

Influence of Chemical Treatments on Flexural Strength of Areca Fiber Reinforced Epoxy Composites

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Abstract: Natural areca fibers were treated with sodium hydroxide, potassium permanganate, benzoyl chloride and acrylic acid to improve interfacial adhesion between the areca fiber and epoxy resin and to fabricate areca fiber reinforced epoxy composites with improved mechanical properties. The untreated and chemically treated areca fiber reinforced epoxy composites were prepared with 40%, 50%, 60% and 70% fiber loadings. The investigation of flexural strength was done for all areca/epoxy composites. Untreated and chemically treated areca/epoxy composites with 60% fiber loading showed higher flexural strength values. The acrylic acid treated areca fiber reinforced epoxy composites with 60% fiber loading showed highest flexural strength values amongst all untreated and chemically treated areca/epoxy composites with same 60% fiber loading.

Keywords: Areca fibers, Chemical composition, Chemical treatments, Aspect ratio, Epoxy composites, Flexural strength

Introduction

In the recent years, there has been a growing interest for manufacturing cost effective and eco-friendly products along with superior properties for utilization in the field of industrial sector. This aroused interest for the researchers to concentrate more on natural fiber reinforced polymer composites. Amongst all natural fiber reinforcing materials areca appears to be a budding fiber, because it is inexpensive, abundantly available and a very high potential perennial crop. Karnataka is India's largest areca nut producing state which has a share of around 50% areca productions in the country. Hence, enormous quantity of unmanaged areca husk is available for further processing. The areca husk is a hard fibrous material covering the endosperm and constitutes about 60-80% of the total weight and volume of the areca fruit. The husk fiber is composed of cellulose with varying proportions

of hemicelluloses (35–64.8%) and lignin (13.0–26.0%), pectin and protopectin¹⁻³. Presently, this highly cellulosic material is being used as a fuel in areca nut process. Thus the use of this husk fiber as structural material requires a detailed study on physical, chemical and mechanical characteristics.

Few investigators studied the mechanical behavior of areca composites with 60% fiber loading and found to have a good flexural strength and adhesion tensile properties⁴⁻⁸. The tensile strength value of areca/epoxy and areca/polypropylene composites is improved when the areca fibers are treated with different concentrations of alkali⁹⁻¹⁰. Permanganate treatment of natural fibers enhanced the chemical interlocking at the interface and provided better adhesion with the matrix¹⁰⁻¹¹. Benzoylated flax fiber reinforced low density polyethylene composites reported 6% and 33% improvement on tensile strength and moisture resistance properties¹². Tensile strength and flexural strength of acrylated jute fiber reinforced epoxy-phenolic matrix composites were increased by 42.2% and 13.9% respectively¹³. Flexural strength of bamboo/epoxy composites increased with increase in fiber volume up to 30% and then decreased. Chemical treatments increased flexural strength due to removal of outer fiber surface, increase in cellulose content and interfacial adhesion. After 30%, the observed decrease is due to poor interfacial bonding resulting in spaces between fiber and matrix generating a weak structure¹⁴. Hence, surface modifications of natural fibers are one of the most important areas in the field of technical utilization of natural fiber reinforced composites in industrial sector. The most commonly used matrix material in composite fabrication is epoxy resin because of its good adhesive nature, good electrical insulating properties, good mechanical properties and good chemical and environmental resistance.

Flexural strength is the ability of the material to resist deformation under load. It is important to know the weight bearing capacity of many materials and hence based on these calculations it is possible to choose appropriate materials for industrial sector. Flexural strength plays a significant role in civil, mechanical, aerospace engineering and design.

In the present study chemical composition and aspect ratio of areca fibers were determined. Surface modification of areca fibers were done by subjecting areca fibers to chemical treatments with alkali, potassium permanganate, benzoyl chloride and acrylic acid respectively. Untreated and chemically treated areca fiber reinforced epoxy composites were prepared with 40%, 50%, 60% and 70% fiber loadings. The flexural strength of all untreated and chemically treated areca fiber reinforced epoxy composites were evaluated by following standard procedures. The aim of this research work is to study the influence of chemical treatments on flexural strength of areca fiber reinforced epoxy composites under different fiber loadings.

