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RESEARCH ARTICLE

A STUDY OF INFLUENCE OF CHEMICAL TREATMENTS ON FLEXURAL STRENGTH AND IMPACT STRENGTH OF ARECA FIBER REINFORCED NATURAL RUBBER COMPOSITES

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ABSTRACT

The aim of this research work is to study the influence of chemical treatments on flexural strength and impact strength of areca fiber reinforced natural rubber composites. Hence in this study, untreated, alkali treated, permanganate treated, benzoylated and acrylated areca fiber reinforced natural rubber composites were fabricated by using heat press machine under 40%, 50%, 60% and 70% fiber loadings at a compounding temperature of 130 ^{o}C. The evaluation of flexural strength and impact strength of untreated and all chemically treated areca-natural rubber composites was done by following ASTM standard procedures. With increase in fiber loading from 40% to 60%, the flexural strength and impact strength of untreated and all chemically treated areca-natural rubber composites were increased and beyond, they showed a decline. Amongst untreated and all chemically treated areca-natural rubber composites, the highest flexural strength and impact strength was shown by acrylated areca-natural rubber composites with 60% fiber loading.

INTRODUCTION

Today we are in the midst the revolution triggered by the onset of advanced natural fiber reinforced polymer composite materials because these polymer composite materials displays macroscopic properties superior to its parent constituents. At present, natural fiber reinforced natural rubber composites find remarkable importance in its end-use applications since these natural fiber reinforced natural rubber composites exhibit the combined behaviour of soft, elastic rubber matrix and the stiff, strong fibrous reinforcement. Natural Rubber (NR) is insoluble in water, alcohol, acetone, dilute acids and alkalis but soluble in ether, carbon disulphide, carbon tetrachloride, petrol and turpentine. Natural rubber shows superior building tack, which is essential in many products like tyres, hoses and belts. Natural rubber has several advantages such as low cost, low hysteresis, high resilience, excellent dynamic properties and fatigue resistance (Teh *et al*., 2004). The natural resilience of rubber is related to molecular flexibility, amorphous structure and very low intermolecular force of attraction. Hence natural rubber exhibits swift and easy response to force and release of force. Natural Rubber is produced by plants and hence it is renewable, inexpensive and creates no health hazard problems. The molecular weight, molecular weight distribution and non-rubber constituents of natural rubber are affected by clonally variation, season and use of yield stimulants, tapping system and method of preparation (Subramanyam, 1972). Hence natural rubber obtained from the same lot has been used in this study. The specifications of natural rubber granules are given in the Table 1.

Table 1 Specifications of Natural Rubber

Parameters	Value
Dirt content, % by mass	0.03
Volatile matter, % by mass	0.50
Nitrogen, % by mass	0.30
Ash, % by mass	0.40
Initial plasticity number, P_0	38
Plasticity Retention Index	78

Natural rubber possesses high tensile strength and modulus due to strain-induced crystallization and it also possesses good crack propagation resistance. The poor mechanical properties of raw natural rubber can be improved by mixing ingredients such as accelerators, activators, cross linking agents and fillers (Ismail and Turky, 2001). The significant improvements in

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properties of natural rubber are observed when the fillers like natural fibers are incorporated into natural rubber matrix (Dutta *et al*., 2001). But, the vital problem observed in the fabrication of natural fiber reinforced natural rubber composites is inappropriateness between the hydrophilic nature of the fiber and the hydrophobic nature of rubber which in turn leading to poor mechanical properties to natural rubber composites (Ismail *et al*., 2006). This problem can be solved by the surface modification of natural fibers by various chemical treatments as these chemical treatments help in reducing the hydrophilicity of natural fibers and in producing good adhesion between the treated fiber and the natural rubber matrix. Hence, surface modifications of natural fibers by various chemical treatments are one of the most important areas in the field of technical utilization of natural fiber reinforced polymer composites in various engineering sectors. Among many natural fibers, areca fiber appears to be a budding fiber, because it is cheap, available in huge quantities and a very high potential persistent crop and areca fibers have an immense potential in the composite field. The botanical name of areca is Areca Catechu Linnaeus and it belongs to the Arecaceae (Palmae), palm family and Arecoideae subfamily. 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. At present, 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.

