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### CONFERENCE PAPER

## EVALUATION OF SURFACE TREATMENT EFFECTS ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF RATTAN (CALAMUS BECCARII) FIBER

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#### Abstract

The goal of this study is to thoroughly comprehend the advantages of rattan (*Calamus beccarii*) as potential reinforcement in polymeric composites. The influence of various chemical treatments on thermal, morphological, mechanical and water absorption characteristics of natural rattan (RA) stem fiber were investigated. In this research, RA fiber surface was modified through different chemical treatments such as alkalization, bleaching, and benzylation. The presence of voids and rough surfaces was investigated on SEM micrographs which are due to removal of lignin, wax, and oils from the fiber surface to a large extent. The increase in tensile strength and Young's modulus confirms improvement in the mechanical properties of the RA fiber after chemical treatment. It was observed that alkali-treated RA fibers exhibit highest mechanical properties (295.28 MPa tensile strength, 8.23 GPa Young's modulus). Thermogravimetric analysis confirms that there was an increase in the thermal stability of the fiber after chemical treatment. Overall results confirm that the RA fiber is appropriate for use as a reinforcing phase in composite materials for prospective engineering semi-structural applications such as roofing sheets, bricks, door panels, furniture panels, interior paneling, storage tanks, and pipelines.

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#### Introduction:-

Growing concern toward environmental safety has encouraged the researcher to synthesize new green materials. Eco-friendly, bio-degradability, and lightweight have become vital considerations in the fabrication of new material (Graupner, Herrmann and Mussig 2009). Currently, synthetic fibers like glass, carbon, and aramid are widely being used in polymer-based composites because of their high stiffness and strength properties (Rout et al. 2001).

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However, these fibers have serious drawbacks in terms of their biodegradability, initial processing costs, recyclability, energy consumption machine abrasion, health hazards, etc. (Bledzki and Gassan 1999). Natural fibers can eliminate these drawbacks of synthetic fibers. Natural fibers have several tailor-made properties like lightweight, renewable, low cost, high specific modulus, high cellulose content, low density, nontoxic, easy fiber surface modification, relative nonabrasiveness, and easy for processing (Natarajan, Kumaravel, and Palanivelu 2016; Santhanam et al. 2016).

Natural fibers are classified based on their origins, coming from plants, animals, or minerals. Commonly, natural fibers, that is, extracted from fruits, seeds, stems, and roots of the plant known as plant fibers have been used as reinforcement in composite material. Also, these plant fibers have many traditional applications such as making of ropes, cords, hats, mats, and strings. Plant fibers are lignocellulosic fibers in which cellulose is the principal constituent, which is bounded by hemicellulose and lignin compound (Bledzki and Gassan 1999). Besides all the benchmarking properties of the plant fiber, it has few limitations like poor adhesion at the fiber-matrix interface, weak thermal stability, and moisture sensitive. The morphological changes by different chemical treatments can eliminate the deficiencies (Kabir et al. 2012). Chemical treatments can clean the fiber surface, chemically modify the surface, stop the moisture absorption process, and increase surface roughness to be more compatible with hydrophobic polymers. Chemical treatment increases the surface roughness of the fiber surface by removing the amorphous constituents that surrounds the external surface of the fiber thus improving the fiber and matrix bonding. Many researchers have experimented with different chemical treatments on the fiber surface. Surface modification results in increase in the thermal stability of the fiber and enhancement of mechanical properties (Dehury, Nayak, and Mohanty 2021). Manimaran and co-authors (Manimaran et al. 2020) successfully extracted *Aristida adscensionis* fibers and studied their various properties. They found high cellulose content of 70.78% whereas the contents of lignin and wax were equal to 8.9% and 2.26%, respectively. Moreover, the fiber has a low density equal of 790 kg/m<sup>3</sup> whereas fiber diameter is equal to about 46 μm. The tensile strength and modulus was approximately 15.25 MPa and 0.78 GPa, respectively.

