INVESTIGATION OF NANO ALUMINA AND SURFACE TREATMENT EFFECTS ON PHYSICAL AND MECHANICAL PROPERTIES OF ARECA FIBER REINFORCED EPOXY COMPOSITES

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Received September 2, 2025

This study explores the effects of nano alumina and surface treatment on the physical and mechanical performance of areca palm stem (APS) fiber reinforced epoxy composites. The composite samples were prepared using the conventional hand layup method, maintaining a constant APS fiber content of 20 wt%, while varying sodium hydroxide concentrations (0%, 3%, 6%, and 9%) and nano alumina filler loadings (0 wt%, 2.5 wt%, 5 wt%, and 7.5 wt%). This study demonstrates that incorporating alumina nanofillers and sodium hydroxide treatment of fibers can enhance the mechanical properties of APS fiber reinforced composites. The incorporation of alumina nanoparticles into treated composites enhanced elasticity, toughness, strength, ductility, and hardness as the nanofiller concentration increased up to 5 wt%. Compared to untreated composites, incorporating 6% sodium hydroxide treated areca fibers and 5 wt% nanofillers leads to a 53.60% improvement in tensile strength and a 43.05% improvement in flexural strength. The maximum impact energy (5.81 J) and hardness (59.5 H_V) are achieved at 7.5 wt% nano alumina reinforced APS fiber composites treated with 6% sodium hydroxide. Morphological analysis conducted with a scanning electron microscope revealed various defects such as fiber pull-outs, interfacial behaviour issues, internal cracks, and voids.

Keywords: areca fiber, mechanical properties, nanofiller, NaOH treatment

INTRODUCTION

Natural plant fibers are widely available across the globe and offer several advantages, including high specific strength, renewability, and biodegradability. They also require relatively low energy for processing, are cost-effective to produce, and exhibit favourable mechanical properties, making them attractive for sustainable material applications.[1] While natural fibers offer numerous advantages, they also present certain limitations, including weak interfacial adhesion with polymer matrices, high moisture absorption, and variability in mechanical properties.[2] These limitations arise primarily from the inherent hydrophilic nature of natural fibers and their poor chemical compatibility with many polymer matrices, particularly non-polar systems.

This challenge has prompted extensive research into both chemical and physical surface modification techniques to improve fiber–matrix compatibility and maximize mechanical performance. Various chemical treatments such as alkali, benzyl chloride, silane, and acetylation have been employed to modify fiber surfaces, not only by increasing surface roughness for mechanical interlocking, but also by altering the chemical functionality of the fibers to enhance interfacial bonding and wettability with the polymer matrix.[3,4] Among these, alkali treatment is widely used as it removes hemicelluloses, lignin, and surface impurities, thereby exposing reactive hydroxyl groups and increasing surface reactivity. This modification improves fiber–matrix adhesion through enhanced hydrogen bonding and better wetting, especially in polar matrices, such as epoxies and polyesters. In contrast, non-polar matrices, such as polyolefins, exhibit limited chemical affinity with natural fibers

and often require compatibilizers to achieve effective bonding. Additionally, the incorporation of particulate fillers, such as nano-alumina, can further improve interfacial compatibility by acting as a bridging medium and enhancing stress transfer across the fiber–matrix interface.[5,6] Yousif *et al*.[7] demonstrated that alkaline treatment of kenaf fibers enhanced the flexural strength by approximately 36%, compared to 20% improvement in untreated fibers through wax removal and surface roughening, which enhanced interfacial adhesion and stress transfer efficiency.

Areca fiber is extracted from the husk of *Areca catechu* (betel nut), an abundant agricultural by-product. In tropical regions like India, where betel nut cultivation is widespread, these fibers represent a substantial renewable resource with significant annual production potential.[8,9] Areca fibers demonstrate excellent mechanical properties, including high strength, environmental friendliness, and an impressive strength-to-weight ratio, while also being cost-effective, biodegradable, and non-toxic.[10] Mohanty *et al.*[11] investigated the mechanical behavior of thermoplastic composites reinforced with randomly oriented short areca sheath fibers. Their findings indicated that treating the areca fiber with benzyl chloride significantly improved its compatibility with the matrix, and an optimum fiber loading of 27 wt% led to enhanced overall composite performance. Rahman *et al*.[12] reported that alkali treatment notably improved the tensile and flexural properties of coir and betel nut fiber composites when compared to those made with untreated fibers. Yousif and Nirmal[13] observed that chemical treatment improved flexural properties by 28% and increased hardness by 6% relative to unreinforced polyester matrix.

