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Physical and mechanical performance of surface treated areca fiber and nano alumina reinforced epoxy composites

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ABSTRACT

Areca palm stem fiber reinforced epoxy composites modified with alkaline surface treatment and nano alumina were investigated to evaluate improvements in physical and mechanical performance. Composite laminates were fabricated by the hand layup method with 20 wt. % areca palm stem fiber, while sodium hydroxide treatment levels (0, 3, 6, and 9 %) and nano alumina loadings (0, 2.5, 5, and 7.5 wt. %) were varied. The fabricated composite specimens containing 5 wt. % nano alumina, subjected to varying treatment concentrations, consistently exhibited reduced void content and enhanced density. Mechanical characterization showed a significant enhancement in strength and toughness due to the combined effects of fiber surface activation and nanoparticle reinforcement. A 6 % NaOH treatment with 5 wt. % nano alumina resulted in a 53.60 % increase in tensile strength and a 43.05 % increase in flexural strength compared to untreated composites. Maximum impact energy (5.81 J) and hardness (59.5 HV) were obtained at 7.5 wt. % nano alumina with 6 % NaOH-treated fibers. Scanning electron microscopy revealed reduced fiber pull-out and improved interfacial bonding, though voids and microcracks were observed at higher filler loadings.

KEYWORDS

areca fiber • mechanical properties • nanofiller • NaOH treatment • scanning electron microscope

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Introduction

Natural plant fibers are abundantly available worldwide and offer numerous advantages, including high specific strength, renewability, excellent biodegradability, reduced energy processing requirements, low production costs, and favourable mechanical characteristics [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 [2,3]. These limitations pose a significant challenge, prompted extensive research into chemical and physical surface modification techniques to improve compatibility and maximize mechanical properties. Various chemical treatments such as alkali, benzyl chloride, silane, and acetylation have been employed to

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modify fiber surfaces, mechanical interlocking and enhancing wettability with the polymer matrix [4,5]. Among chemical treatments, alkali treatment process enhances fiber surface roughness and removes hemicellulose and lignin, which in turn increases the number of reactive hydroxyl groups, thereby improving interfacial bonding with the polymer matrix [6]. Yousif et al. [7] demonstrated that alkaline treatment of kenaf fibers enhance the flexural strength by approximately 36 %, compared to 20 % improvement in untreated fibers through wax removal and surface roughening, which enhance interfacial adhesion and stress transfer efficiency.

Areca fiber is extracted from the husk of Areca catechu (betel nut), an abundant agricultural byproduct. 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 exhibit excellent mechanical properties, including high strength, environmentally friendly, and an impressive strength-to-weight ratio, while also being cost-effective, biodegradable, and non-toxic [10]. Nayak and Mohanty [11] investigated the mechanical, 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 in comparison with untreated counterparts. 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 [14–17]. The incorporation of nanoparticles such as nano silica [18], nano alumina [19], carbon nanotubes [20], and nano titanium dioxide [21] into natural fiber reinforced epoxy composites is a significant research focus, aiming to combine the environmental advantages of natural fibers with the enhanced mechanical performance offered by nanoparticles. 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 [22]. Kumar et al. [23] 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 [24]. Chowdary et al. [25] 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. [26] 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 composite.

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, 2.5, 5, 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.

Materials and Methods

Materials

Areca fibers were selected as the reinforcement phase because they are derived from renewable biological sources, are naturally abundant, and intrinsic mechanical properties such as high specific strength and modulus. The Areca palm (Areca catechu) stems used in this study were obtained from a plantation in Chirala, Andhra Pradesh, India. 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 h to eliminate residual moisture. Nano alumina particles were selected as the 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 a supplier Fiber Source 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 [8,27,28]. 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 and 3.96 g/cm³, respectively.