Oladele and Agbabiaka investigated the effect of mercerization treatment (KOH and NaOH) on sisal fiber (Oladele and Agbabiaka 2015). They found that KOH treated sisal fiber reinforced polypropylene composites gave the best flexural and tensile properties when compared to NaOH treatment. Madhu and co-authors studied the effect of various chemical treatments on *Agave Americana* fiber (Madhu et al. 2020). They found that the chemical treatments on the fibers brought a significant reduction in the amorphous contents like hemicellulose, lignin and other impurities and made them less resistant to water molecules. In addition, the thermal properties of the fibers were enhanced after surface modification. Al-Oqla et al. investigated the mechanical, thermal and interfacial characteristics of Jordanian lignocellulosic fibers. They found that palm fiber type shows highest mechanical properties followed by olive fiber. Lemon and loquat fibers were the best in thermal stability (Al-Oqla et al. 2019). Kabir et al. reported that benzylation treatment reduces the hydrophilic nature of the fiber, thereby increasing the strength of the composite (Kabir et al. 2012). Nayak and Mohanty found that benzylation enhances the thermal stability of the fiber (Nayak and Mohanty 2019). Sodium chlorite treated sisal fiber gave higher tensile strength and also there was decline in the moisture absorption rate reported by Li and co-authors (Li, Panigrahi, and Tabil 2009). Szczepanowska (Szczepanowska 2017) described various types of rattan and epidermis morphology, topography, and cellular structure were characterized by a multi-scale and a multi-sensory approach using optical microscopy, confocal laser scanning microscopy, and field-emission scanning electron microscopy (FESEM).

In this present research work, the natural fiber, that is, obtained from the plant rattan (RA) belongs to the subfamily “Calamoideae” and is widely used in making chairs, clothing, medicines, handicraft, and arts and also its fruit is edible. Rattan fibers contain 73.83% of cellulose, 12.49 of hemi-cellulose, 10.15% of lignin, 3.15% of aqueous extract, and 0.37% of pectin (Sahoo et al. 2019). However, in the composite field of application, literatures about the rattan stem fiber are very scanty. Hence, the present work aims to modify the surface of the rattan fiber by various chemical treatments to enhance the fiber-matrix adhesion during composite fabrication in future. This also aims to find out the effects of chemical treatment on various properties like mechanical, thermal and morphological properties of rattan fibers.

## Materials and Methods:-

### Materials:-

Rattans are categorized as a member of the palm family (Palmae or Arecaceae) which has roughly around 600 species among which *Calamus beccarii* is one of them. It has spiny, brown stems, 50 m tall, 4 cm diameter. Rattan has significant properties that make them popular in industries such as furniture, handicraft and other relevant industries for being durable, light weight, elastic, and flexible (Olawale and Wasiu 2013). Rattan is consisted of lignin, cellulose, and hemicellulose. The raw material for the present study, rattan stems from a species named *Calamus beccarii* (Figure 1), was collected from local places of Bhubaneswar, Odisha, India. Chemicals like sodium hydroxide, sodium chlorite and benzyl chloride of analytical grades were purchased from Mohapatra Chemicals, Bhubaneswar and used without any further purification.



Figure 1. (a) Rattan plant, (b) stem and (c) extracted fibers from RA stem.

### Methods:-

#### Fiber extraction process:-

The rattan fiber was collected from rattan plant (Figure 1(a)). Rattan cane was then separated from the plant as shown in Figure 1(b). The rattan cane between inter-nodes was sliced by using a slicer. The sliced rattan was then immersed in water for 5 days. The fibers were extracted from the rattan cane and thoroughly washed with distilled water to remove any impurities and dried in sunlight to remove moisture from the fiber. The fibers extracted at this stage were designated as untreated (raw) fibers (Figure 1(c)).

