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Research Paper

Optimization of Alkali Treatment for *Stipa tenacissima* Fibers in Polyester-Based Composites

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Abstract
Water soaking was used to recover fibers from the Djelfa region in southern Algeria, which were then modified with sodium hydroxide at various concentrations (3%, 6%, and 9%) for 5 h at 40°C. Manual stacking and compression molding procedures were used to create composite samples containing 10 vol % treated and untreated esparto fibers in a polyester matrix. The samples were analyzed using FTIR, XRD, TGA, SEM, and three-point flexural testing. Esparto fibers treated with 9% NaOH demonstrated a 60.39% higher crystallinity index versus raw fibers. Flexural characteristics analysis revealed that composites reinforced with esparto fibers modified with 9% NaOH exhibited superior mechanical performance relative to those containing untreated fibers, with improvements of 47.90% in flexural strength and 48.46% in flexural modulus, respectively. These findings indicate that when chemically treated, *Stipa tenacissima* fibers hold strong potential as a sustainable alternative to synthetic fibers for reinforcing polymer composites in structural applications.

Keywords
Composite, Alkali Treatment, *Cynodon dactylon*, Adhesion, Hydrophilic

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1. INTRODUCTION

With the considerable growth in the utilization of composite materials over the last decades and the accompanying environmental challenges, the scientific community has been actively exploring sustainable alternatives to mitigate ecological impacts. Among these, natural fibers in particular, have emerged as feasible options for stiffening polymer composites due to their benefits such as low weight, biodegradability, low cost, availability, and environmental friendliness (Shaker et al., 2020; Siakeng et al., 2019). Nonetheless, the integration of plant-based fibers into composites presents certain drawbacks, primarily their hydrophilic nature. This property leads to moisture absorption from the surrounding environment (Hashim et al., 2012; Okuda and Sato, 2004), causing dimensional instability and poor fiber-matrix bonding, which thereby reducing the mechanical strength of the final material.

A widely adopted strategy to address this issue is the alkaline (NaOH) treatment of plant fibers. This chemical treatment improves fiber surface properties by providing a rougher texture, which facilitates stronger interfacial interaction with the polymer (Benyahia and Merrouche, 2014; Melouki et al., 2023). The alkali acts by disrupting the lignin structure, thereby loosening the interfiber bonds and making the fibers more flexible and less prone to damage (Geng et al., 2006; Corradini et al., 2007).

Several studies have examined how such treatments affect the mechanical properties of natural fiber-reinforced composites. Obasi et al. (2014), for example, demonstrated improved tensile behaviour in epoxy composites fortified with oil palm fibres following alkali modification, which was related to the elimination of hemicellulose and surface contaminants. Similarly, Fiore et al. (2015) discovered that kenaf/epoxy composites modified with NaOH exhibited increased modulus values compared to untreated systems. Laib et al. (2025) found that treating Alfa fibres with 4% NaOH for varied durations significantly improved mechanical performance, with tensile strength reaching 17.94 MPa and tensile modulus of 47.6 GPa. The enhancements were linked to improved interfacial adhesion and increased crystallinity, which facilitated stress transfer within the composite. In another study, Ayalew and Wodag (2022) developed polyester composites reinforced with sisal fibers at varied concentrations (2%, 6%, and 10%) of NaOH for 24 to 72 hours. Optimal properties were obtained with 6% NaOH for 48 hours and a fiber-to-matrix ratio of 30:70, achieving tensile

and flexural strengths of 44.0 MPa and 50.8 MPa, respectively. The research confirmed that alkalization not only boosts mechanical performance but also reduces water uptake in contrast to raw fiber composites. Addour et al. (2022) developed unsaturated polyester composites using Alfa fibers treated with 5% NaOH for different periods. Fibers treated for 5 h produced composites with a flexural strength of 64.30 MPa-an increase of 32% over raw fiber composites. Alam et al. (2022) further explored the influence of 5% NaOH on rattan and bamboo fibers used alongside 5% eggshell powder in hybrid laminated polyester composites.