Experimental

Areca empty fruits were obtained from Madhu Farm House, Nilagal, Davangere, Karnataka, India. The analytical grade reagents were purchased from Qualigens Company and used as received.

Fiber extraction

The dried areca empty fruits were soaked in de-ionized water for about five days. This process is called retting; allowing the fiber to be removed from the fruit easily. The fibers were removed from the fruit and separated with a comb. After drying in the room temperature, the fibers were combed in a carding frame to further separate the fibers into an

individual state. Then a sieve was used to remove broken fibers and impurities. The resulting fibers were treated in the condition (temperature 30 °C, RH 70%) for 72 h.

Extractible content

5 g of Air dried areca fiber was placed in a Soxhlet extraction unit. A mixture of ethanol and toluene was used as solvent and the extraction process was continued for a period of five hours. After extraction, the sample was rinsed with ethanol and hot water and dried up to constant weight at the temperature of 60 °C. The extractible were calculated as a percentage of the oven dried test sample.

Lignin content

2 g of Extracted sample was placed in a flask and 15 mL of 72% sulphuric acid was added. The mixture was stirred frequently for three hours at 25 °C and 200 mL of distilled water was added to the mixture. Then the mixture was boiled for two hours and cooled. After 24 h, the lignin was transferred to the crucible and washed with hot water repeatedly until becoming acid free. The collected lignin was dried at 105 °C and cooled down in desiccators and weighed. The drying and weighing were repeated until constant weight was obtained.

Holocellulose content

3 g of Air dried areca fiber was weighed and placed in an Erlenmeyer flask and then 160 mL of distilled water, 0.5 mL of glacial acetic acid and 1.5 g of sodium chloride were added successively. The flask was placed in a water bath and heated up to 75 °C for an hour and then an additional of 0.5 mL of glacial acetic acid and 1.5 g of sodium chloride were added. The additions of acetic acid and sodium chloride were repeated two times hourly. The flask was placed in an ice bath and cooled down to below 10 °C. The holocellulose was filtered and washed with acetone, ethanol and water respectively. After washings, the sample was dried in an oven at 105 °C before weighing.

α -Cellulose content

2 g of Holocellulose was placed in a beaker and 10 mL of 17.5% sodium hydroxide solution was added. The areca fibers were stirred up by glass rod so that they could be soaked with sodium hydroxide solution vigorously. Then the sodium hydroxide solution was added to the mixture periodically (once in every five minutes) for half an hour and the mixture temperature was kept at 20 °C. About 33 mL of distilled water was added in the beaker and kept it for an hour. The holocellulose residue was filtered and transferred to the crucible and washed with 100 mL of 8.3% sodium hydroxide, 200 mL of distilled water, 15 mL of 10% acetic acid and with water, successively. The crucible with α -cellulose was dried and weighed.

Hemicelluloses content

The hemicelluloses content of areca fiber was determined by calculating the difference between holocellulose and α -cellulose¹⁵⁻¹⁷.

Alkali treatment

Areca fibers were soaked in a stainless steel vessel containing NaOH with density of 6% at the room temperature (30-32 °C) for 1 h. The alkali treated fibers were immersed in distilled water for 24 h to remove the residual NaOH. Final washing was done with distilled water containing little amount of acetic acid. Fibers were dehydrated in an oven at 70 °C for 3 h.

Potassium permanganate treatment

Areca fibers, pre-treated with 6% alkali were immersed in 0.5% KMnO₄ in acetone solution for 30 min. The fibers were then decanted and dried in air.