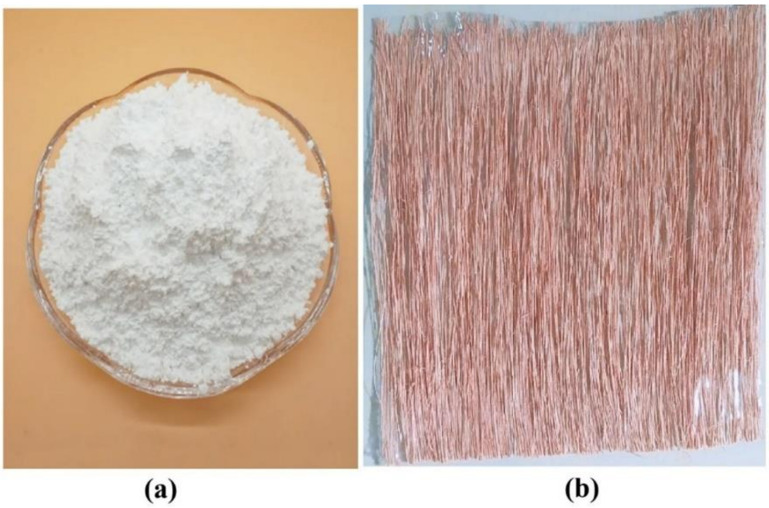

Fig. 1. Reinforcement material (a) nano alumina and (b) APS fiber

Table 1. Properties of nano alumina powder

Property	Units	Value
Particle density	(g/cm^3)	3.96
Surface area	(m^2/g)	5.00–20.00
Thermal stability	°C	>1200
Thermal conductivity	W/m·K	30.00–35.00
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/cm^3	1.34
Cellulose	%	65.02
Hemicelluloses	%	8.26
Lignin	%	18.62
Tensile strength	MPa	320.00–876.00
Young's modulus	GPa	42.00–48.00
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 underwent alkali treatment using varying concentrations of sodium hydroxide (NaOH) (0, 3, 6, and 9 %). This alkali treatment was carried out to partially remove hemicellulose and lignin from the fiber surface. As a result, the surface roughness increased, which improved 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 h 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 h 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 a filler. Epoxy resin and hardener were mixed in a 10:1 ratio by weight. For composites containing nano alumina, the specified weight percentages of nanoparticles (2.5, 5, and 7.5 wt. %) were added to the resin, and the resulting resin–nanoparticle mixture was subjected to mechanical stirring for 5 min, followed by sonication for 30–45 min in pulse mode to ensure homogeneous dispersion and prevent 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 h, 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.

Fig. 2. Fabricated composite specimens

Table 3. Experimental parameters and their compositions

Sample	NaOH concentrations	Areca (wt. %)	Nano alumina (wt. %)	Total reinforcement	Matrix (wt. %)
C1	0	20	0	20.0	80.0
C2			2.5	22.5	77.5
C3			5.0	25.0	75.0
C4			7.5	27.5	72.5
C5	3	20	0	20.0	80.0
C6			2.5	22.5	77.5
C7			5.0	25.0	75.0
C8			7.5	27.5	72.5
C9	6	20	0	20.0	80.0
C10			2.5	22.5	77.5
C11			5.0	25.0	75.0
C12			7.5	27.5	72.5
C13	9	20	0	20.0	80.0
C14			2.5	22.5	77.5
C15			5.0	25.0	75.0
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

(Fourier transform infrared) 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 [specify instrument model/model number if available] spectroscopy to characterize functional groups and assess the effects of alkali treatment on fiber surface chemistry. The analysis provided critical evidence of chemical modification success and revealed correlations between surface chemistry and composite performance. The theoretical density ρ_{ct} 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 Eq. (1). The composite density was measured according to ASTM D792 using the water displacement method. The calculation of theoretical density facilitated a comparison with the experimental density ρ_{ca} to estimate the void content V_v in the composites as shown in Eq. (2):

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

$$V_v = \frac{\rho_{ct} - \rho_{ca}}{\rho_{ct}}, \tag{2}$$

where W_A, W_n, and W_m are weight fractions of areca, nano alumina, and matrix, respectively; ρ_A, ρ_{n}, ρ_m is density of areca, nano alumina, and matrix, respectively.