They observed mechanical enhancements in tensile, flexural, and impact resistance by 9%, 2%, and 7%, respectively, as well as a 4% increase in stiffness. Mishra et al. (2003) showed that sisal/glass hybrid polyester composites treated with 5% NaOH exhibited higher tensile, flexural, and impact strengths than those treated with 10% NaOH. Saraswati et al. (2025) introduced aminated carbon nanofiber fillers into epoxy and polyimide matrices, resulting in composites with superior mechanical strength, thermal stability, and electromagnetic shielding properties compared to unmodified nanofiber systems. Alfa grass *(Stipa tenacissima)*, commonly found in semi-arid regions of North Africa and Southern Europe, has gained attention for its fiber potential (Boudjellal et al., 2021; Boumediene et al., 2021). In Algeria, *Stippa tenacissima* covers approximately three million hectares (Beroual et al., 2021). Belonging to the Poaceae family, this drought-resistant grass grows up to one meter tall and consists mainly of cellulose (38.8–46.1%), hemicellulose (24.0–33.5%), and lignin (19.9–24.0%) (Borchani et al., 2015; Trache et al., 2016). Its fibers, which average 113 μm in diameter and in density range from 0.89 to 10 g/cm^3 (Grine et al., 2023) exhibit, show promising mechanical properties and are suitable for use in unsaturated polyester-based composites. Technically, Alfa fibers exhibit a favorable combination of tensile strength, low density, and moderate stiffness, making them suitable for the evolution of lightweight and structurally efficient materials. Economically, they represent a readily available and inexpensive local resource in North Africa (Derabi et al., 2020). From an ecological perspective, they are biodegradable and renewable, aligning well with sustainability objectives and the shift move away from synthetic reinforcements (Abdelhak, 2024). Furthermore, their chemical composition makes them ideal candidates for surface modifications such as alkalization or silanization, which improve adhesion to polymer matrices by increasing surface roughness and removing non-cellulosic components (Ben Daoud et al., 2022). Their natural insulating properties also make them suitable for applications in the construction and automotive sectors, as well as in packaging.

This study focuses on assessing how different NaOH concentrations (3%, 6%, and 9%) affect the flexible properties of short Alfa fiber-reinforced composites. The chemical modification was conducted at 40°C for 5 hours, and both untreated and treated fibers were incorporated into an unsaturated polyester resin at a constant fiber content of 20%. Composites were made via hand lay-up moulding, and their flexural characteristics were assessed to establish the efficacy of the alkali treatment.

2. EXPERIMENTAL SECTION

2.1 Materials

This investigation used sodium hydroxide (NaOH, Riedel-de Haën), acetic acid (CH_3COOH, PROLABO, 99%, density: 1.05 g/cm^3), a hot detergent solution (2%), and an industrial isophthalic unsaturated polyester resin (UP). The resin was cured with 1% methylethyl ketone peroxide (MEKP) and 0.5% cobalt naphthenate. The *Stipa tenacissima* fibres utilized in this investigation came from Algeria's Djelfa area.

2.2 Preparation of Fibers

Stipa tenacissima, frequently referred to as Alfa grass, is an annual plant that grows in the Mediterranean's arid and semi-arid regions. It is widely recognized as a valuable natural raw material due to its elevated cellulose content and fibrous structure, which make it particularly suitable for reinforcement in composite materials. Its abundance, renewability, and promising mechanical characteristics justify its use in the present study. Initially, the Alfa fibers underwent a scouring treatment involving a 2% hot detergent solution, aimed at eliminating surface contaminants and loosely attached residues. After completely washed with distilled water, the stems were dried at room temperature for 72 hours. The stems were first kept for 60 days at room temperature in plastic bags that were shielded from the sun. After that, they were cut into segments that were 6 cm long, and the fibrils were separated by grinding them in a tiny grain mill. To get rid of volatile ingredients and tiny particles, the resultant material was sieved. In order to improve separation and softening, the fibres were then filtered, cleaned with acetic vinegar, and then completely rinsed with distilled water. A fan-assisted oven set to 80°C for five hours was used for drying. In order to serve as a reference material for further alterations, these processed fibres were identified as raw Alfa fibres.