Benzoyl chloride treatment

During benzoylation treatment, 6% alkali pre-treatment was used to activate the hydroxyl groups of the fibers. This alkali pre-treated areca fibers were soaked in 6% NaOH and agitated with benzoyl chloride for 15 min. Then the treated areca fibers were soaked in ethanol solution for 1 h to remove benzoyl chloride that adhered to the fiber surface, washed thoroughly using distilled water and dried in air.

Acrylic acid treatment

6% Alkali treated areca fibers were immersed in 5% acrylic acid solution at 50 °C for a period of 1 h and then treated fibers were washed thoroughly using distilled water and dried in air.

Composite fabrication

The composite preparation process was performed in the following order. First, the Epoxy-556 resin of density 1.15-1.20 g/cm³ and the HY951 hardener were mixed in 10:1 ratio. One half of resin was placed inside the mixing chamber for about 1 min at 20 RPM; then fibers were added over a period of 2 min. Then, the other half of the epoxy resin was placed inside the mixing chamber and the mixing speed was increased to 30 RPM for 5 min. The resulting material was compression molded at a pressure of 0.5 MPa using a Santec compression molding press. Finally, the composites were post-cured at room temperature for 15 days. The volume fractions 40%, 50%, 60% and 70% of fiber was carefully controlled during the mixing of two ingredients. The moulds have been prepared with dimensions of 300 mm x 300 mm x 10 mm. The equation (1) was used in the composite fabrication where W_f is the weight of fiber (g), W_m is the weight of matrix (g), V_f is the fiber volume fraction (%), ρ_m is the density of matrix (g/cm³) and ρ_f is the density of fiber (g/cm³).

$$V_f = \frac{\frac{W_f}{\rho_f}}{\frac{W_f}{\rho_f} + \frac{W_m}{\rho_m}} \quad (1)$$

Flexural strength

Flexural test of areca/epoxy composites was performed using 3-point bending method on a universal testing machine (Instron 5566) according to ASTM D790-03 procedure. Specimens prepared for the flexural test were cut with help of zig saw and the specimen dimensions were 160 mm span length, 50 mm width and 10 mm thickness. The specimens were tested at a crosshead speed of 2 mm/min. At least five replicate specimens were tested and the results were presented as an average of tested specimens. The flexural tests were conducted at a standard laboratory atmosphere, 30±2 °C and 65% relative humidity (RH 65%).

Results and Discussion

Chemical composition of areca fibers

Natural lignocellulosic fibers are three-dimensional polymeric composites primarily made up of cellulose, hemicelluloses and lignin. Natural lignocellulosic fibers may differ in chemical

composition, but they have very similar properties such as thermo plasticity, moisture sorption, flammability and degradation by acids, bases and ultraviolet radiation. All of these characteristics will result in the specific end use of natural lignocellulosic fibers in polymer composite fabrication. The chemical composition of the major components of areca fiber is listed in Table 1. The experimental value of the cellulose content in the areca fiber is in agreement with the previous studies¹⁸⁻¹⁹.

Table 1. Chemical composition (wt %) of areca fiber

Reference	Cellulose	Hemi-celluloses	Lignin	Moisture content	Ash content
Experimental	55.82	34.28	6.82	1.80	1.28
Ramachandra <i>et al.</i> ¹⁸	35-64.8	29-33.1	13-26	--	1.1-2.1
Yusriaha <i>et al.</i> ¹⁹	53.20	32.98	7.20	--	1.05

Fiber size distribution

Fibers with high cellulose content, high aspect ratio *i.e.* length to diameter ratio and low microfibrillar angle *i.e.* the angle between fiber axis and the micro fibrils, possess superior mechanical properties²⁰. Fiber size is a very important parameter in natural fiber reinforced polymer composite technology. Areca fiber length and diameter distributions before compounding were depicted in the following figures. Areca fiber dimensions were obtained via optical microscopy (Olympus SZ-PT). Based on the results of Figure 1, Figure 2 and Figure 3 areca fibers can be approximated as cylindrical shape with an average length of 39 mm and with an average diameter of 0.347 mm and giving aspect ratio, L/D of 112.39²¹⁻²².