The hybridization of natural fiber reinforced polymer composites with inorganic particulate fillers resulted in substantial enhancements in mechanical performance, physical characteristics, and wear resistance relative to traditional composite systems.[14-16] The incorporation of nanoparticles (nano alumina, carbon nanotubes, nano silica, and nano titanium dioxide) into natural fiber reinforced epoxy composites is a significant research area, aiming to combine the environmental advantages of natural fibers with the enhanced mechanical performance offered by nanoparticles.[17-20] The addition of nanoparticles (*e.g*., nano-alumina) can substantially enhance the mechanical performance of composites through improved filler-matrix interfacial bonding and more efficient stress distribution.[21] Kumar *et al*.[22] demonstrated that incorporating 3 wt% nano clay into bamboo/epoxy laminates increased their flexural and tensile strengths by 27% and 40%, respectively, compared to the pure composite. Dhanasekar *et al*. investigated the effect of nano silica particles on the density, mechanical, and tribological properties of sisal/hemp hybrid nanocomposites. Their results demonstrated that a 6 wt% nano silica content significantly enhanced tensile strength (48.13% increase) and impact strength (1.9 times higher) compared to unreinforced composites.[23] Chowdary *et al*.[24] demonstrated that incorporating nano silica enhances the tensile and flexural performance of sisal and kevlar fiber reinforced polyester composites, with maximum tensile and flexural strengths observed at a 4 wt% concentration. Patnaik *et al*.[25] studied the mechanical performance of epoxy composite reinforced with needle punch nonwoven jute fiber and nano alumina. At 5 wt% filler content, the composite exhibited a 30% increase in tensile strength and 20% increases in flexural strength compared to the untreated system.

In addition to surface roughness, the chemical compatibility between natural fibers, particulate fillers, and the polymer matrix plays a critical role in determining the overall performance of composites. Chemical treatments such as alkali (NaOH) treatment modify the fiber surface by removing hemicelluloses, lignin, and other amorphous constituents, thereby exposing a higher concentration of hydroxyl (–OH) groups on the fiber surface. This enhances wettability and promotes stronger interfacial adhesion with polar polymer matrices, such as epoxy, through hydrogen bonding and secondary interactions. The effects of nano alumina and sodium hydroxide treatments on the mechanical characteristics of areca fiber reinforced epoxy composites have not been explored in the literature. This research systematically examines the combined effects of sodium hydroxide treatment (at concentrations of 0%, 3%, 6%, and 9%) and nano-alumina reinforcement (0 wt%, 2.5 wt%, 5 wt%, and 7.5 wt%) on the physical and mechanical characteristics of epoxy based areca fiber composites. The findings will provide fundamental insights for optimizing the mechanical performance and long term durability of APS fiber reinforced epoxy composites, facilitating their adoption in demanding industrial applications.

EXPERIMENTAL

Materials

Areca fibers were selected as the reinforcement phase due to their favourable combination of sustainability, natural abundance, and intrinsic mechanical properties, including high specific strength and modulus. The Areca palm (*Areca catechu*) stems used in this study were obtained from a plantation in Chirala, Andhra Pradesh. The collected stems were subjected to a 14-day water retting treatment. Subsequently, the retted stalks were mechanically separated using a wooden mallet to extract the fibers. The extracted fibers were thoroughly rinsed under running water and subsequently air dried at room temperature for 48 hours to eliminate residual moisture.

Nano alumina particles were selected as filler material due to their well-established ability to enhance mechanical properties and improve wear resistance in polymer matrices. Nano alumina particles used in the study were sourced from the Fiber Source supplier in Chennai, India. Figure 1 shows the reinforcement materials. Table 1 presents the physical characteristics of the nano alumina filler, while Table 2 summarizes the physical and mechanical properties along with the chemical composition of the areca palm stem fibers.[26-28]

The epoxy resin matrix and hardener, procured from Sree Industrial Composite Products (Hyderabad, India), had a specified density of 1.1 g/cm³. The densities of APS fibers and nano alumina were recorded as 1.34 g/cm³ and 3.96 g/cm³, respectively.

(a) (b)

Figure 1: Reinforcement materials (a) nano alumina and (b) APS fiber

Table 1
Properties of nano alumina powder

Property	Units	Value
Particle density	(g/cm^3)	3.96
Surface area	(m^2/g)	5–20
Thermal stability	°C	> 1200°C
Thermal conductivity	W/m·K	30–35
Average particle size	nm	20–100
Crystal phase		α
Solubility in water	mg/L	0.01–0.1
Color		Off white

Table 2
Properties of areca palm stem fiber

Property	Units	Value
Density	g/cm³	1.34
Cellulose	%	65.02
Hemicelluloses	%	8.26
Lignin	%	18.62
Tensile Strength	MPa	320 – 876
Young's Modulus	GPa	42 − 48
Elongation at Failure	%	1.47–1.48

Sodium hydroxide treatment of fibers

Sodium hydroxide (NaOH) treatment was employed to modify areca fiber surfaces by enhancing their roughness and improving interfacial adhesion with the epoxy matrix. The fibers were treated with varying NaOH concentrations (0%, 3%, 6%, and 9%) to partially remove hemicellulose and lignin constituents. This chemical

treatment increased surface roughness and promoted better mechanical interlocking at the fiber-matrix interface. The surface treatment process involved immersing pre-cleaned areca fibers in an aqueous sodium hydroxide solution at room temperature (28 ± 2 °C) for 4 hours to modify the fiber surface. After treatment, the fibers were thoroughly rinsed with distilled water until a neutral pH was reached, ensuring the removal of any residual alkali. Finally, the fibers were oven-dried at 70 ± 2 °C for 24 hours to eliminate all moisture before their incorporation into composites.