2.3 Alkaline modification of Alfa Fibres

Sodium hydroxide was chosen for the chemical modification of Alfa fibres due to its well-documented propensity to dissolve amorphous components such as hemicellulose and lignin. This treatment enhances the fiber-matrix interface by improving fiber roughness and surface activity (Rai et al., 2025). Following the preliminary cleaning, the fibers were immersed in NaOH solutions of 3%, 6%, and 9% by weight. The treatment was conducted at 40°C for 5 hours with a fiber-to-solution weight-to-volume ratio of 1:20. After treatment, the fibres were neutralised with an acetic acid solution and rinsed with purified water. oven-dried for 48 hours at 80°C.

2.4 Composites Fabrication

To ensure homogeneous dispersion, the prepared fibers were uniformly distributed in a mold with dimensions of 300 × 300

× 5 mm^3. A 5 wt% unsaturated polyester (UP) resin-based diluent was applied to the randomly oriented fiber mats to improve their integrity during the molding stage. The mold was subsequently closed and pressure was applied to compress the mat into a coherent structure. Composite panels were fabricated using the same mold dimensions. The reinforcement consisted of four random fiber mats (Table 1), which were thoroughly impregnated with polyester resin and then compressed using the mold cover. Five composite plates were made using the hand lay-up approach (Figure 1), using the method provided by (Ben Brahim and Ben Cheikh, 2007). The soaked mats were dried for 24 hours at 50°C. All formulations contained a fiber weight fraction of 40%.

Table 1. Coding of Various Composite Samples

Composites	Material code
Raw fibres / Unsaturated Polyester	UAF/UP
Alkali treated fibres (3 %) / Unsaturated Polyester	ATAF35/UP
Alkali treated fibres (6 %) / Unsaturated Polyester	ATAF65/UP
Alkali treated fibres (9 %) / Unsaturated Polyester	ATAF95/UP

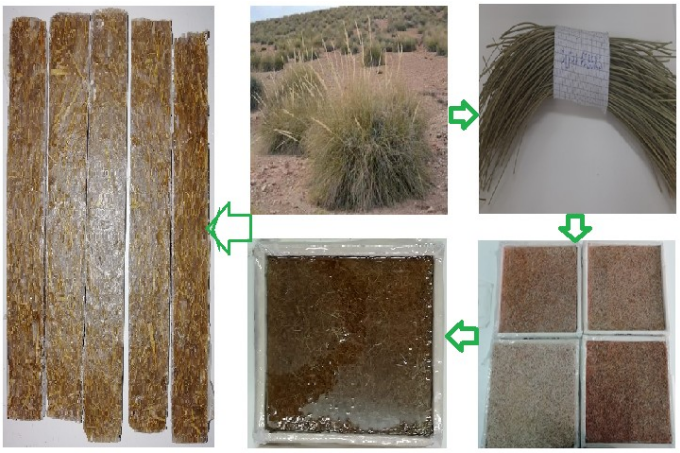

Figure 1. Composite Fabrication Process

2.5 Characterization

FTIR analysis of Alfa fibers was performed utilizing a Shimadzu FTIR-8300 spectrometer (Japan) operating in the range of 4000 to 400 cm^{-1}. To determine the crystallinity of the fibers, an X'Pert High Score diffractometer was used. The diffraction patterns (2θ) were recorded over a range of 10° to 40° using Cu-Kα radiation at 40 kV and 20 mA. The crystallinity index (CI) was computed using the method of Segal et al. (1959), which is provided in Equation (1):

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

where; I_{002} is the intensity of the primary Cellulose I peak at roughly 22°, whereas Iam is the intensity related to amorphous cellulose at roughly 16.53°.

The structure of treated and raw Alfa fibers was tested by SEM (JEOL model JSM 6490-LV) at a resolution of 3.0 nm, an accelerating voltage of 30 kV, and under high vacuum conditions. To assure conductivity, each sample's cross section was coated with a small layer of gold. The Alfa fibres were thermally analyzed using a Shimadzu TGA-50 thermogravimetric analyzer. Specimens were heated from 25 to 700°C at a steady rate of 10°C per minute. To keep the atmosphere inert throughout heating, nitrogen gas was continually supplied at a rate of 20 ml/min.