**Surface treatment of fibers:-** Chemical surface treatment is commonly used to modify natural fibers and enhance their bonding with polymer matrices. Natural fibers are inherently hydrophilic due to the presence of hydroxyl (-OH) groups in their lignocellulosic structure, leading to moisture absorption, swelling, and degradation. Chemical treatments remove amorphous constituents such as lignin, hemicellulose, and impurities, thereby increasing cellulose exposure, surface roughness, and crystallinity, which improves mechanical and thermal properties while reducing water absorption. In this study, rattan (RA) fibers were subjected to alkalization, benzylation, and bleaching treatments. Alkalization using 5% NaOH at 80°C for 1 hour reduces hydrophilicity by disrupting hydrogen bonds and removing impurities. Benzylation replaces hydroxyl groups with benzoyl groups, making the fibers more hydrophobic and enhancing matrix adhesion. Bleaching with sodium chlorite (NaClO<sub>2</sub>) removes residual lignin and impurities through oxidation. These treatments collectively improve the overall performance of the fibers for structural and outdoor applications.

**Tensile properties:-** A Universal Testing Machine (Instron 3369 UTM) was used to measure the tensile strength of the RA fibers. Single fiber of all treated and untreated RA fibers was analyzed for their tensile strength and Young's modulus as per ASTM D 3379-75. A thick sheet of paper frame was prepared which hold the specimen in the machine. A slot was cut in the centre of the paper frame having length equal to the gage length of the specimen. A single filament of RA fiber was attached to both the ends of the slot in the paper using glue. The actual gage length

of the fiber was measured using a Vernier caliper to be 50 mm. During the test a full-scale load of 1 N and feed rate of 0.5 mm/min was maintained. Both sides of the frames were cut carefully without disturbing the set-up as a whole. The environmental conditions were maintained at 23°C temperature and 54% humidity. 10 samples were tested and their average value was recorded.

**Thermogravimetric analysis (TGA):**-The thermal stability of RA fibers was characterized using a thermogravimetric analyzer (TA, Q50 V20.13 Build 39). About 10 mg of fibers are exposed to atmospheric air with a heating rate of 10°C min<sup>-1</sup> from room temperature to 700°C.

**Morphological characterization:**-Scanning electron microscope (SEM) of the RA fibers was performed to observe the surface morphological changes in the fiber. 10nm of gold layer coating was applied on all the treated and untreated samples before the graphs were taken. Then the surface morphology of fibers was obtained using the Leo Supra 35VP SEM operating at 20 kV generated the Scanning Electron graphs.

## Results and Discussion:-

### Tensile Properties:-

The mechanical properties of untreated and various chemically treated RA fibers are presented in Figure 2. A significant increase in tensile strength and Young's modulus of all the chemically treated RA fibers was observed in Figure 2(a, b) as compared to untreated RA fibers. The enhancement in mechanical behavior was possibly due to removal of amorphous constituents present on the surface of the fibers, which acts as a barrier in case of untreated RA fiber (Fidelis et al. 2013). Chemical treatment also enhances the crystallinity of the fibers which makes the fiber strong and enhances the mechanical properties. The stress transfer between the fiber cell increases due to removal of the unwanted and cemented materials and the fiber becomes more homogenous. Among all treated fibers alkali treated fibers exhibits highest tensile and Young's modulus of 295.28 MPa and 8.23GPa respectively. The obtained tensile strength is more than alkali treated vetiver fibers i.e 204.34MPa (Jena, Mohanty, and Nayak 2020), Agave Americana fiber i.e 282MPa (Madhu et al. 2020), Pennisetum orientale fiber i.e 98.91 MPa (Nayak 2022) and Jordanian palm fiber i.e 160MPa (Aloqla, Hayajneh, and Fares 2019) This increased value in NaOH (alkalization) may be due to the formation of the strong covalent bond between the cellulose chain, which results in close packing and stiffness of the fiber. In addition, it was observed that sodium chlorite treated fibers reveals highest elongation at break as compared to other treated and untreated fibers which fractures by deforming much under the tensile load.