Composites fabrication

The composite fabrication involved reinforcing surface modified continuous APS fibers into an epoxy matrix with nano alumina as filler. Epoxy resin and hardener were mixed in a 10:1 ratio by weight. For composites containing nano alumina, four different weight percentages (0 wt%, 2.5 wt%, 5 wt%, and 7.5 wt%) were added to the resin. This mixture underwent mechanical stirring for 5 min, followed by sonication for 30-45 min in pulse mode, ensuring homogeneous dispersion and preventing nanoparticle agglomeration. The treated APS fibers were incorporated into the epoxy matrix using the hand layup technique. Prior to fabrication, a mold release agent was applied to the mold surface. The epoxy resin and layered APS fibers were then alternately arranged, ensuring thorough fiber wetting and eliminating air bubbles through controlled rolling process. The composites were then cured under light pressure of 0.5 MPa at room temperature (28 ± 3 °C) for 24 hours, followed by post-curing for physical and mechanical tests. Figure 2 displays the fabricated composite specimens for subsequent physical and mechanical testing. The process was systematically repeated for different sodium hydroxide (NaOH) treatment concentrations and nano alumina filler loadings. Table 3 summarizes the complete experimental design, including all parameter variations and composite compositions.

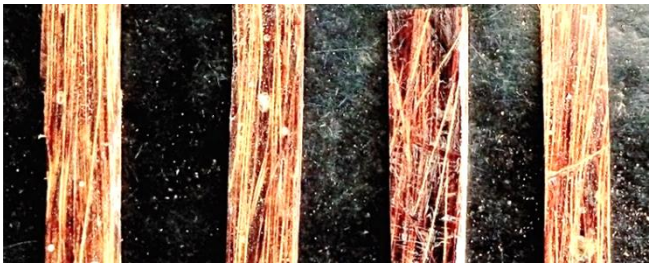


Figure 2: Fabricated composite specimens

Table 3
Experimental parameters and their compositions

Sample	NaOH concentrations	Areca fiber (wt%)	Nano alumina (wt%)	Total reinforcement	Wt% of matrix
C1	0	20	0	20	80
C2			2.5	22.5	77.5
C3			5	25	75
C4			7.5	27.5	72.5
C5	3	20	0	20	80
C6			2.5	22.5	77.5
C7			5	25	75
C8			7.5	27.5	72.5
C9	6	20	0	20	80
C10			2.5	22.5	77.5
C11			5	25	75
C12			7.5	27.5	72.5
C13	9	20	0	20	80
C14			2.5	22.5	77.5
C15			5	25	75
C16			7.5	27.5	72.5

Physical and mechanical testing

The developed APS fibers reinforced epoxy composites were subjected to extensive physical and mechanical characterization to evaluate their performance properties. FTIR spectroscopy was employed to characterize chemical composition alterations and surface functional group modifications in both untreated and NaOH treated fibers. FTIR spectra were acquired in the 4000–400 cm^{-1} spectral range using spectroscopy to characterize functional groups and assess the effects of alkali treatment on fiber surface chemistry. FTIR spectra were

acquired using the ATR mode. To address material heterogeneity, spectra were collected from multiple locations on each sample, and representative spectra were used for analysis. All spectra were baseline-corrected and normalized with respect to a reference peak to ensure reliable comparison. The analysis provided critical evidence of chemical modification success and revealed correlations between surface chemistry and composite performance. The theoretical density of the APS fibers reinforced composites was determined using the rule of mixtures, based on the weight fractions and densities of the individual constituents, as shown in Equation (1). The composite density was measured according to ASTM D792 using the water displacement method. This calculation facilitated a comparison with the experimental density to estimate the void content in the composites.

$$\rho_{ct} = \frac{1}{(W_A/\rho_A) + (W_n/\rho_n) + (W_m/\rho_m)} \quad (1)$$

$$V_v = \frac{\rho_{ct} - \rho_{ca}}{\rho_{ct}} \quad (2)$$

The mechanical properties of the fabricated APS fiber reinforced epoxy composites were comprehensively evaluated. Tensile and flexural properties were evaluated using a tensometer testing machine. Tensile properties were characterized according to ASTM D3039-76 using rectangular specimens (153 × 12.7 × 4 mm³). Testing was conducted at a constant crosshead speed of 5 mm/min until failure, with strain measured using an extensometer to determine both tensile strength and elastic modulus. Flexural properties were determined via three-point bending tests conducted in accordance with ASTM D790-07. Rectangular specimens (125 × 12.7 × 4 mm³) were loaded at a constant crosshead speed of 5 mm/min, with flexural strength and modulus calculated from the resulting load and displacement. The impact resistance of the hybrid composite samples was evaluated using the Izod impact tester. Impact resistance was evaluated using an Izod impact tester according to ASTM D256 standards, with notched specimens (64 × 12.7 × 4 mm³). Surface hardness measurements were performed independently using a Vickers microhardness tester. A minimum of five specimens per composite configuration were tested for each mechanical property. Mean values with corresponding standard deviations are reported to ensure data reliability and enable statistical comparison. Finally, scanning electron microscopy (SEM) was used to evaluate the fracture surface morphology, which revealed various defects, such as fiber pull-outs, interfacial bonding quality, nano-alumina dispersion, internal cracks, and voids.