2.6 Mechanical Tests

Flexural properties, including strength and modulus, were assessed using Machine (Zwick Z50, Zwick/Roell, Germany) operating at a crosshead speed of 1 mm/min. Testing was performed on rectangular specimens with dimensions of 110 × 22 × 21 mm^3. The specimens underwent a three-point bending test in accordance with the ASTM D790 standard. Load-displacement curves were used to calculate the flexural strength and flexural modulus of the composites, using Equations (2) and (3), respectively:

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

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

where, L distance between the two supports (mm), b specimen width (mm), d specimen thickness (mm), P (N): maximum load applied before failure (N), and m slope of the linear component of the load-deflection curve (N/mm).

3. RESULTS AND DISCUSSION

3.1 Infrared Spectral Analysis

Figure 2 displays the FTIR spectrum of non-treated and alkali-treated Alfa fibers. According to Dalmis et al. (2020) and Keskin et al. (2020), hydroxyl groups in lignin, hemicellulose, and cellulose exhibit a broad absorption band centered at 3359.17 cm^{-1}, which corresponds to O−H stretching vibrations. The asymmetric and symmetric stretching vibrations of C−H bonds, mostly associated with −CH and $-CH_2$ groups in cellulose and hemicellulose, produce two different peaks at 2925.87 cm^{-1} and 2857.23 cm^{-1}, respectively (Deghfel et al., 2025; Yew et al., 2019). The absorption bands at 1718.91 cm^{-1} and 1248.78 cm^{-1} indicate the presence of carbonyl and ester functional groups often seen in hemicellulose and lignin (Raju et al., 2021; Erdogan et al., 2016). These peaks significantly diminished or disappeared after alkaline treatment, indicating partial or complete removal of these components. The bands at 1638.83 cm^{-1} and 1242 cm^{-1} correspond to C=C and C−O stretching vibrations of acetyl groups in lignin (Kabir et al.,

2012; Rahmatullah et al., 2022). The peak at 1253.6 cm^{-1}, representing the C=O stretching of lignin's acetyl groups, was notably reduced, further confirming the partial elimination of lignin (Syafri et al., 2021). The absorption at 1030 cm^{-1} is due to C−O stretching in hemicellulose (Boopathi et al., 2012), and the peak at 1149.76 cm^{-1} derives from the asymmetric stretching of C−O−C bonds in both cellulose and hemicellulose (Benyahia et al., 2013). The disappearance of this band after treatment indicates degradation or extraction of hemicellulose. Finally, the band found at 1043.93 cm^{-1} relates to C−O stretching vibrations connected to hydroxyl and ether groups in cellulose (Maache et al., 2017). The post-treatment FTIR spectra revealed a noticeable reduction or complete disappearance of the absorption bands at approximately 1718.91, 1248.78, 1253.6, and 1149.76 cm^{-1}, signifying the effective removal of lignin and hemicellulose. These findings align with previous studies on Tossa/Daisee jute fibers Kılınc et al. (2018) and *Althea officinalis* L. fibers (Kasyapi et al., 2013).