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 (Rajan and Kurup, 2005). There is no literature available on the influence of chemical treatments on flexural strength and impact strength of areca fiber reinforced natural rubber composites. Hence, in this research work, areca fibers were treated with sodium hydroxide, potassium permanganate, benzoyl chloride and acrylic acid to minimize the hydrophilicity of natural areca fibers and to produce good adhesion between the areca fibers and natural rubber matrix so that areca fiber reinforced natural rubber composites with improved properties can be obtained. Then untreated and above said chemically treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings were fabricated by using heat press machine at a compounding temperature of 130 ^{o}C. The investigation of flexural strength and impact strength of untreated and all chemically treated areca fiber reinforced natural rubber composites was done by following ASTM standard procedures. The influence of above said chemical treatments and effect fiber loading on the flexural strength and impact strength of areca fiber reinforced natural rubber composites was studied.

MATERIALS AND METHODS

MATERIALS

Areca empty fruits were obtained from Madhu Farm House, Nilogal, Davangere, Karnataka, India. The analytical grade reagents were purchased from Qualigens Company and used as received. Natural Rubber (NR) granules which were used as matrix material in this work for the preparation of areca fiber reinforced thermoplastic elastomeric polymer composites was obtained from Akolite Synthetic Resins, Mangalore. Natural rubber granules used for the study was ISNR 5 (Indian Standard Natural Rubber-5) grade and light in colour.

Fabrication of areca fiber reinforced natural rubber composites

The fabrication of untreated, alkali treated, potassium permanganate treated, benzoyl chloride treated and acrylic acid treated areca fiber reinforced natural rubber composite plates was carried out by using compression moulding technique. The areca fiber is used as reinforcement material and the natural rubber granules were taken as matrix material. The weight fractions, 40%, 50%, 60% and 70% of areca fiber was carefully controlled during the mixing of two ingredients. The resulting material was compression moulded to the dimension of 300 x 300 x 5.0 mm^3. The composite preparation process was performed in the following order. First, the heat press was pre-heated to 60 ^{o}C. Then the pressure was set as 0 MPa and the temperature was raised to 100 ^{o}C. After that the pressure was raised to 5 MPa and temperature was raised to 130 ^{o}C. Further, raised the pressure to 15 MPa, maintained the pressure and temperature for 30 min. Finally, lowered the pressure to 0 MPa, lowered the temperature to 30 ^{o}C and composite plate was removed from the heat press. The specimens were post cured for 48 h before the test.

Testing of flexural strength

Three – point static flexural tests of untreated and all chemically treated areca fiber reinforced natural rubber composites were carried out according to ASTM D 790-10 standard procedure using a Universal Testing Machine at a crosshead speed of 10 mm/min at a standard laboratory atmosphere of 30±2 ^{o}C and 65% relative humidity (RH 65%). The specimens prepared for the flexural test were cut with help of zig saw and the specimen dimensions used were 80 mm x 10 mm x 5 mm.

$$\text{Flexural strength, } \sigma_f = \frac{3PL}{2bd^2} \quad \text{(1)}$$

The flexural strength was calculated from the measured load according to the equation (1) given above where σ_f = flexural strength, P = maximum applied load, L = length of support span (mm), b = width of specimen (mm) and d = thickness of specimen (mm). At least 5 replicate specimens were tested and the results were presented as an average of tested specimens.