RESULTS AND DISCUSSION

FT-IR analysis

FTIR analysis was carried out to examine the chemical modifications in APS fibers after NaOH treatment at different concentrations (0%, 3%, 6%, and 9%), as shown in Figure 3. Prior to analysis, samples were dried to minimize the influence of absorbed moisture. The broad band observed around 3445 cm^{-1} is attributed to O–H stretching vibrations of hydroxyl groups in cellulose and hemicelluloses, although minor contributions from residual moisture cannot be completely excluded. A gradual decrease in its intensity with increasing NaOH concentration indicates partial removal of hemicelluloses and surface impurities, leading to increased exposure of cellulose. The peak around 2890 cm^{-1} corresponds to C–H stretching vibrations of aliphatic groups present in cellulose and lignin. Changes in this region with alkali treatment are associated with alterations in the fiber composition; however, this band alone cannot be considered a definitive indicator of delignification. More reliable evidence is observed at 1632 cm^{-1}, assigned to C=O stretching of lignin and other carbonyl-containing components, where a reduction in intensity suggests partial removal of lignin during treatment. It is important to note that FTIR (ATR mode) predominantly reflects surface chemical changes; therefore, the observed modifications are mainly associated with the fiber surface rather than the bulk structure. The removal of non-cellulosic components and the increased availability of surface hydroxyl groups enhance fiber wettability and promote hydrogen bonding with the polar epoxy matrix. This improved chemical affinity, together with better surface characteristics, contributes to enhanced interfacial bonding in the composites. Overall, the FTIR results indicate that alkali treatment effectively modifies the surface chemistry of APS fibers.

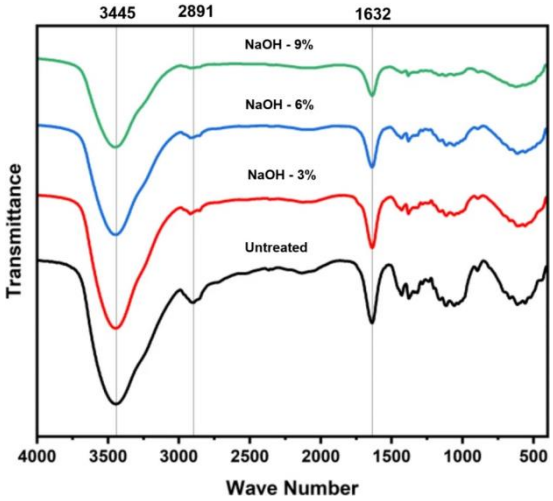


Figure 3: FTIR spectra of untreated and treated APS fibers

Table 4
Densities and void percentage of the nano alumina filled APS fiber composites

Sample	Theoretical density (g/cm^3)	Actual density (g/cm^3)	Void percentage (%)
C1	1.141	1.067	6.481
C2	1.163	1.097	5.687
C3	1.185	1.123	5.284
C4	1.209	1.141	5.578
C5	1.141	1.076	5.672
C6	1.163	1.104	5.059
C7	1.185	1.130	4.643
C8	1.209	1.149	4.967
C9	1.141	1.082	5.195
C10	1.163	1.108	4.709
C11	1.185	1.134	4.327
C12	1.209	1.153	4.586
C13	1.141	1.078	5.467
C14	1.163	1.101	5.294
C15	1.185	1.127	4.951
C16	1.209	1.146	5.174

Density and void content

Table 4 presents the density and void content analysis of APS fiber reinforced epoxy composites, evaluating the effects of different surface treatments and varying weight percentages of nano alumina filler. Among all tested compositions, the untreated APS fiber/epoxy composite containing 7.5 wt% nano alumina filler demonstrated the maximum experimental density of 1.141 g/cm^3. With sodium hydroxide (NaOH) treatment, the composite density progressively increased to 1.149 g/cm^3 (3 wt% NaOH), 1.153 g/cm^3 (6 wt% NaOH), and 1.146 g/cm^3 (9 wt% NaOH), indicating a positive correlation between alkali concentration and densification. This observation indicates that the NaOH treatment, by enhancing surface roughness and wettability, promotes superior fiber-matrix adhesion, thereby resulting in reduced void content and improved composite compaction. The 9% NaOH treatment resulted in a noticeable decrease in density, likely due to over-treatment damaging the cellulose crystalline structure and reduced fiber flexibility, which hindered proper matrix wetting and led to increased void formation.[29] The void content, a critical factor influencing the mechanical performance of composites, consistently decreased as the concentration of NaOH increased, until an optimal treatment level was achieved. Composite specimens incorporating 5 wt% nano alumina, under varying treatment concentrations, consistently exhibited reduced void percentages of 24.75%, 22.76%, 22.11%, and 21.63%, respectively, in comparison to unfilled composites. This observation directly indicates an improvement in fiber-matrix interfacial adhesion, attributable to the alkali treatment's

efficacy in eliminating surface impurities and augmenting fiber roughness. The observed decrease in void content with increasing nanofiller concentration is consistent with established scientific findings. This effect is mainly attributed to the high surface area-to-volume ratio of nanoparticles, which facilitates improved void filling and results in a denser, more compact composite matrix.[30] At higher void content, typically resulting from poor impregnation, the mechanical properties of composites can be severely compromised. These voids act as stress concentration points and reduce the material's ability to effectively transfer loads.