The mechanical properties of the fabricated APS fibers 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 mm^3). 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 mm^3) 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 mm^3). 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

Fourier transform infrared spectroscopy analysis

FTIR spectroscopy analysis was performed to investigate the chemical modifications in APS fibers following NaOH treatments at varying concentrations (0, 3, 6, and 9 %). Figure 3 displays the FTIR spectra, where characteristic absorption peaks corresponding to specific functional groups in the fiber composition are clearly identified. The broad absorption peak observed around 3445 cm^{-1} is attributed to the O-H stretching vibrations, which correspond to the hydroxyl groups present in cellulose and hemicellulose. With increasing NaOH concentration, the intensity of these peak decreases, indicating the partial removal of hemicellulose and the exposure of cellulose components. The peak at 2891 cm^{-1} corresponds to C-H stretching vibrations, primarily from lignin and cellulose. A reduction in peak intensity with higher NaOH concentrations suggests effective delignification of the fibers. Another significant peak is observed at 1632 cm^{-1}, corresponding to the C=O stretching vibrations associated with lignin and other carbonyl-containing compounds. The decrease in intensity at this peak with increasing NaOH treatment concentration confirms the removal of lignin content, enhancing the fiber-matrix bonding potential. The differences in the FTIR spectra between untreated and treated fibers suggest that alkali treatment effectively modifies the fiber surface chemistry by removing impurities and enhancing the availability of reactive hydroxyl groups. This modification leads to improved interfacial bonding between the fibers and epoxy matrix, contributing to enhanced mechanical properties of the composites. Overall, the FTIR analysis confirms that alkali treatment positively affects the chemical structure of APS fibers.

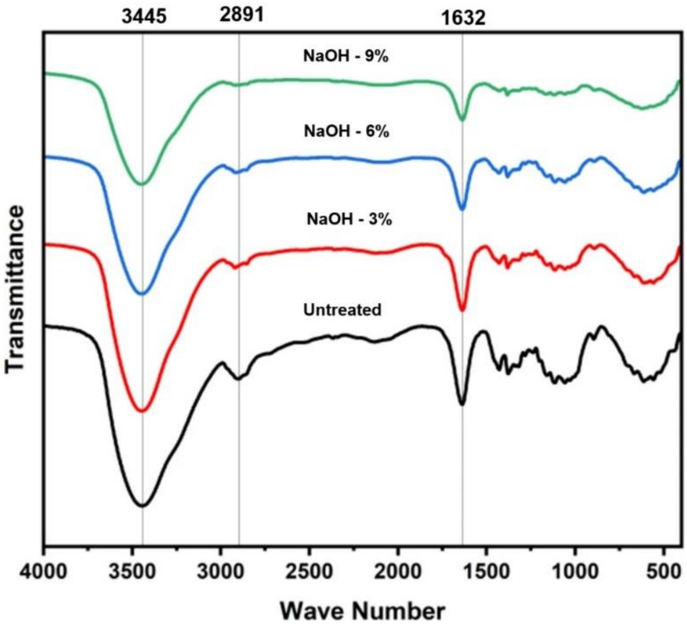

Fig. 3. FTIR spectra of untreated and treated APS fibers

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 had the maximum experimental density 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 increase in density suggests that NaOH treatment, by enhancing surface roughness and wettability, promotes superior fiber–matrix adhesion, thereby reducing void content and improving 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 [24]. 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 reduction in void content 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 [29]. 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.

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

Tensile properties

The influence of NaOH treatment on the tensile strength and modulus of APS fiber reinforced epoxy composites is shown in Figs. 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 composites increase with nano alumina content, reaching an optimum at 5 wt. %, after which the properties decrease 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. 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.

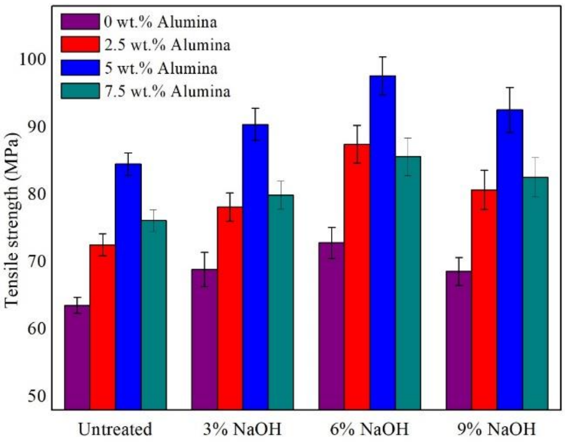

Fig. 4. Impact of nanofiller and surface treatment on tensile strength of composite