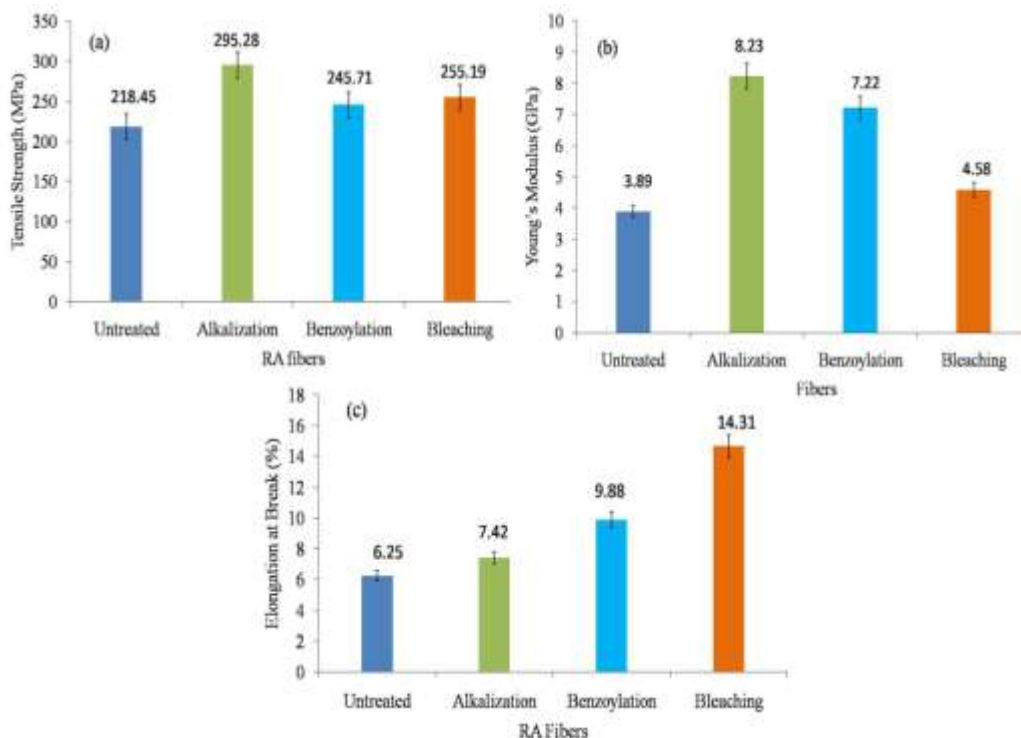


Figure 2. Mechanical properties of untreated and treated RA fibers.

### Thermogravimetric analysis (TGA):-

The TGA curve of untreated and treated RA fiber is shown in Figure 3. The thermogravimetric analysis of natural fiber is highly essential to have an idea of its response toward different temperature ranges. The cellulose, hemicellulose, and lignin degrade at different temperature. From TGA curve it was observed that the degradation of RA fiber was associated with four stage degradation process. The initial weight loss was observed from temperature range of 40 to 120°C which may be due to evaporation of moisture from the fibers (Brigida et al. 2010). Within this temperature range, weight loss of 10% was observed in case of untreated fiber showing its higher moisture content. Whereas, in case of all treated RA fiber, the weight losses was found to be 5 % to 7% of the total fiber weight due to enhance moisture resistance property (Senthamaraikannan and Kathiresan 2018). No significant weight loss was observed in case of untreated RA fiber in the temperature range of 120 to 250°C and in case of treated RA fibers in the temperature range of 120 to 263°C. This shows increased thermal stability of RA fibers in the temperature range of 250 to 263°C due to chemical treatments. The second weight loss of 45% was observed in case of untreated fiber in the temperature range of 250 to 360°C. This weight loss corresponds to depolymerization, thermal degradation of hemicellulose and cellulose. However, in case of alkalization a second weight loss of 34% was observed in the temperature range of 250 to 360°C. This is possibly due to more cellulose crystallinity (Sathishkumar et al. 2013). After 360°C temperature, the lignin starts degrade slowly because of its complex structure till 700°C. At 700°C, the residual char of maximum 2-4% was found in case of treated RA fibers due to strong bonding of lignin and cellulose whereas in case of untreated fiber 7% char was found.