Testing of impact strength

Dynamic Charpy impact test of the untreated and all chemically treated areca fiber reinforced natural rubber composite specimens was conducted on notched composite specimens according to ASTM D 6110-10 using a Universal Impact Testing Machine at a standard laboratory atmosphere of 30±2 ^{o}C and 65% relative humidity (RH 65%). The width and

depth of each specimen was measured with a micrometer screw gauge to the nearest of 0.01 mm and the length was measured to the nearest of 0.1 mm with digital calliper. The dimensions of the specimen used were 130 mm x 12.5 mm x 5 mm. At least 5 replicate specimens were tested and the results were presented as an average of tested specimens.

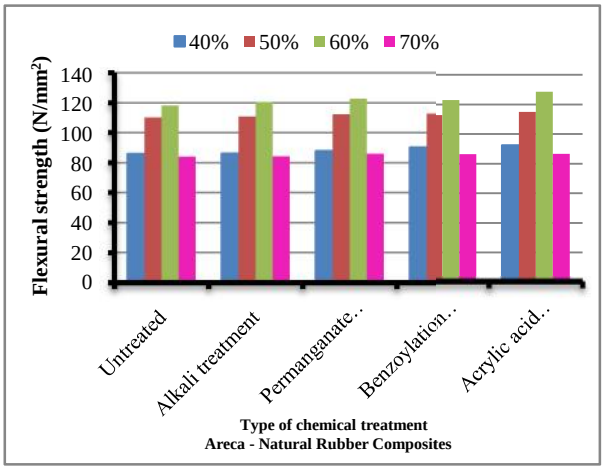
RESULTS AND DISCUSSION

Chemical treatment of areca fibers

Surface modification of areca fiber is done by using chemical treatments such as alkali treatment, potassium permanganate treatment, benzoyl chloride treatment and acrylic acid treatment in order to optimize the effective interfacial bonding between areca fibers and natural rubber matrix so that the areca fiber reinforced Natural Rubber composites with improved properties can be obtained. The chemical treatment procedure, chemical reaction involved and the corresponding changes occurred during the above said chemical treatments of areca fibers have already been explained in detail in the previous study (Dhanalakshmi *et al*., 2015). This study revealed that these chemical treatments improved chemical interlocking at the interface by exposing more reactive groups on the areca fiber surface. Further, chemically treated areca fiber surface became more hydrophobic and accordingly, there is improvement in surface characteristics such as wetting, adhesion and porosity of areca fibers. Also, there is improvement in interfacial adhesion between the treated areca fiber surface and the natural rubber matrix.

Flexural strength of areca fiber reinforced natural rubber composites

Flexural strength is the ability of the material to resist deformation under load and is expressed in MPa or N/mm^2. 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. The flexural strength of untreated and all chemically treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are pictorially represented in Graph 1.



Graph 1 Flexural strength of Areca – NR composites with different fiber loadings

It is observed that the areca fiber content and the chemical modifications of areca fiber significantly influenced the flexural strength of areca - NR composites. Further, irrespective of the chemical treatment, all chemically treated areca - NR composites showed higher flexural strength values than the untreated areca fiber reinforced natural rubber composites with corresponding fiber loadings.

The flexural strength values of untreated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 86.02 N/mm^2, 110.24 N/mm^2, 118.26 N/mm^2 and 84.02 N/mm^2 respectively. The flexural strength of untreated areca fiber reinforced natural rubber composites with 60% fiber loading increased by 37.48% compared to 40% fiber loading, 07.28% compared to 50% fiber loading and for 70% fiber loading, flexural strength decreased by 28.95% when compared to 60% fiber loading.

The flexural strength values of alkali treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 86.28 N/mm^2, 110.86 N/mm^2, 120.52 N/mm^2 and 84.28 N/mm^2 respectively. The flexural strength of alkali treated areca fiber reinforced natural rubber composites with 60% fiber loading increased by 39.68% compared to 40% fiber loading, 8.71% compared to 50% fiber loading and for 70% fiber loading, flexural strength decreased by 30.07% when compared to 60% fiber loading.