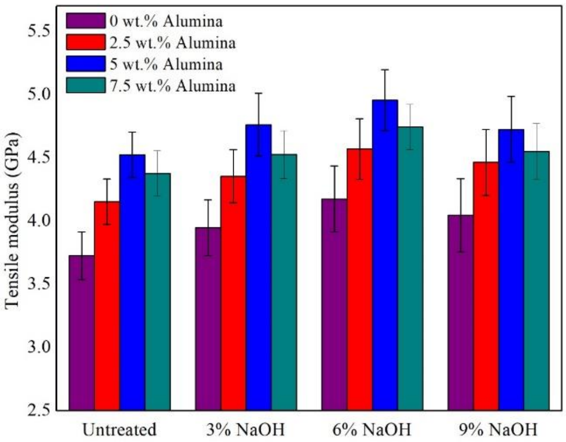

Fig. 5. Impact of nanofiller and surface treatment on tensile modulus of composites

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 tensile modulus value gradually decreased with other loadings: 4.743 GPa (7.5 wt. %), 4.57 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 reduction in tensile strength and tensile modulus of APS fiber reinforced epoxy composites at a 9 % NaOH concentration. This reduction is attributed to the removal of larger amounts of lignin, pectin, and other amorphous components at higher NaOH levels. Beyond an optimal concentration, such aggressive chemical treatment can degrade the cellulose structure itself, compromising fiber integrity [30]. This structural damage 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 Figs. 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.

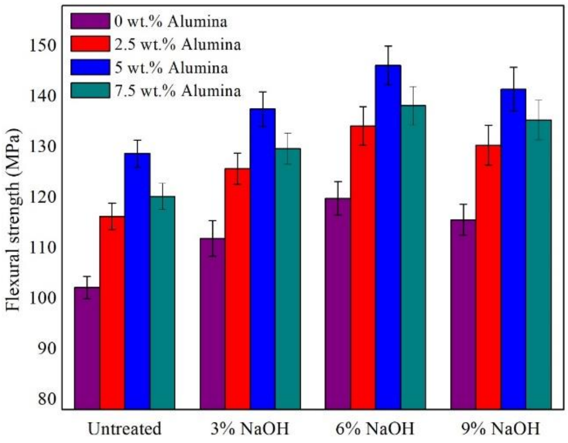

Fig. 6. Effect of nanofiller and surface treatment on flexural strength of composites

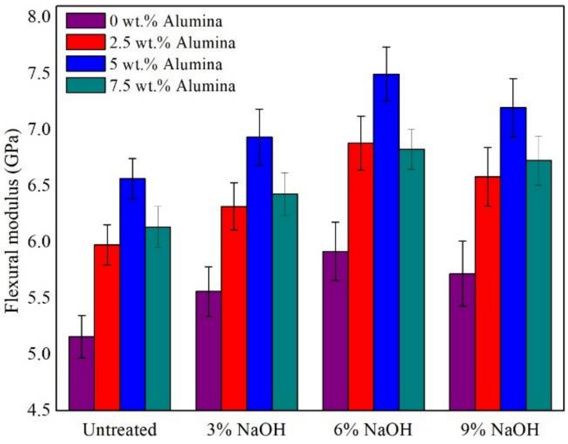

Fig. 7. Effect of nanofiller and surface treatment on flexural modulus of composites

The 5 wt. % of nano alumina filled APS fiber composites showing maximum flexural modulus values of about 6.563, 6.934, 7.496 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, with a 13.56 and 14.21 % increase in flexural strength and modulus, respectively, compared to the 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 [25,31]. This consistency suggests a common threshold for effective nanoparticle dispersion, beyond which agglomeration may occur, leading to a decline in composite properties.

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 had the highest impact energy. This increase in impact energy is attributed to the nanoparticles' ability to deflect and absorb crack propagation, thereby increasing the material's resistance to sudden impacts [2]. At 7.5 wt. % nano alumina loading, the composites exhibited maximum impact energies of 5.32 (untreated), 5.68 (3 % NaOH treated), 5.81 (6 % NaOH treated), and 5.64 J (9 % NaOH treated), corresponding to a 6.8 % increase in impact energy for alkali-treated specimens compared to untreated composites. Composites incorporating 6 % NaOH treated fibers exhibited a marked increase in impact strength (5.81 kJ/m^2), substantially outperforming both untreated fibers and those treated with lower NaOH concentrations. The higher impact strength at this treatment level aligns with previous studies reporting that moderate alkali treatment enhances fiber–matrix bonding, whereas excessively high concentrations can degrade the fiber structure and reduce toughness [32].