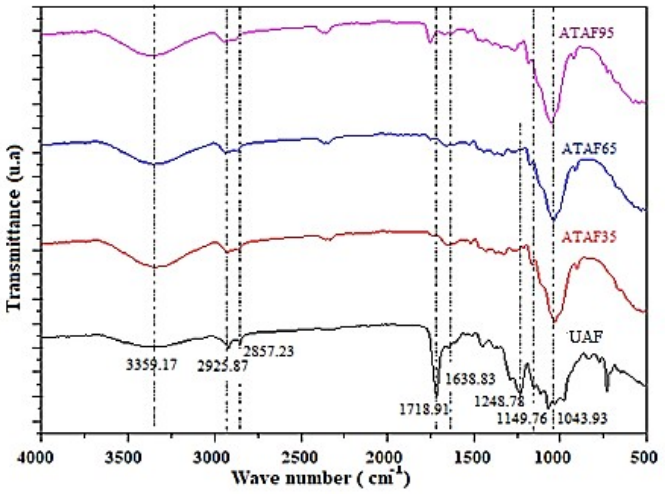

Figure 2. FTIR Analysis of Raw and Chemically Modified Alfa Fibers

3.2 X-Ray Diffraction

Figure 3 presents the X-ray diffraction patterns of raw and alkali-modified Alfa fibers. The diffraction profile of the untreated fiber shows a characteristic cellulose I structure, evidenced by a prominent peak around 2θ = 22°, which corresponds to the (002) crystallographic plane of cellulose I. A less intense diffraction dip observed at 2θ = 16.53° is attributed to the amorphous portion of the material (Iam). The crystallinity index (CI) was estimated by Segal's empirical approach, which is described in the part on experiments. The results, provided in Table 2, demonstrate that the sample ATAF95 had the greatest crystallinity index, with a 60.39% increase over the raw fibre. This significant enhancement suggests improved structural order, which may contribute to better mechanical performance, particularly in terms of flexural strength. The

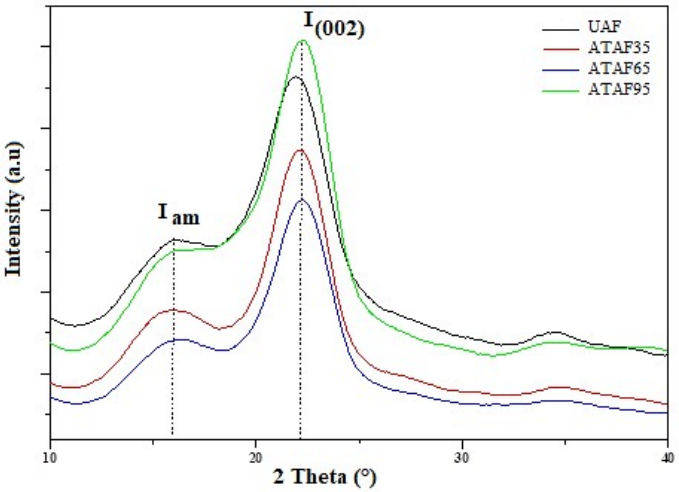

Figure 3. X-ray Diffractograms of Raw and NaOH-Modified Alfa Fibres

observed rise in CI is primarily attributed to the elimination of amorphous constituents, including lignin, pectin, hemicellulose, and surface pollutants, as confirmed by FTIR and SEM characterzation. It could possibly come from the reorganization of cellulose chains into more organized crystalline areas (Ouajai and Shanks, 2005). Borchani et al. (2015) reported similar findings for esparto grass fibers modified with NaOH at concentrations of 1 and 5 wt%. The study demonstrated that alkali treatment enhanced fiber crystallinity by eliminating amorphous components.

Table 2. Alkali-Treated and Untreated Alfa Fiber Crystallinity Index

Material	I (002)	Iam	CI (%)
UAF	381.96	181.96	26.18
ATAF35	186.94	89.09	52.35
ATAF65	244.09	125.65	48.52
ATAF95	256.02	86.78	66.10

3.3 Thermogravimetric Testing

The limited thermal stability of natural fibers constrains their application as reinforcement materials in polymer-based composites. To assess the thermal behavior and degradation profile of both raw and alkali-modified Alfa fibers, thermogravimetric analysis (TGA) was performed. As presented in Figure 4, four distinct weight loss phases were observed. The first phase, occurring between room temperature and approximately 110°C for raw fibers, and up to 125°C for modified fibres, is marked by moderate weight loss (8 %), attributed to the evaporation of residual moisture. Following this stage, thermal stability is maintained up to 280°C for treated fibers, while untreated fibers remain stable only up to 225°C. The next stage, between 200°C and 480°C, involves the thermal degradation of key

organic constituents, including hemicellulose, cellulose, and partially lignin. Weight loss during this phase is approximately 23% for raw fibers and 26% for treated fibers. These temperature ranges match those identified in the literature, particularly by Azwa et al. (2013), who noted that hemicellulose decomposes between 135°C and 400°C. The maximum degradation temperatures (Td max) for the treated fibers were recorded at 271.28°C, 274.91°C, and 282.41°C, compared to 253.48°C for untreated fibers (see Table 3). This improvement is due to the partial elimination of amorphous components, which enhances thermal resistance. Additionally, the alkali modification increases the crystallinity of the fibers by removing amorphous cellulose regions, thereby enhancing thermal stability (Norul Izani et al., 2013). In contrast, untreated fibers, which retain a higher proportion of thermally unstable components, begin to degrade at lower temperatures (Emmanuel et al., 2022).