The flexural strength values of potassium permanganate treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 88.02 N/mm^2, 112.26 N/mm^2, 122.68 N/mm^2 and 86.02 N/mm^2 respectively. The flexural strength of potassium permanganate treated areca fiber reinforced natural rubber composites with 60% fiber loading increased by 39.38% compared to 40% fiber loading, 9.28% compared to 50% fiber loading and for 70% fiber loading, flexural strength decreased by 29.88% when compared to 60% fiber loading.

The flexural strength values of benzoyl chloride treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 90.24 N/mm^2, 112.86 N/mm^2, 122.98 N/mm^2 and 86.52 N/mm^2 respectively. The flexural strength of benzoyl chloride treated areca fiber reinforced natural rubber composites with 60% fiber loading increased by 36.28% compared to 40% fiber loading, 8.97% compared to 50% fiber loading and for 70% fiber loading, flexural strength decreased by 29.65% when compared to 60% fiber loading.

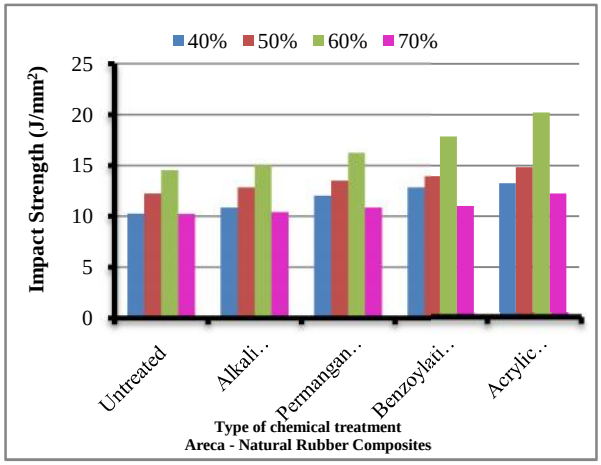
The flexural strength values of acrylic acid treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 92.54 N/mm^2, 114.86 N/mm^2, 128.52 N/mm^2 and 86.68 N/mm^2 respectively. The flexural strength of acrylated areca fiber reinforced natural rubber composites with 60% fiber loading increased by 38.88% compared to 40% fiber loading, 11.89% compared to 50% fiber loading and for 70% fiber loading, flexural strength decreased by 32.56% when compared to 60% fiber loading.

Sodium hydroxide, potassium permanganate, benzoyl chloride and acrylic acid treated areca fiber reinforced natural rubber composites with 60% fiber loading showed 1.91%, 3.74%,

3.99% and 8.68% increase in flexural strength values respectively when compared to untreated areca fiber reinforced natural rubber composites with same 60% fiber loading.

Impact strength of areca fiber reinforced natural rubber composites

Impact strength is the capability of the material to withstand a suddenly applied load and is a very important property of a material governing the life of a structure. Impact tests are used in studying the toughness of material. Toughness is dependent upon temperature and the shape of the test specimen. Impact strength is determined by Charpy impact test. The impact strength is calculated as the ratio of impact absorption to test specimen cross-section (J/mm^2). It is very vital to know optimum fiber loading to get superior impact properties. The impact strength of untreated and all chemically treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are pictorially represented in Graph 2.



Graph 2 Impact strength of areca – NR composites with different fiber

It is observed that the areca fiber content and chemical modifications of areca fiber significantly influenced the impact strength of areca fiber reinforced natural rubber composites. Irrespective of the chemical treatment, all chemically treated areca fiber reinforced natural rubber composites showed higher impact strength values than the untreated areca fiber reinforced natural rubber composites with corresponding fiber loadings.
The flexural strength values of untreated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 10.26 J/mm^2, 12.24 J/mm^2, 14.56 J/mm^2 and 10.22 J/mm^2 respectively. In case of untreated areca fiber reinforced natural rubber composites, the impact strength of 60% fiber loading increased by 41.91% compared to 40% fiber loading, 18.95% compared to 50% fiber loading and for 70% fiber loading, it decreased by 29.81% when compared to 60% fiber loading.