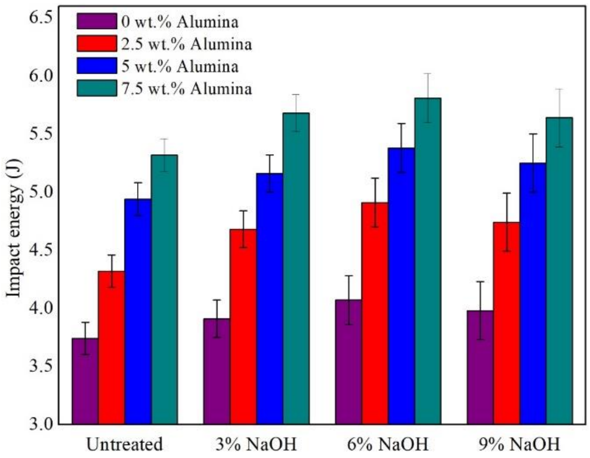

Fig. 8. Effect of nanofiller and surface treatment on impact energy of composites

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. Surface hardness continued to increase with increasing nano alumina content, reaching its highest value at 7.5 wt. % loading, which confirms its effectiveness as a reinforcing filler. The 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 [33]. Surface hardness measurements revealed maximum values of 55.2 (untreated), 57.4 (3 % NaOH), 59.5 (6 % NaOH), and 58.7 HV (9 % NaOH) for composites containing 7.5 wt. % nano alumina. The 6 % NaOH treated composite exhibited the highest 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.

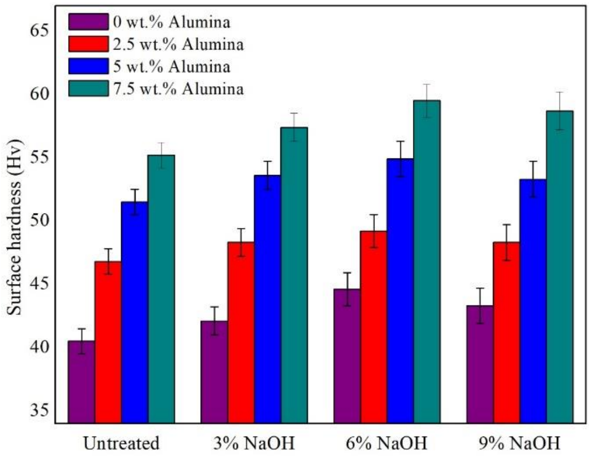

Fig. 9. Effect nanofiller and surface treatment on hardness of composites

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 hemicellulose and lignin. These surface characteristics can adversely affect reinforcement and matrix adhesion. Figure 10(b) presents the SEM micrographs of NaOH treated APS fiber, revealing significant modifications in the fiber surface morphology. The alkali treatment effectively removes pectin, impurities, and hemicelluloses, yielding a cleaner, rougher, and more fibrillated surface. This enhanced roughness and exposure of cellulosic fibrils improve fiber wettability with the epoxy matrix and facilitate mechanical interlocking, ultimately strengthening interfacial adhesion.

