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CONFERENCE PAPER

INFLUENCE OF BENZOYLATION TREATMENT ON MECHANICAL AND THERMAL PROPERTIES OF VETIVER FIBER REINFORCED EPOXY COMPOSITES

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Abstract

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This study focuses on the mechanical and thermal characterization of a novel composite material comprising vetiver grass fiber (Chrysopogonz izanioides) (VF) as reinforcement within an epoxy matrix. The fibers were chemically modified using benzyl chloride to enhance interfacial compatibility with the epoxy resin. Mechanical properties, including tensile strength, flexural strength, and impact strength, were evaluated for composites containing varying weight fractions of treated fibers. The results indicate that the composite exhibits maximum mechanical performance at an optimal fiber loading of 20 wt%.The interfacial interactions between the fiber and polymer matrix were further analyzed using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) at this optimal fiber content. Based on both characterization and property evaluation, it is concluded that the VF/epoxy composite demonstrates superior performance at a fiber loading of 20 wt%.

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Introduction:-

Growing environmental awareness across the globe has significantly influenced material engineering and design practices. Among the pressing environmental issues, global warming demands focused attention from the scientific community. This phenomenon arises from multiple factors, including both natural processes and human activities. One of the prominent anthropogenic contributors is the extensive use of plastics and composite materials reinforced with synthetic fibers.Since the early development of composite materials using straw and mud, researchers and engineers have advanced toward the fabrication of low-cost fiber-reinforced polymer (FRP) composites incorporating synthetic fibers within polymer matrices. However, the application of synthetic fibers is increasingly being limited due to several drawbacks such as non-biodegradability, high initial processing costs, poor recyclability, high energy consumption, machine abrasion, and potential health hazards [1].Consequently, there has been a shift in research focus toward natural fibers as sustainable alternatives to synthetic reinforcements.

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The selection of natural fibers for polymer composites primarily depends on their well-defined structural and mechanical properties. A variety of natural fibers, including hemp, kenaf, flax, areca sheath, sisal, jute, banana fiber, date palm, and bagasse [2–6], have demonstrated significant potential in the development of eco-friendly composite materials. These fibers offer several advantages such as low density, high specific modulus, non-toxicity, ease of processing, and the ability to absorb CO_2 during their growth cycle [7–8].Despite these benefits, the primary limitation of natural fibers lies in their hydrophilic nature, which leads to poor compatibility with typically hydrophobic polymer matrices, resulting in weak interfacial bonding. To overcome this issue, appropriate surface modification through chemical treatments is essential. Such treatments enhance fiber–matrix compatibility and improve interfacial adhesion, making it one of the key challenges and active research areas in the field of composite materials.A review of the existing literature indicates that extensive research has been carried out on various natural fibers, as discussed earlier, focusing on their properties and the influence of chemical treatments on their performance. However, with respect to sea purslane fibers, which are relatively newly identified, no significant studies have been reported on their surface modification or the effects of chemical treatment on their properties.

Numerous investigations have explored the chemical treatment of natural fibers to enhance interfacial characteristics and induce morphological changes in the fiber structure [1, 8–11]. For instance, Srinivasa and Bharath [12] examined the impact of alkali treatment on the impact behavior of areca fiber reinforced polymer composites. Nayak and Mohanty [10] studied the effect of chemical treatment on water absorption, as well as the mechanical and thermal properties of areca sheath fibers. Similarly, Sunit et al. [13] investigated the durability, mechanical, thermal, and morphological characteristics of chemically treated rattan fibers. In another study, Jena, Mohanty, and Nayak [14] analyzed the influence of surface modification on the physical and thermal properties of vetiver fibers. Vetiver grass (Chrysopogonzizanioides) is a tropical plant that adapts well to a wide range of environmental conditions. It is commonly found in steep, sloping regions and near natural water bodies. With regard to the chemical treatment of vetiver grass fibers, only limited studies have been reported to date. In one such study, both untreated and alkali-treated vetiver fibers were effectively utilized as reinforcing fillers in polypropylene composites, resulting in improved material properties [15]. In another investigation, Vinayagamoorthy et al. [16] reported that chemical treatment of vetiver fibers enhanced the tensile, compressive, and impact strengths of vetiver/vinyl ester composites by 26.8%, 28.65%, and 30.44%, respectively.

Furthermore, Sutapun, Suppakarn, and Ruksakulpiwat [17] examined the characteristics of vetiver fibers before and after alkali treatment and observed that treated fibers exhibited higher decomposition temperatures due to the removal of hemicellulose and lignin components.Recent studies highlight the growing importance of composite materials in engineering applications. Ajithkumar et al. [21] emphasized the potential of natural fiber-reinforced composites in commercial sectors due to their sustainability and performance advantages. Dash et al. [22] analyzed flexural and vibration behavior of cenosphere-filled hybrids, showing improved structural response. Gurjar et al. [23] investigated luffa–cenosphere composites, demonstrating enhanced elastic properties. Lokesh et al. [24] reported improved mechanical and acoustic performance of calamus-based hybrids. Charan et al. [25] examined thermal enhancement in metal matrix composites, indicating improved heat transfer under forced convection.From the studies conducted thus far on vetiver fibers, it is evident that most research has focused on specific chemical treatments and their influence on particular properties. However, in the present study, an attempt has been made to investigate the enhancement of mechanical and thermal properties of epoxy composites reinforced with chemically treated vetiver fibers.