Tensile properties

The influence of NaOH treatment on the tensile strength and modulus of APS fiber reinforced epoxy composites is shown in Figures 4 and 5, comparing untreated and chemically treated fibers across nano alumina filler content of 0%, 2.5%, 5%, and 7.5%. The tensile strength of APS fiber reinforced epoxy composites was significantly influenced by the nano alumina filler content and chemical treatments applied to the APS fibers. The tensile properties of the APS fiber reinforced composite specimens demonstrate an enhancement with increasing nano alumina content, reaching an optimal concentration at 5 wt%, beyond which a reduction in properties is observed at 7.5 wt% filler loading. The integration of nano alumina into APS fiber reinforced epoxy composites has been shown to substantially reduce void formation, consequently leading to an enhancement of their tensile properties.[31] At this optimal 5 wt% nano alumina concentration, the peak tensile strength values recorded were 84.54 MPa for untreated composites, 90.42 MPa for 3% NaOH treated composites, 97.63 MPa for 6% NaOH-treated composites, and 92.59 MPa for 9% NaOH treated composites. Compared to unfilled and untreated composites, the addition of 5 wt% nano alumina led to tensile strength enhancements of 33.01%, 42.25%, 53.60%, and 45.67% for composites with untreated fibers, and those treated with 3%, 6%, and 9% NaOH, respectively.

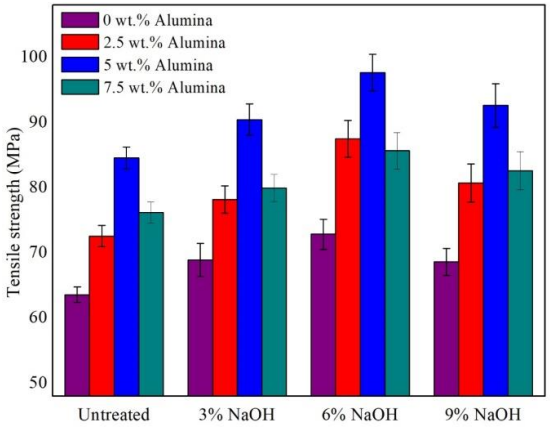


Figure 4: Impact of nanofiller and surface treatment on tensile strength of composite

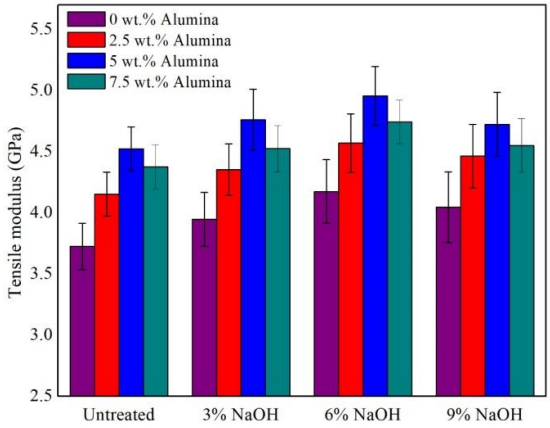


Figure 5: Impact of nanofiller and surface treatment on tensile modulus of composites

By modifying the fiber-matrix interface, surface treatments on areca fibers strengthen adhesion and optimize stress transfer, resulting in enhanced ductility and overall mechanical performance. The 6% NaOH-treated APS fiber composites exhibited optimal tensile modulus (4.956 GPa) at 5 wt% filler loading. This gradually decreased with other loadings: 4.743 GPa (7.5 wt%), 4.570 GPa (2.5 wt%), and 4.174 GPa (0 wt%). The results revealed significant differences in tensile properties between untreated and NaOH treated APS fiber composites, with the extent of improvement varying systematically with NaOH concentration (3%, 6%, and 9%). The study observed a decreasing trend in the tensile properties of APS fiber-reinforced epoxy composites at a 9% NaOH concentration. This decline is attributed to the fact that higher NaOH concentrations remove larger amounts of lignin, pectin, and other amorphous components. Beyond an optimal concentration, such aggressive chemical treatment can degrade the cellulose structure itself, compromising fiber integrity.[32] This deterioration ultimately weakens the intrinsic tensile strength of the individual fibers, resulting in a direct decline in the composite's overall tensile properties.