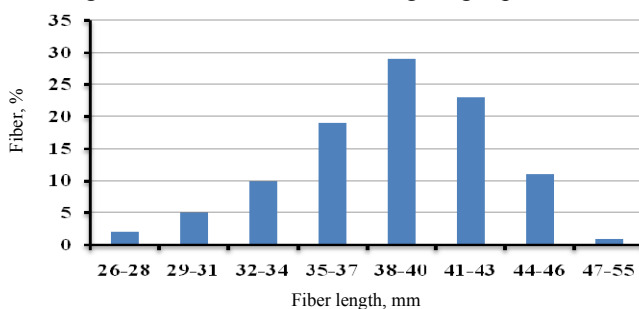


Figure 1. Areca fibers length distribution

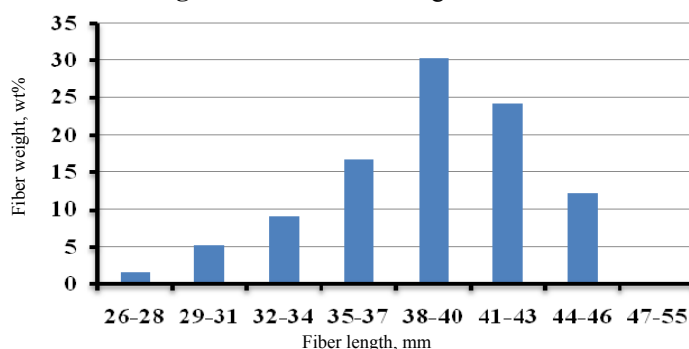


Figure 2. Areca fibers weight distribution

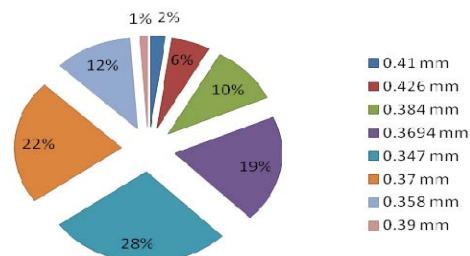
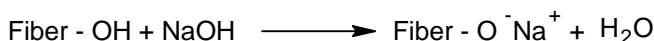


Figure 3. Areca fibers diameter distribution

Chemical treatments of areca fibers

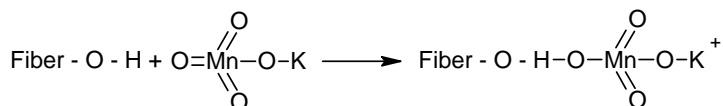
Chemical treatments facilitate efficient coupling with polymeric resin by exposing more reactive groups on the fiber surface and as a result, the interfacial properties can be improved and natural fiber reinforced polymer composites with better mechanical properties can be prepared^{3,23-31}.

Alkali treatment



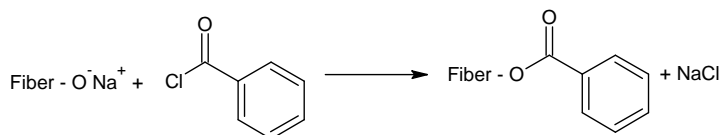
Alkali treatment of areca fibers results in the formation of Fiber-cell-O-Na groups between the cellulose molecular chains. The hydrogen bonding present in the network structure is removed and new reactive hydrogen bonds are formed between the cellular molecular chains and the natural crystalline structure of the cellulose relaxed.

Potassium permanganate treatment



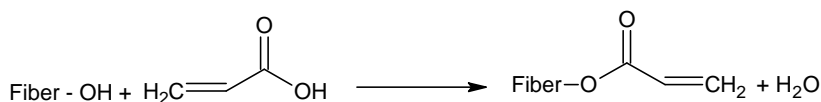
Cellulose manganate is formed by the reaction between highly reactive permanganate ions (Mn^{3+}) with cellulose hydroxyl groups. Permanganate ions carve the fiber surface due to the reaction with the lignin constituents.