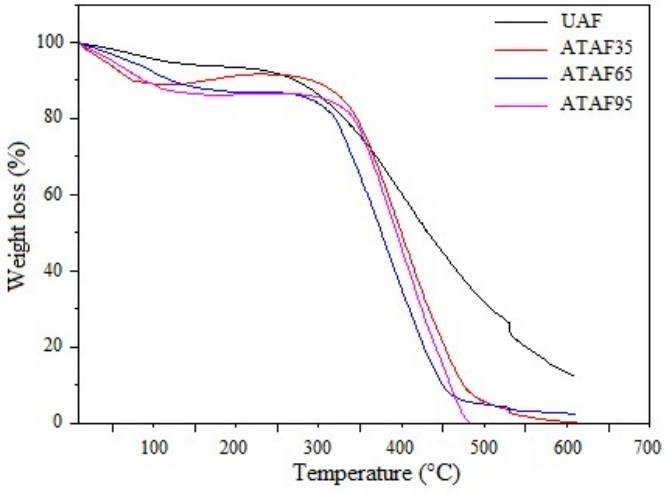

Figure 4. TGA Analysis of Alkali-Modified and Raw Alfa Fibers

Table 3. Degradation Temperatures of Treated and Untreated Fibers

Material	T (°C)
UAF	253.48
ATAF35	271.28
ATAF65	274.91
ATAF95	282.41

3.4 Morphological Analysis

The surface morphology of Alfa fibers undergoes notable modifications following alkali treatment. Figure 5 illustrates the microstructural differences between raw and modified fibers. As shown in Figure 5(a), the untreated Alfa fibers (UAF) display a relatively smooth surface covered with waxy substances, oils, and various impurities. Figures 5(b), 5(c), and 5(d) demonstrate that treated fibers exhibit a rougher and cleaner surface, with exposed fibrils resulting from the elimination of hemicellulose, lignin, and surface waxes. These structural alterations are compatible with the FTIR study findings, which confirm the elimination of non-cellulosic components.

The findings are consistent with those obtained by Kassim et al. (2013), who demonstrated that alkali treatment effectively cleans the fiber surface, thereby enhancing adhesion between fibers and polymer matrices. Similar effects were observed by Mylsamy and Rajendran (2010) in *Agave americana* fibers and by Sghaier et al. (2009) in doum palm fibers, where alkali treatment led to a marked reduction in surface impurities and improved interfacial compatibility. Meghlaoui et al. (2019) also confirmed that the treatment efficiently eliminates hemicellulose, lignin, and waxes. Additionally, Nguyen and Nguyen (2022) reported a significant increase in surface roughness and better structural alignment in water hyacinth fibers following alkaline treatment. Nguyen and Nguyen (2022) discovered an important rise in surface roughness and better structural alignment in water *hyacinth* fibers after alkaline treatment.

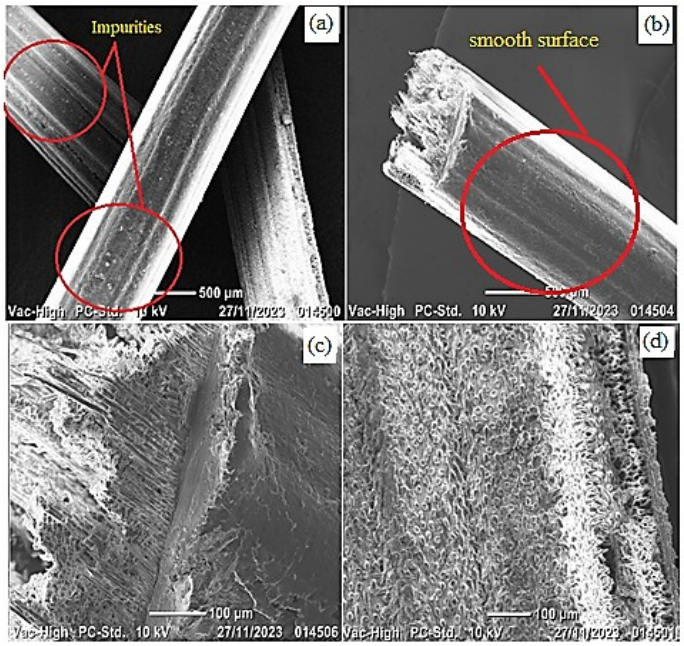

Figure 5. SEM Images of Raw and NaOH-Modified Alfa Fibres a) UAF; b) ATAF35; c) ATAF65; d) ATAF95