Flexural properties

The flexural properties of APS fiber reinforced epoxy composites were evaluated as a function of surface treatment and nano alumina filler content (0-7.5 wt%), with the results presented in Figures 6 and 7. Optimal filler content was achieved with 5 wt% nano alumina, leading to the highest flexural strength and modulus values across all fiber treatments. The composites with 5 wt% nano alumina exhibited the highest the flexural strengths observed were 128 ± 7.8 MPa for untreated APS fiber, 137 ± 5.4 MPa for 3% NaOH treated, 146 ± 5.2 MPa for 6% NaOH treated, and 141 ± 4.9 MPa for 9% NaOH treated composites. At a 7.5 wt% nano alumina loading, particle agglomeration becomes prominent, leading to weakened interfacial bonding, non-uniform dispersion, and increased void formation. These factors collectively contribute to the observed reduction in flexural properties compared to composites with lower filler concentrations. Compared to untreated and unfilled composites, the incorporation of 5 wt% nano alumina resulted in flexural strength improvements of 26.02%, 34.61%, 43.05%, and 38.47% for the untreated, 3% NaOH treated, 6% NaOH treated, and 9% NaOH treated composites, respectively.

The 5 wt% of nano alumina filled APS fiber composites showing maximum flexural modulus values of about 6.563 GPa, 6.934 GPa, 7.496 GPa and 7.196 GPa for untreated, 3%NaOH, 6%NaOH, and 9%NaOH treated composites, respectively. Among these, the 6% NaOH treated composites showed the most significant improvement, demonstrating a 13.56% and 14.21% increase in flexural strength and modulus compared to their untreated composites. The results suggest the 6% NaOH treatment is an optimal balance between surface modification and fiber integrity. However, the 9% NaOH treated composites exhibited reduced flexural properties due to excessive delignification and potential fiber degradation at higher alkali concentrations. The optimal reinforcement observed at a 5 wt% nano alumina loading is consistent with previously reported findings for similar nanofiller reinforced polymer composites. This alignment indicates a common threshold for effective nanoparticle dispersion, beyond which agglomeration may occur, leading to a decline in composite properties.

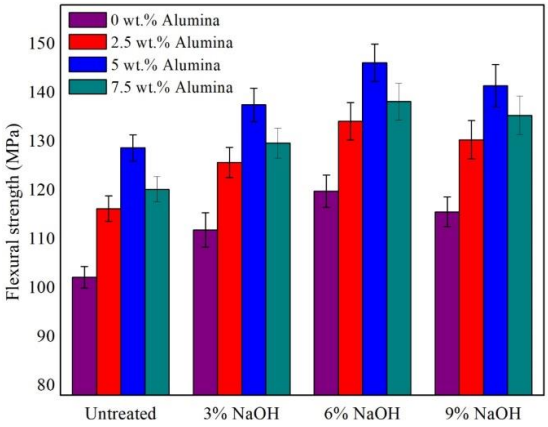


Figure 6: Effect of nanofiller and surface treatment on flexural strength of composites

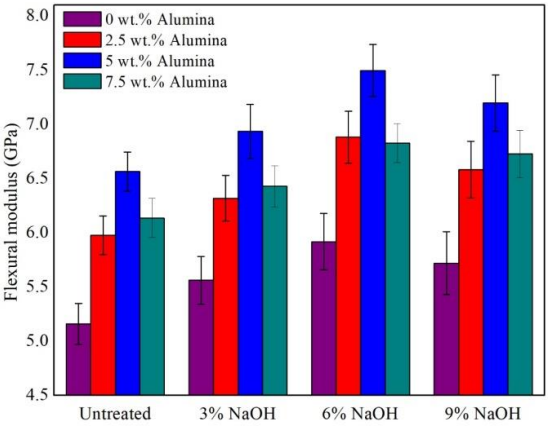


Figure 7: Effect of nanofiller and surface treatment on flexural modulus of composites

Impact energy

Figure 8 displays the impact energy results for untreated and chemically treated APS fiber composites at varying nano-alumina filler concentrations (0%, 2.5%, 5%, and 7.5%). Among all tested compositions, the APS fiber composites containing 7.5 wt% nano alumina demonstrated superior impact energy. This improvement is attributed to the nanoparticles' ability to deflect and absorb crack propagation, thereby increasing the material's resistance to sudden impacts.[28] At 7.5 wt% nano alumina loading, the composites exhibited maximum impact energies of 5.32 J (untreated), 5.68 J (3% NaOH treated), 5.81 J (6% NaOH treated), and 5.64 J (9% NaOH treated), demonstrating a 6.8% improvement for alkali-treated specimens compared to untreated composites. Composites incorporating 6% NaOH treated fibers demonstrated a marked increase in impact strength (5.81 kJ/m²), substantially outperforming both untreated fibers and those treated with lower NaOH

concentrations. This observation is consistent with prior research demonstrating that optimal treatment improves toughness, while excessive treatment causes fiber degradation.[30]