Benzoyl chloride treatment



Benzoyl chloride forms an ester linkage to the areca fibers and thus reducing its hydrophilicity and making it more compatible with polymer matrix.

Acrylic acid treatment



Acrylic acid provides more access of reactive cellulose macro radicals to the polymerization medium by reacting with the cellulosic hydroxyl groups of the fiber. An ester linkage is formed by the carboxylic acids present in acrylic acid during reaction with fiber.

Flexural strength

Flexural strength is the material's ability to bend without undergoing major deformities. It is the maximum stress experienced by the material at the point of rupture. The flexural strength values of untreated and all chemically treated areca fiber reinforced epoxy composites under 40%, 50%, 60% and 70% fiber loadings were given in Table 2.

Table 2. Flexural Strength values of areca/epoxy composites

Areca fiber	Flexural strength values in N/mm ² at given fiber loadings (wt %)			
	40%	50%	60%	70%
Untreated	30.24	38.48	42.68	24.28
Alkali treated	52.54	61.28	84.86	26.84
Potassium permanganate treated	54.38	64.96	88.98	27.26
Benzoyl chloride treated	56.02	66.58	94.28	28.46
Acrylic acid treated	57.54	68.52	96.84	28.98

It is observed that the fiber content and chemical modifications of areca fiber greatly influenced the flexural strength of areca/epoxy composites. The effect of chemical treatments on flexural strength of untreated and all chemically treated areca fiber reinforced epoxy composites with different fiber loadings are depicted in Figure 4.

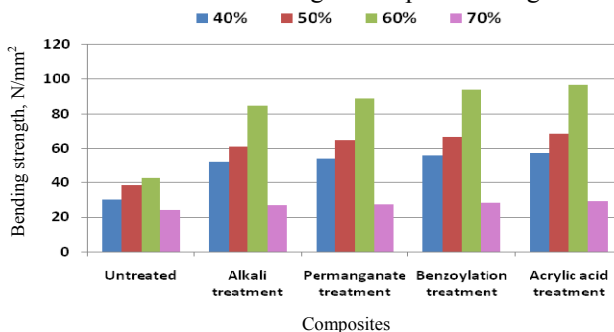


Figure 4. Flexural strength of areca fiber reinforced epoxy composites with different fiber loadings

Untreated areca fibers are hydrophilic whereas the epoxy resin is hydrophobic. This resulted in incompatibility between the areca fiber and the epoxy resin. Hence, the untreated areca/epoxy composites indicated low flexural strength values compared to chemically treated areca/epoxy composites. The flexural strength of untreated epoxy composites with 60% fiber loading increased by 41.13% compared to 40% fiber loading, 10.91% compared to 50% fiber loading and for 70% fiber loading flexural strength decreased by 43.11% compared to 60% fiber loading.

Alkali treatment of areca fibers takes out certain portion of hemicelluloses, lignin, adhesive pectin, waxy epidermal tissue and oil covering materials and reduced fiber diameter and thereby increased aspect ratio. As a result effective fiber surface area for good adhesion with the matrix is increased^{3, 26-29}. Hence, alkali treated areca/epoxy composites

showed increased flexural strength values when compared with that of untreated areca/epoxy composites. The flexural strength of alkali treated epoxy composites with 60% fiber loading increased by 61.52% compared to 40% fiber loading, 38.48% compared to 50% fiber loading and for 70% fiber loading flexural strength decreased by 68.37% compared to 60% fiber loading.

Permanganate ions carved the fiber surface due to the reaction with the lignin constituents and as a result areca fiber surface became rough. This improved chemical interlocking at the fiber interface and provided better adhesion with the polymeric resin^{3, 9, 11}. So, permanganate treated areca/epoxy composites showed increased flexural strength values when compared with that of untreated areca/epoxy composites. The flexural strength of permanganate treated areca/epoxy composites with 60% fiber loading increased by 63.63% compared to 40% fiber loading, 36.98% compared to 50% fiber loading and for 70% fiber loading flexural strength decreased by 69.36% when compared to 60% fiber loading.