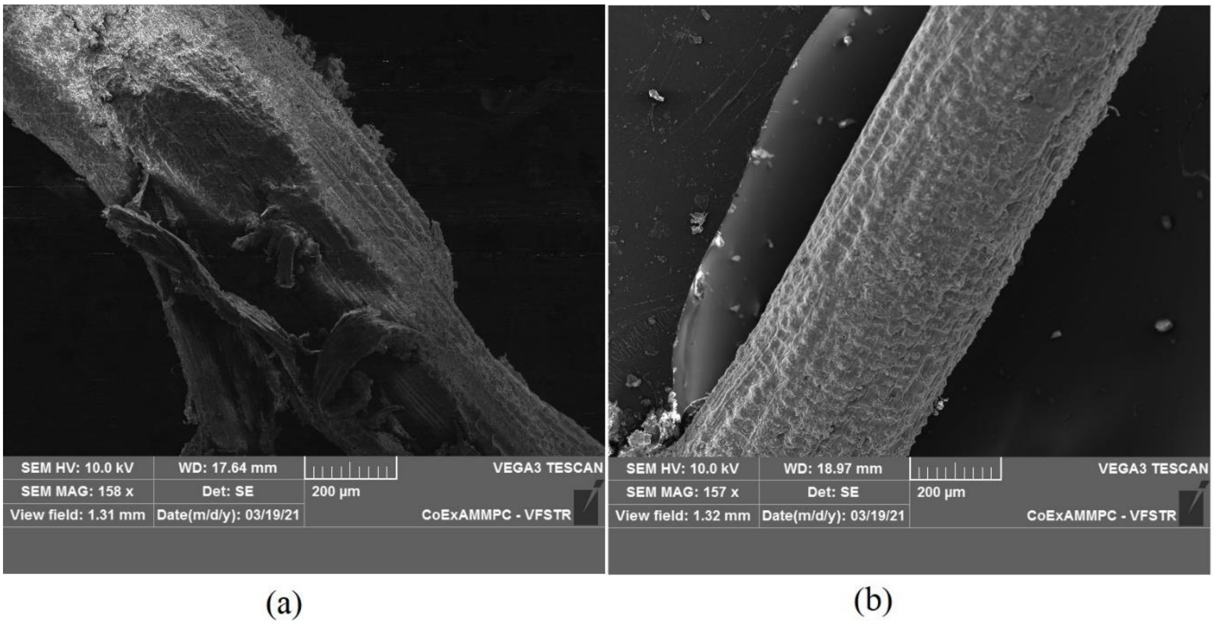

Fig. 10. SEM images of APS fiber (a) untreated, (b) 6% NaOH treated

Figure 11 shows the SEM images assessing the dispersion of nano alumina particles within the epoxy matrix. Figure 11(b,c) represents the uniform distribution of nanoparticles at 5 wt. % of nano alumina concentration for achieving optimal mechanical properties. In contrast, Fig. 11(d) shows the 7.5 wt. % nano alumina sample, where agglomeration of nanoparticles was observed. Such particle agglomeration can introduce defects, reduce the effective filler–matrix interfacial area, act as stress concentration points, and initiate microcracks. The micrographs display matrix fiber breakage, deformation, and crack propagation, providing direct visual evidence that supports the observed tensile properties.