3.5 Flexural Strength and Modulus

The flexural behavior of composites, displayed in Figures 6 and 7, highlights a marked enhancement for alkali-modified samples, particularly ATAF95/UP, which yielded the highest flexural strength. (Figure 6), showing a 47.90% increase compared to the UAF/UP composite (Table 4). This notable enhancement is ascribed to better interaction among the alkali-modified fibre and unsaturated polyester. The improved mechanical perfor-

mance is most likely due to enhanced fiber-matrix adhesion resulting from by fiber fibrillation and the removal of surface contaminants during alkali treatment. Similar findings were found by Ullah et al. (2025) and Pradeep et al. (2011) reported similar results in studies on bamboo-reinforced epoxy composites and sisal fiber-polyester composites, respectively.

Likewise, the flexural modulus of the composites elevated after alkali treatment (Figure 7). The ATAF/UP composite achieved the greatest modulus value of 6.19 GPa, representing a 48.46% improvement over the untreated fiber composite. These findings underscore the positive effect of chemical treatment on the mechanical characteristics of prepared samples.

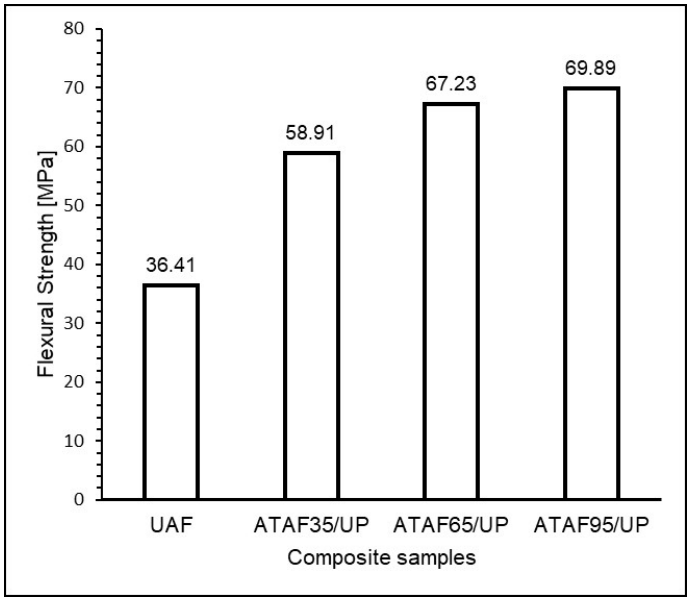

Figure 6. Flexural Strength of Modified and Raw Alfa Fiber Composites

Table 4. Mechanical Properties of the Investigated Composites

Material	Flexural Strength (MPa)	Flexural Modulus (GPa)
UAF/UP	36.41±2.9	1.51±0.2
ATAF35/UP	58.91±7.3	2.07±0.88
ATAF65//UP	67.23±6.33	2.42±0.97
ATAF95//UP	69.89±3.01	2.93±0.69

3.6 Morphological Analysis

The surfaces of the specimens subjected to flexural test were examined using a scanning electron microscope, as shown in Figure 8. In Figure 8(a), the micrograph of the composite reinforced by raw Alfa fibres shows poor adherence at the fibre-matrix contact. accompanied by visible delamination. This observation suggests the presence of a thick layer of surface impurities, including waxes and pectins, on the untreated fibers