The flexural strength values of alkali treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 10.85 J/mm^2, 12.84 J/mm^2, 15.02 J/mm^2 and 10.42 J/mm^2 respectively. In case of alkali treated areca fiber reinforced natural rubber composites, the impact strength of 60% fiber loading increased by 38.43% compared to 40% fiber loading, 16.98% compared to 50% fiber loading and for 70% fiber loading, it decreased by 30.63% when compared to 60% fiber loading.

The flexural strength values of potassium permanganate treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 12.04 J/mm^2, 13.52 J/mm^2, 16.24 J/mm^2 and 10.86 J/mm^2 respectively. For potassium permanganate treated areca fiber reinforced natural rubber composites, the impact strength of 60% fiber loading increased by 34.88% compared to 40% fiber loading, 20.12% compared to 50% fiber loading and for 70% fiber loading, it decreased by 33.13% when compared to 60% fiber loading.
The flexural strength values of benzoyl chloride treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 12.86 J/mm^2, 13.96 J/mm^2, 17.86 J/mm^2 and 11.02 J/mm^2 respectively. In case of benzoyl chloride treated areca fiber reinforced natural rubber composites, the impact strength of 60% fiber loading increased by 38.88% compared to 40% fiber loading, 27.94% compared to 50% fiber loading and for 70% fiber loading, it decreased by 38.30% when compared to 60% fiber loading.

The flexural strength values of acrylic acid treated areca fiber reinforced natural rubber composites with 40%, 50%, 60% and 70% fiber loadings are 13.26 J/mm^2, 14.82 J/mm^2, 20.22 J/mm^2 and 12.24 J/mm^2 respectively. In acrylic acid treated areca fiber reinforced natural rubber composites, the impact strength of 60% fiber loading increased by 52.49% compared to 40% fiber loading, 36.44% compared to 50% fiber loading and for 70% fiber loading, it decreased by 39.47% when compared to 60% fiber loading.

Sodium hydroxide, potassium permanganate, benzoyl chloride and acrylic acid treated areca fiber reinforced natural rubber composites with 60% fiber loading showed 3.16%, 11.54%, 22.66% and 38.87% increase in impact strength values respectively when compared to untreated areca fiber reinforced natural rubber composites with same 60% fiber loading.

Influence of chemical treatments and effect fiber loadings on flexural strength and impact strength of areca fiber reinforced natural rubber composites

The flexural strength and impact strength of natural fiber reinforced polymeric composites depends on the nature of the fiber, polymer and fiber–matrix interfacial bonding (Joseph *et al.*, 2003). The hydrophilic nature of untreated areca fibers and the hydrophobic nature of natural rubber matrix resulted in inappropriateness between the areca fibers and the natural rubber matrix. Hence, the untreated areca fiber reinforced natural rubber composites indicated low flexural strength and impact strength values when compared to chemically treated areca fiber reinforced natural rubber composites of corresponding fiber loadings.

Alkali treatment of areca fibers takes out certain portion of hemicelluloses, lignin, adhesive pectin, waxy epidermal tissue and oil covering materials and reduced areca fiber diameter and

thereby increased its aspect ratio. As a result, effective fiber surface area available for good adhesion with the natural rubber matrix is increased (Dipa *et al*., 2001, Kumar *et al*., 2013, Leonard *et al*., 2007, Mishra *et al*., 2001, Prasad *et al*., 1983, Xue *et al*., 2007).

Permanganate ions react with the lignin constituents and carve the areca fiber surface. As a result, areca fiber surface became physically rough. This improved chemical interlocking at the interface and provided better adhesion with the polymeric resin (Joseph *et al*., 2002, Rahaman *et al*., 2007).

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 (Joseph *et al*., 1996, Joseph *et al*., 2002, Manikandan *et al*., 2001, Wang *et al*., 2007, Xue *et al*., 2007,).

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 available for good adhesion with the natural rubber matrix is increased and stress transfer capacity at the interface is enhanced (Bessadok *et al*., 2007).