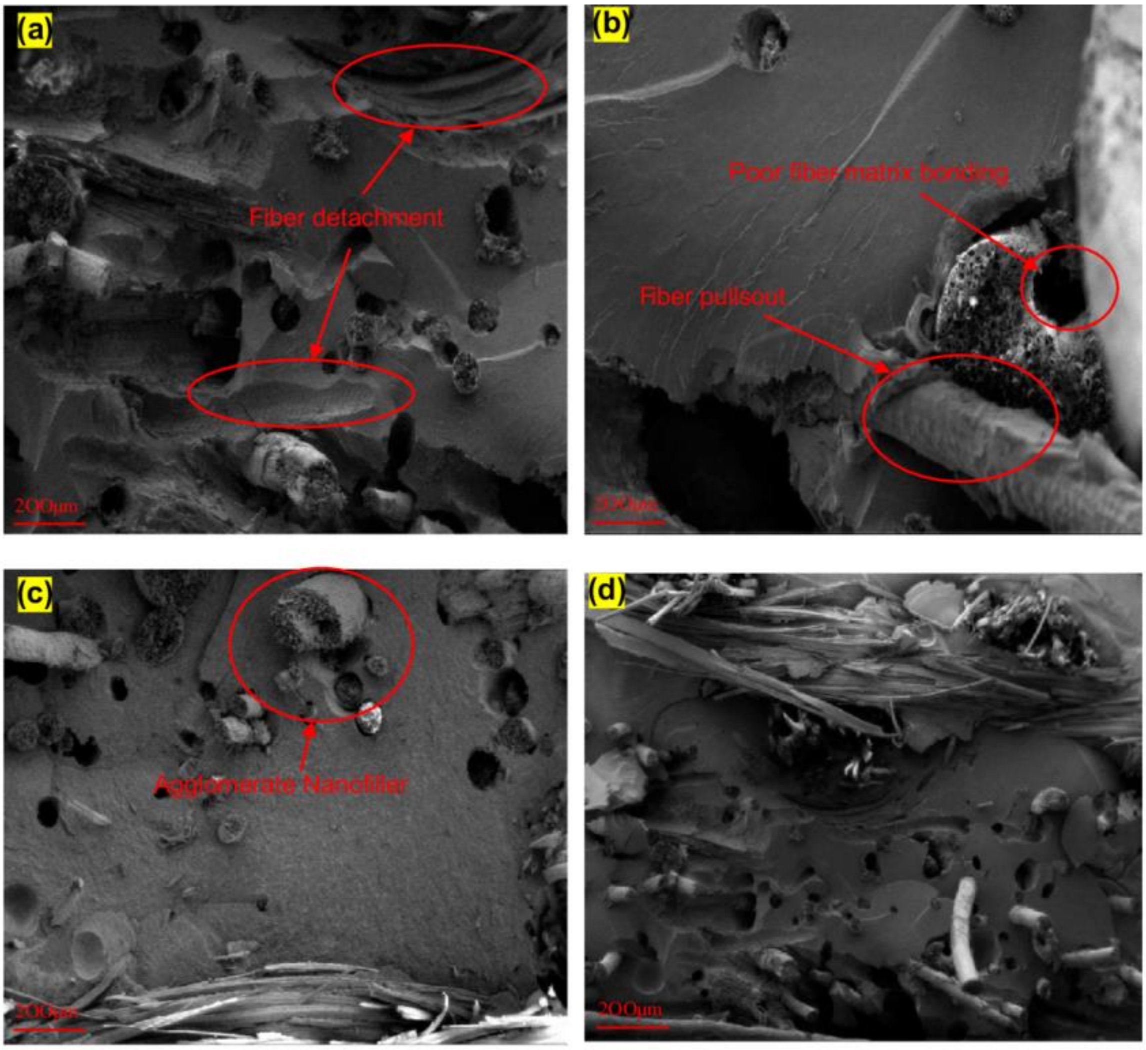

Fig. 11. SEM spectrographs of the tensile fracture surface of composites with (a) 0 % nano filler, (b) 2.5 % nano filler, (c) 5 % nano filler and (d) 7.5 % nano filler

Conclusion

This study evaluated the combined effects of alkali surface treatment and nano alumina reinforcement on the performance of APS fiber–reinforced epoxy composites. The results confirmed that both fiber surface modification and nanoparticle dispersion play critical roles in determining the mechanical response of the material. The main scientific conclusions are as follows:

1. Alkali treatment at 6 % NaOH provided the most effective fiber surface modification, producing a cleaner, fibrillated surface that enhanced fiber-matrix interfacial adhesion and stress transfer.
2. A synergistic reinforcement effect was observed between 6 % NaOH treatment and nano alumina addition, resulting in mechanical improvements greater than those achieved by either treatment alone.
3. The composite containing 5 wt. % nano alumina with 6 % NaOH-treated fibers delivered the highest overall tensile and flexural performance, with increases of 53.6 and 43.05 %, respectively, compared to the untreated composite.
4. Impact resistance and surface hardness were maximized at 7.5 wt. % nano alumina, indicating that energy absorption and indentation resistance benefit from higher filler loading, even though tensile and flexural properties begin to decline beyond 5 wt. % due to particle agglomeration.
5. Performance deterioration at 9 % NaOH and nano alumina levels above 5 wt.% confirmed two key failure mechanisms: (i) excessive alkali treatment damages the cellulose structure of fibers, and (ii) excess nanoparticles agglomerate, reducing interfacial area and inducing stress concentration.

CRediT authorship contribution statement

Siva Bhaskara Rao Devireddy: conceptualization, methods, writing – original draft; **Khandavalli Sunil Ratna Kumar**: data curation, formal analysis, writing – review & editing; **Ravi Lalitha Narayana**: investigation, resources, writing – review & editing; **Gopala Rao Thellaputta**: methodology, validation, writing – review & editing; **Vadlamudi Tara Chand**: project administration, supervision, writing – review & editing.

Conflict of interest

The authors declare that they have no conflict of interest.

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