Surface hardness

Vickers microhardness testing was utilized to evaluate the surface hardness of the APS fiber reinforced composites, offering quantitative insight into their resistance to plastic deformation under standardized indentation loads. Figure 9 presents a comparative analysis of surface hardness across various NaOH treatment conditions, evaluated for both unfilled composites and those reinforced with nano alumina. The continuous increase in surface hardness observed up to 7.5 wt% nano alumina suggests that this filler concentration offers effective reinforcement. he increased surface hardness of the composites with higher weight fractions is primarily due to the inherent hardness of the alumina nanoparticles. These nanoparticles act as effective stiffening agents within the polymer matrix by restricting the mobility of polymer chains, consequently enhancing the material's resistance to surface penetration. Surface hardness measurements revealed maximum values of 55.2 Hv (untreated), 57.4 Hv (3% NaOH), 59.5 Hv (6% NaOH), and 58.7 Hv (9% NaOH) for composites containing 7.5 wt% nano alumina. The 6% NaOH treated composite demonstrated superior hardness, showing improvements of 7.98% over untreated, 3.65% over 3% NaOH treated, and 1.36% over 9% NaOH treated specimens. The 6% NaOH treated composites with 7.5 wt% nano alumina exhibited superior surface hardness (59.5 Hv), highlighting a synergistic effect between the optimized fiber surface modification and nanoparticle reinforcement.

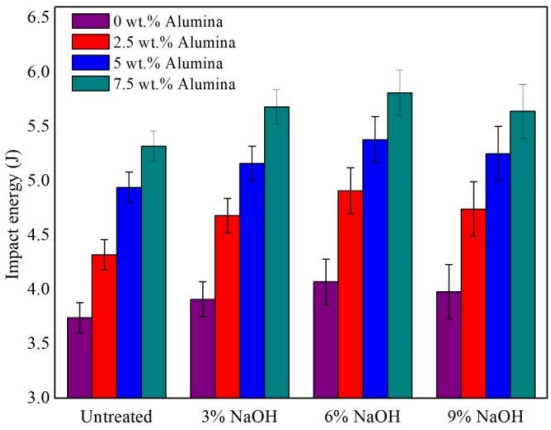


Figure 8: Effect of nanofiller and surface treatment on impact energy of composites

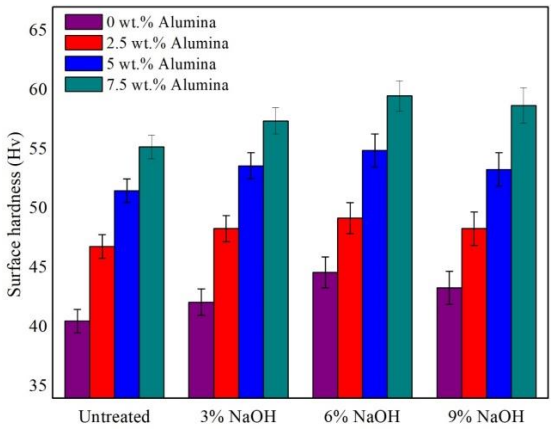
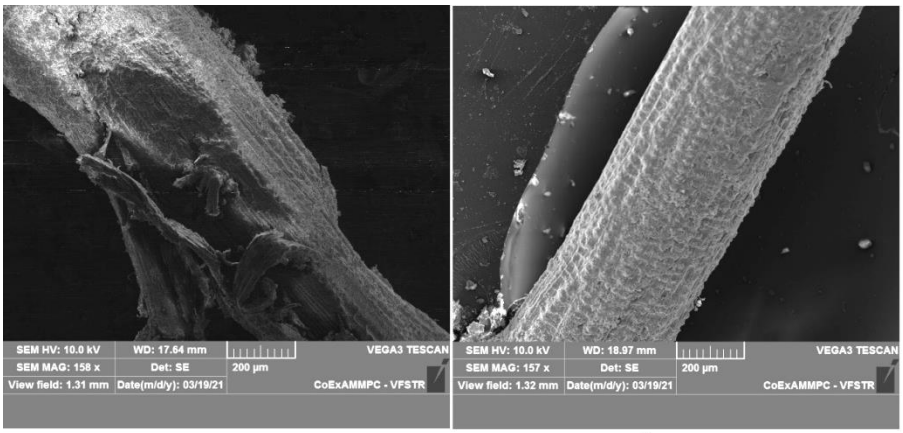


Figure 9: Effect nanofiller and surface treatment on hardness of composites

Surface morphology

Scanning electron microscopy (SEM) analysis was performed to examine the surface morphology of untreated and NaOH-treated APS fiber, as well as the fracture surfaces of the resulting APS fiber reinforced epoxy composites, to evaluate the effects of nano alumina and surface treatment on fiber microstructure and composite failure mechanisms. Figure 10(a) displays the SEM images of untreated APS fiber, revealing a relatively rough surface with visible waxy layers, impurities, and amorphous constituents, such as hemicelluloses and lignin. These surface characteristics can adversely affect reinforcement efficiency and matrix adhesion. Figure 10(b) presents the SEM micrographs of NaOH-treated APS fiber, showing significant modifications in the fiber surface morphology. The alkali treatment effectively removes pectin, impurities, and hemicelluloses, resulting in a cleaner and more fibrillated surface. Although the treated fibers exhibit increased surface roughness, it is important to note that the roughness features are predominantly at a finer scale compared to the untreated fibers. Such finer-scale roughness may be relatively less effective in promoting mechanical interlocking with the epoxy matrix. However, the overall interfacial adhesion is still improved due to the removal of surface impurities and enhanced exposure of reactive cellulosic sites, which increase wettability and promote better physicochemical bonding between the fiber and matrix. Therefore, the improvement in interfacial properties can be attributed to a combined effect of surface cleaning, fibrillation, and enhanced chemical compatibility rather than surface roughness alone.