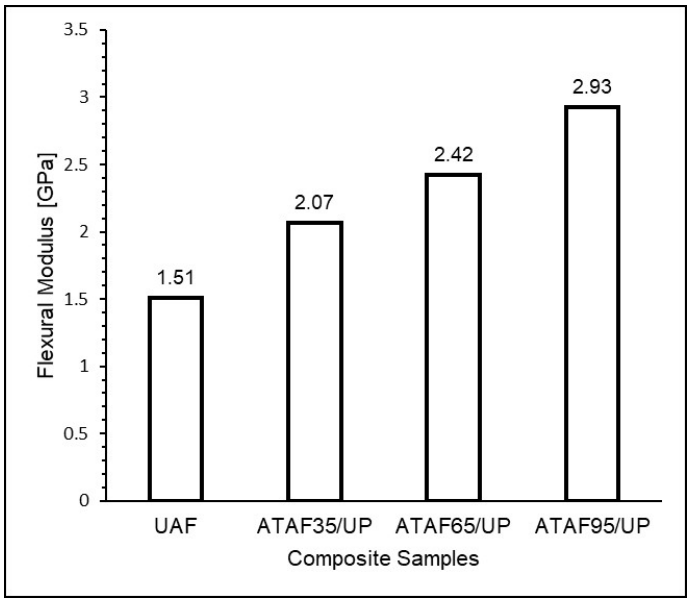

Figure 7. Flexural Modulus of Modified and Raw Alfa Fiber Composites

(Ishak et al., 2013; Rashid et al., 2016). Such a surface layer likely hindered effective bonding, resulting in weak interfacial interactions. Moreover, fibre being pulled out and voids were discovered, indicating insufficient mechanical conjunction among the fibre and the matrix. These structural discontinuities likely contributed to the reduced flexural strength observed in the UAF/UP composite. In contrast, Figure 8(b) shows a

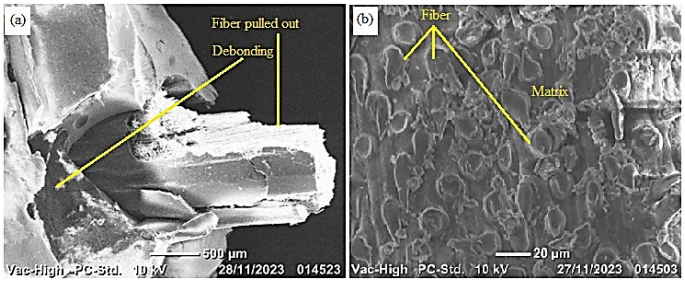

Figure 8. Structural Characterization of the Composite at (a) UAF/UP and (b) ATAF95/UP

much-improved interfacial morphology for the alkali-treated fiber composite. The fibers appear well embedded in the matrix, with no visible signs of debonding or interfacial gaps. This denotes strong interfacial adhesion, which enhances load transfer capability and the overall mechanical integrity of the composite. These findings align with those published by Herlin et al. (2025), who observed improved fiber-matrix bonding in composites enhanced with alkali-modified fibers due to increased surface roughness and mechanical interlocking. Similar findings were reported by Sathishkumar et al. (2017) in their investigation of vinylester composites reinforced with jute fiber

mats, where alkali treatment significantly enhanced interfacial adhesion. Overall, SEM observations support the mechanical test results for ATAF35/UP, ATAF65/UP, and ATAF95/UP composites, confirming that alkali treatment improves both the structural and mechanical performance of prepared samples.

4. CONCLUSIONS

This research focuses on assessing the effects of alkaline modification of Alfa fibers on the structural and mechanical behavior of unsaturated polyester composites. The fibers were chemically modified with NaOH solutions at concentrations of 3%, 6%, and 9% for 5 hours at 40°C. The results revealed that alkaline treatment successfully eliminated hemicellulose and lignin, as proven by FTIR spectra, and significantly enhanced the crystallinity index, which reached 60.02% in fibers treated with 9% NaOH, as revealed by XRD analysis.

Thermogravimetric analysis indicated improved thermal resistance in the treated fibers. Furthermore, mechanical characterization showed a notable enhancement in flexural performance, with a 47.90% rise in flexural strength and a 48.46% increase in flexural modulus in the composite reinforced with 9% NaOH-treated fibers. SEM imaging further confirmed improved fiber-matrix interaction, attributed to the elimination of surface contaminants after alkaline treatment.

Therefore, the findings highlight the possibility of alkali-modified Alfa fibres as effective reinforcement components in polymer composites, particularly for engineering applications requiring enhanced mechanical and thermal performance.

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