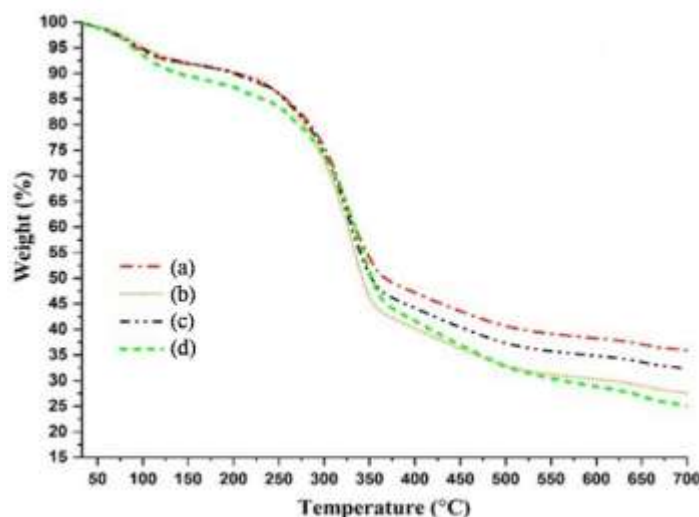


Figure 3. TGA of (a)Alkalization (b)Benzoylation (c)Bleaching (d) Untreated RA fibers.

### Morphological analysis of RA fibers:-

#### Untreated:-

The SEM of untreated RA fiber is shown in Figure 4a. From SEM image it was seen that the raw RA fiber is covered with fatty and waxy substances making the fiber surface smooth and shiny. Also, the white patches on raw fiber accounts for impurities present on the fiber surface due to binding of hemicellulose and lignin.

#### Alkalization:-

Figure 4b shows the effect of alkalization on the surface of the fiber. This treatment eliminates hemicellulose, wax, and oils covering the external surface of the fiber. As a result, the fiber surface becomes clean and more uniform due to the elimination of micro-voids. Also, there is a reduction in fiber diameter, which enhances the aspect ratio (Oladele and Agbabiaka 2015).

#### Benzoylation:-

The SEM image of benzoyl chloride treated RA fibers is shown in Figure 4c. Pin holes and rough surface is clearly visible in the figure. This is due to the removal of lignin, wax, and oil covering the fiber surface by exposing more hydroxyl (OH) group on alkali pretreatment. Additionally, OH groups were replaced by benzoyl groups after being

treated with benzoyl chloride, which improved the fiber's hydrophobic character and fiber-matrix adhesion (Mobarak et al. 2018).

#### Bleaching:-

The SEM of bleaching treatment is shown in Figure 4d. It can be seen from SEM image that the fiber surface becomes rougher on bleaching due to dissolution of lignin, wax and pectin. This bleaching effect releases oxygen that oxidizes the fiber impurities. The fiber contains more pores and cavities. The liberation of  $\text{ClO}_2$  reacts with hydroxyl groups of hemicellulose and lignin constituents of the fiber by removing lignin from the fiber surface (Nayak and Mohanty 2019). These combined effects remove moisture from the fiber and enhance the water absorptivity property.

Figure 5 shows the fiber cross-section before and after alkali-treatment. From the figure it can be seen that chemical treatment not only reduces the voids content but also decreases the fiber diameter.