(a) (b)

Figure 10: SEM images of APS fiber (a) untreated, (b) 6% NaOH treated

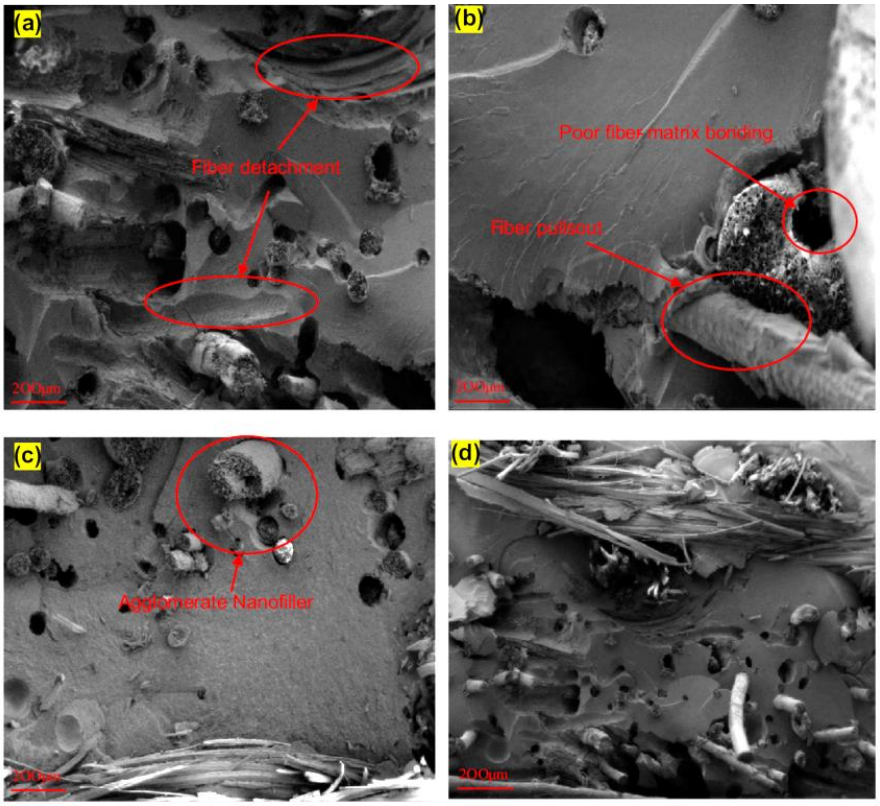


Figure 11: SEM spectrographs of the tensile fracture surface of composites with (a) 0% nano filler, (b) 2.5% nano filler, (c) 5% nano filler and (d) 7.5% nano filler

Figure 11 shows the SEM images illustrating the dispersion of nano alumina particles within the epoxy matrix. Figure 11(b) and (c) present relatively uniform distribution of nanoparticles at 5 wt% nano alumina concentration, which contributes to achieving optimal mechanical properties. In contrast, Figure 11(d) corresponds to the 7.5 wt% nano alumina sample, where noticeable agglomeration is observed. This clustering introduces defects, such as voids and particle-rich regions, reduces the effective filler–matrix interfacial area, and acts as stress concentration sites that can initiate microcracks. Furthermore, the SEM micrographs clearly reveal the presence of defects including fiber pull-out, matrix cracking, and interfacial debonding, which are detrimental to the overall mechanical performance of the composites. These defects are mainly attributed to inadequate dispersion at higher nanoparticle loadings and processing-induced void formation. The presence of such imperfections weakens the interfacial bonding between fiber and matrix, thereby reducing stress transfer efficiency. The micrographs also display matrix–fiber breakage, deformation, and crack propagation, providing

direct visual evidence that supports the observed tensile properties. Overall, while improved dispersion at lower nano alumina content enhances mechanical performance, excessive loading leads to defect formation that adversely affects the composite behavior.

CONCLUSION

This study investigated the combined effects of alkali treatment and nano alumina reinforcement on APS fiber reinforced epoxy composites. Alkali treatment with 6% NaOH effectively modified the surface of APS fibers. SEM analysis revealed the removal of surface impurities and the development of a rough, fibrillated texture, which significantly enhanced fiber-matrix interfacial bonding and improved stress transfer within the composite. The results reveal a compelling synergy between controlled alkali treatment and nano alumina reinforcement, where their combined effect yields mechanical property improvements exceeding the sum of individual modifications. The composite containing 5 wt% nano alumina with 6% NaOH treated fibers demonstrated optimal mechanical performance, exhibiting 53.6% higher tensile strength and 43.05% greater flexural strength compared to untreated composites. At a higher loading of 7.5 wt% nano alumina (with 6% NaOH treated fibers), the composite achieved peak impact resistance and surface hardness properties. Excessive NaOH treatment at 9% led to degradation of the fiber structure, while nano-alumina concentrations above 5 wt% caused particle agglomeration, both contributing to a marked reduction in the composite's tensile and flexural properties.

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