an enhancement in the modulus of elasticity (ATAF25/UP). The maximum tensile modulus was 47.6 GPa, representing a 35 % increase compared to the untreated fiber-reinforced composites.

Figure 6

Effect of chemical treatment time on the tensile modulus of the studied composite



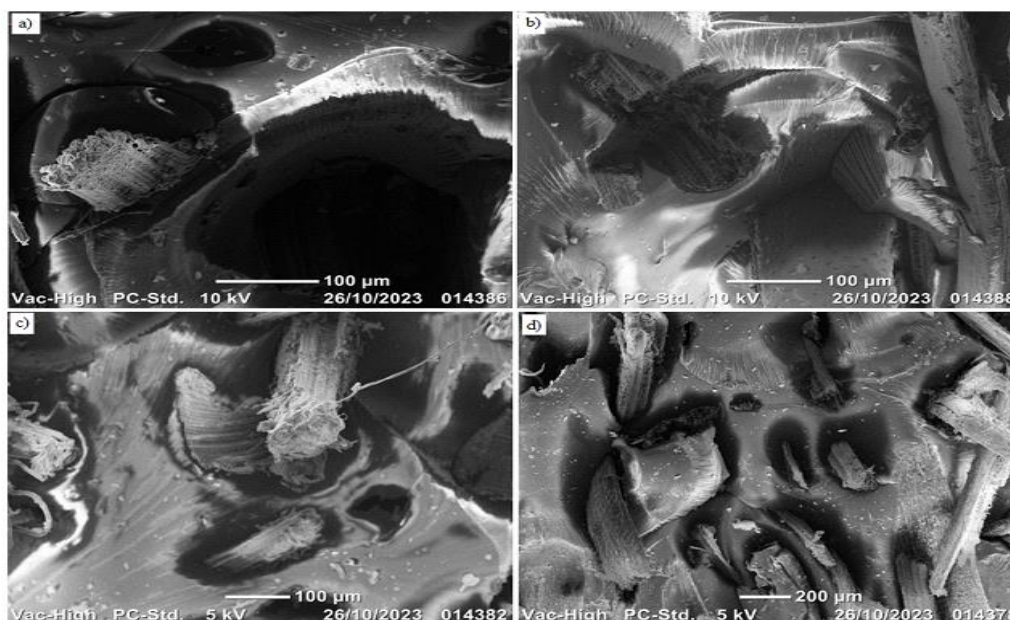
3.6 MORPHOLOGICAL CHARACTERIZATION OF COMPOSITES BY SCANNING ELECTRON MICROSCOPY

Figure 7 (a-d) presents micrographs of the fractured surfaces of composites composed of unsaturated polyester reinforced with Alfa fibers, as observed through a scanning electron microscope (SEM). The SEM images of the UTAF/UP, ATAF23/UP, ATAF25/UP, and ATAF224/UP formulations were analyzed to assess the impact of alkaline treatment on the fiber-matrix interface. Figure 7a illustrates the existence of microvoids and cavities on the UTAF/UP sample's surface, signifying incompatibility between the two phases resulting from

inadequate interfacial adhesion and the disparity in surface energies (or polarities) between the hydrophilic filler and the hydrophobic polymer [58]. The micrographs of the fractured surfaces of the treated Alfa/UP composites (Figures 7b and 7c) exhibit enhanced adhesion and compatibility between the Alfa fibers and the UP matrix, with a reduction in voids and cavities following the modifications. Nonetheless, the ATAF224/UP sample (Figure 7d) exhibits discernible gaps, indicating insufficient adhesion between the fibers and the polymer matrix. The inadequate bonding enabled fiber displacement from the matrix, as corroborated by the tensile test outcomes.

Figure 7

SEM micrographs of untreated and treated composites; (a) UTAF, (b) ATAF21, (c) ATAF25, (d) ATAF224



4 CONCLUSION

This study successfully manufactured and characterized alfa-reinforced polyester-based composites. The mechanical properties of the composite material were engineered to be enhanced through the pre-manufacturing treatment of the fibers. The mechanical testing of the material containing Alfa (*Stipa tenacissima*) fiber treated with 2 wt % NaOH solution for 5 h demonstrated an elevated mechanical strength, with tensile strength measuring 17.94 MPa and tensile modulus at 47,6 GPa. This finding indicates strong fiber-matrix adhesion, facilitating effective stress transfer from the matrix to the fiber. The morphological analysis of the treated

fiber indicated that the longitudinal surface examination demonstrated an increase in roughness following prolonged alkali treatment.

Moreover, the alkaline treatment enhanced the crystallinity index of the Alfa fibers. The ATAF25 sample attained the highest value, with the crystallinity index rising by 17 % relative to untreated fibers. The treated ATAF21/UP, ATAF23/UP, ATAF25/UP, and ATAF224/UP composites exhibited an upward trend in crystallinity index values, correlating with their favorable mechanical properties. FT-IR analysis of the composites demonstrated the elimination of hemicelluloses, lignin, and other surface contaminants from the fiber surface. These findings may expand the potential applications of Alfa fiber-reinforced polyester composites as viable materials.

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