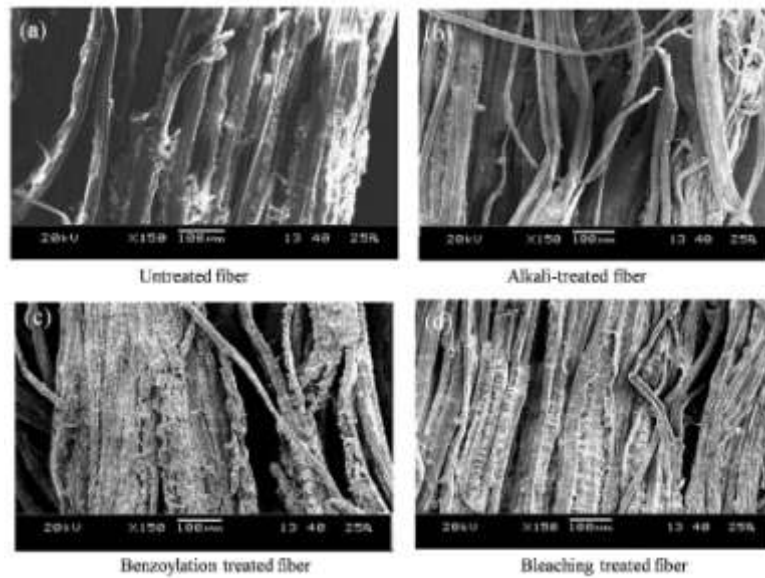


Figure 4. SEM images of untreated and treated RA fibers.

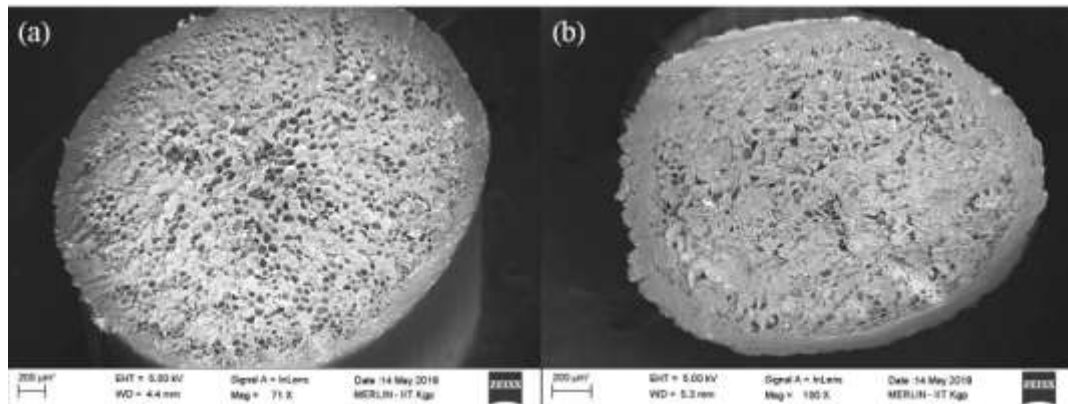


Figure 5. Cross-section of RA fiber (a) Untreated (b) alkali treated

#### Conclusion:-

The present study systematically investigated the influence of different chemical surface treatments—alkalization, benzoylation, and bleaching—on the mechanical, thermal, and morphological properties of rattan (*Calamus beccarii*) fibers. The results clearly demonstrate that chemical modification significantly enhances the overall performance of the fibers by removing amorphous constituents such as lignin, hemicellulose, waxes, and surface impurities.

Among all treatments, alkalization proved to be the most effective, yielding the highest tensile strength (295.28 MPa) and Young's modulus (8.23 GPa), attributed to improved cellulose exposure, increased crystallinity, and better stress transfer within the fiber structure. Thermogravimetric analysis confirmed enhanced thermal stability of treated fibers, with reduced moisture content and delayed degradation temperatures compared to untreated fibers. Morphological observations through SEM revealed cleaner, rougher surfaces with reduced diameter and void content, which are favorable for improved fiber–matrix interfacial bonding. Overall, the study establishes that surface-treated rattan fibers exhibit superior mechanical strength, improved thermal resistance, and favorable surface characteristics, making them promising candidates for reinforcement in polymer composites. These findings highlight the potential application of rattan fibers in eco-friendly, lightweight, and semi-structural engineering components such as panels, roofing materials, and interior structures.

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