Hence, alkali treated, potassium permanganate treated, benzoyl chloride treated and acrylic acid treated areca fiber reinforced natural rubber composites showed improved flexural strength and impact strength values when compared with that of untreated areca fiber reinforced natural rubber composites.

With increase in fiber loading from 40% to 60%, the flexural strength and impact strength values of all untreated as well as chemically treated areca - NR composites increased and beyond 60% fiber loading, they showed a decline. That is areca fiber reinforced natural rubber composites exhibited maximum flexural strength and impact strength values at 60% fiber loading. 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. As fiber loading increases, more force is required to pullout the fibers and thereby increases the flexural strength and impact strength. It has also been reported that high fiber content increases the probability of fiber agglomeration and further it results in regions of stress concentration and requires less energy for crack propagation (Karmakar *et al*., 2007).

The observed increase in flexural strength and impact strength values with chemical modification and as well as with increase in fiber loadings up to 60% is in good agreement with the results reported in literature (Girones *et al*., 2011, Jayaraman 2003, Md. Rezaur Rahaman *et al*., 2010, Patel and Parsania 2010, Swamy *et al*., 2004) . These results suggest that the areca fiber reinforced natural rubber composites are capable of showing high flexural strength and impact strength values because of strong interfacial bonding between the areca fibers and natural rubber matrix at 60% fiber loading.

Further, in the cases of areca – NR composites with 40% and 50% fiber loadings, lesser areca fiber fraction is used and here, areca fibers are serving as the crucial reinforcement in the NR composites and hence, the reduced fiber amount resulted in a significant decrease in flexural strength and impact strength values compared to areca fiber reinforced natural rubber composites with 60% fiber loading (Dhanalakshmi *et al*., 2014).

The decrease in flexural strength and impact strength values for untreated as well as chemically treated areca – NR composites beyond 60% fiber loading is due to fact that the melted rubber could not reach each of the areca fiber surfaces because of the smaller amount of matrix material. And also, there is poor interfacial adhesion and inefficient stress transfer from matrix to fibers at 70% fiber loading (Ratna Prasad *et al*., 2009). The factors contributing to the lower flexural strength and impact strength values may also be due to the random alignment of short areca fibers and the presence of voids in the areca fiber reinforced natural rubber composites (Zuhari *et al*., 2010). The decrease in impact strength may also be due to the micro spaces between the areca fibers and the natural rubber matrix which initiates micro cracks on impact and results in crack propagation leading to failure (Srinivasa *et al*., 2013).

This study results clearly indicated that chemical treatments are very successful in surface modification of areca fibers and in improving the flexural strength and impact strength values of areca fiber reinforced NR composites. Amongst all the chemical treatments carried out, acrylic acid treated areca - NR composites of 60% fiber loading showed maximum flexural strength and impact strength values. So, these chemically treated areca - NR composites with 60% fiber loadings are best suitable for applications where high flexural strength and impact strength are required.

CONCLUSIONS

The low molecular weight compounds such as waxy epidermal tissue, adhesive pectin and hemicelluloses present in the natural fibers are slightly removed by chemical treatments and hence natural fiber surface become physically rough. Also, there is introduction of some reactive groups into the fiber surface. This research work clearly revealed 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 properties of polymer composites. Chemical treatments resulted in increased flexural strength and impact strength values for areca fiber reinforced natural rubber composites. For all untreated and chemically treated areca fiber reinforced natural rubber composites, highest values of flexural strength and impact strength is observed at 60% fiber loading. Amongst all the chemical treatments carried out, acrylic acid treated areca fiber reinforced natural rubber composites of 60% fiber loading showed maximum flexural strength and impact strength values followed by benzoyl chloride treatment, permanganate treatment and alkali treatment at the same 60% fiber loading. Hence, areca fiber reinforced natural rubber composites can be considered as a very promising material for the fabrication of light weight materials and can be effectively used in industrial sector like automobile body building, office furniture, partition panels and packaging industry.

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