Benzoylation treatment results in an introduction of ester linkage with areca fibers and it improved chemical interlocking at the interface and provided effective fiber surface area for good adhesion with the matrix^{3,12,26,29-30}. As a result, benzoyl chloride treated areca/epoxy composites showed higher flexural strength values when compared with that of untreated areca/epoxy composites. The flexural strength of benzoyl chloride treated areca/epoxy composites with 60% fiber loading increased by 68.30% compared to 40% fiber loading, 41.60% compared to 50% fiber loading and for 70% fiber loading flexural strength decreased by 69.81% when compared to 60% fiber loading.

During acrylic acid treatment, most of the hemicelluloses and lignin are removed and there is replacement of hydroxyl group by hydrophobic ester groups in the areca fiber. As a result, effective fiber surface area for good adhesion with the matrix is increased and stress transfer capacity at the interface is enhanced^{3,25,32}. Hence, the acrylic acid treated areca/epoxy composites showed higher flexural strength values when compared with that of untreated areca/epoxy composites. The flexural strength of acrylic acid treated areca/epoxy composites with 60% fiber loading increased by 68.30% compared to 40% fiber loading, 41.33% compared to 50% fiber loading and for 70% fiber loading flexural strength decreased by 70.07% when compared to 60% fiber loading.

The flexural strength values of untreated as well as all chemically treated areca/epoxy composites increased with increase in fiber loading from 40% to 60% and beyond 60% it showed a decline. That means areca/epoxy composites with 60% fiber loading exhibited maximum flexural strength. This is because of better fiber distribution in matrix, less fiber fractures and effective transfer of load from matrix to fibers at 60% fiber loading. The observed increase in flexural strength values with chemical modification and as well as with increase in fiber loading up to 60% is in good agreement with the results reported in literature^{4,5,14,33}.

The decrease in flexural strength values for untreated as well as chemically treated areca/epoxy composites beyond 60% fiber loading is due to poor interfacial adhesion and inefficient stress transfer from matrix to fibers³⁴.

Alkali, potassium permanganate, benzoyl chloride and acrylic acid treated areca/epoxy composites with 60% fiber loading showed 98.83%, 108.48%, 120.90% and 126.90% increase in flexural strength values respectively when compared to untreated areca/epoxy composite with same 60% fiber loading. These results clearly evidenced that chemical treatments are very effective in surface modification of the areca fibers and in improving the flexural strength of chemically treated areca/epoxy composites. Amongst all the chemical treatments carried out,

acrylic acid treated areca fiber reinforced epoxy composites of 60% fiber loading showed maximum flexural strength. So, these chemically treated areca fiber reinforced epoxy composites with 60% fiber loadings are best suitable for applications where high flexural strength is required.

Conclusion

Chemical modification of natural fiber would remove the impurities like pectin, fat and lignin present in the fiber. On the other hand, a rougher fiber surface may result after the chemical treatment due to the introduction of some reactive groups into the fiber. From these studies, it is clearly concluded that chemical treatments of areca fibers are of greater importance in modifying the fiber surface, in reducing the hydrophilic nature of the fiber, in enhancing the fiber matrix adhesion and thereby increasing the mechanical properties of the polymer composites. Flexural strength value of areca fiber reinforced epoxy composites is increased after chemical modification and maximum value of flexural strength is observed at 60% fiber loading for all untreated and chemically treated areca/epoxy composites. Amongst all the treatments carried out, acrylic acid treated areca/epoxy composites of 60% fiber loading showed maximum flexural strength value, followed by benzoyl chloride treatment, permanganate treatment and alkali treatment at same 60% fiber loading. Hence, based on the availability, low cost and good strength characteristics, the areca fiber reinforced epoxy composites can be considered as a very promising material for fabrication of light weight material and can be effectively used in industrial sector like automobile body building, office furniture, packaging industry, partition panels, etc.

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