Materials and Methods:-

Materials:-

Vetiver grass leaves were collected from local areas near Kendrapada, Odisha, India. The collected leaves were thoroughly washed with water and dried to remove moisture. Fibers were extracted by manually peeling the vetiver leaves and were designated as raw fibers (Figure 1). The extracted fibers had lengths ranging from 15 to 20 cm and diameters between 0.342 and 0.984 mm. The chemicals used for surface treatment, namely sodium hydroxide (NaOH) and benzoyl chloride, were procured from the local market.

Figure 1.Vetiver grass and extracted vetiver fibers.

Methods:-

Surface treatment of fibers:-

In the authors' previous study [14], vetiver fibers were treated with various chemicals, and it was observed that benzoylation-treated fibers exhibited superior tensile strength compared to untreated and other chemically treated fibers. Therefore, in the present work, benzoylation-treated fibers were selected for composite fabrication.Initially, untreated vetiver fibers were immersed in a 5% NaOH solution at 80 °C for approximately 1 hour. The fibers were then cooled to room temperature, thoroughly washed with water to remove residual alkali, and subsequently oven-dried at 70 °C for 24 hours. These fibers were termed as alkali pre-treated fibers.The alkali pre-treated fibers were further immersed in a solution containing 10% NaOH and benzoyl chloride for about 15 minutes, followed by soaking in ethanol for 1 hour to ensure complete removal of unreacted benzoyl chloride from the fiber surface. The fibers were then washed with distilled water and oven-dried at 80 °C for 24 hours. During benzoylation, benzoyl groups replace the hydroxyl (–OH) groups present in the fibers, thereby reducing hydrophilicity and enhancing compatibility with the polymer matrix.

Fabrication of VF/epoxy composites:-

The composites were fabricated using the hand lay-up technique. A mold of dimensions 300 × 300 × 5 mm was prepared and coated with wax to facilitate easy removal of the composites after curing.Five different composite samples were prepared by varying the weight fraction of vetiver fiber reinforcement (10 wt%, 15 wt%, 20 wt%, 25 wt%, and 30 wt%). The fiber weight percentage was calculated with respect to the total weight of the composite specimen. For example, in the case of the 10 wt% composite, 10 wt% of vetiver fibers were uniformly distributed within the mold, followed by the addition of 90 wt% epoxy matrix prepared by mixing epoxy resin and hardener in a ratio of 10:1.After pouring the matrix into the mold containing the reinforcement, a load of 20 kg was applied, and the composite was allowed to cure at room temperature for 24 hours. Additionally, a 20 wt% untreated VF/epoxy composite was fabricated and used as a control sample for comparison purposes.

Testing methods:-

The mechanical properties of the fabricated composites, including tensile, flexural, and impact strengths, were evaluated in accordance with relevant ASTM standards. For each composite type, ten specimens were tested, and the average values were recorded for analysis.Tensile testing was performed using an INSTRON 3365 universal testing machine (Figure 2) with a maximum load capacity of 400 kN, following ASTM D638–14 at a crosshead speed of 2 mm/min. Dog-bone-shaped specimens with dimensions of 165 mm × 19 mm × 5 mm were prepared from the fabricated composite plates.Flexural tests were conducted in accordance with ASTM D790–10 using the same testing machine, maintaining a loading speed of 2 mm/min. Impact strength was determined using a Charpy impact testing machine (Model: IT-30 (D), Make: Fuel Instruments & Engineers Pvt. Ltd.) as per ASTM D256–10. Notched specimens with dimensions of 76 mm × 20 mm and a notch angle of 90° were used for the test.The chemical characteristics of the composites were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD) techniques.

Figure 2. INSTRON 3365 tensile testing machine.

Scanning electron microscope (SEM):-

The surface morphology of tensile-fractured composite specimens was examined using a HITACHI SU3500 Scanning Electron Microscope (SEM) to understand the fracture behavior and interfacial characteristics.

Results and Discussion:-

Table 1. Comparison of mechanical properties of various fabricated composites

Composites	**Tensile strength (MPa)**	**Flexural strength (MPa)**	**Impact strength (KJ/m^2)**
Untreated VF/epoxy			
20 wt%	18.22 ± 0.89	32.34 ±1.27	3.24 ± 0.45
Treated VF/epoxy			
10 wt%	25.45±1.45	45.03±1.09	4±0.35
15 wt%	27.24±1.36	48.25±1.75	12±0.87
20 wt%	35.67±2.03	65.61±1.36	17±0.76
25 wt%	32.39±1.98	58.05±2.02	14±0.90
30 wt%	30.55±2.14	52.21±1.09	13±0.66

Mechanical Properties:-

The mechanical properties of vetiver fiber-reinforced epoxy composites (Table 1) indicate an initial increase with increasing fiber loading, followed by a decline beyond the optimum fiber content of 20 wt%. The tensile and flexural strength values of the fabricated composites are plotted against varying fiber weight percentages in Figure 3(a) and 3(b).The results show that tensile strength increases with fiber loading up to 20 wt%, after which it decreases. This behavior can be attributed to improved fiber–matrix interfacial bonding up to the optimal fiber content, beyond which the bonding weakens, leading to reduced mechanical performance. A similar trend is observed in flexural strength, which also reaches a maximum at 20 wt% fiber loading before declining. The reduction in flexural strength at higher fiber contents is likely due to poor dispersion and fiber agglomeration within the matrix.The maximum values obtained at 20 wt% fiber loading are 35.67 MPa for tensile strength, 65.61 MPa for flexural strength, and 17 kJ/m² for impact strength. At higher fiber loadings, increased void content and stress concentration sites act as crack initiation points, resulting in reduced impact strength beyond the optimum fiber content.

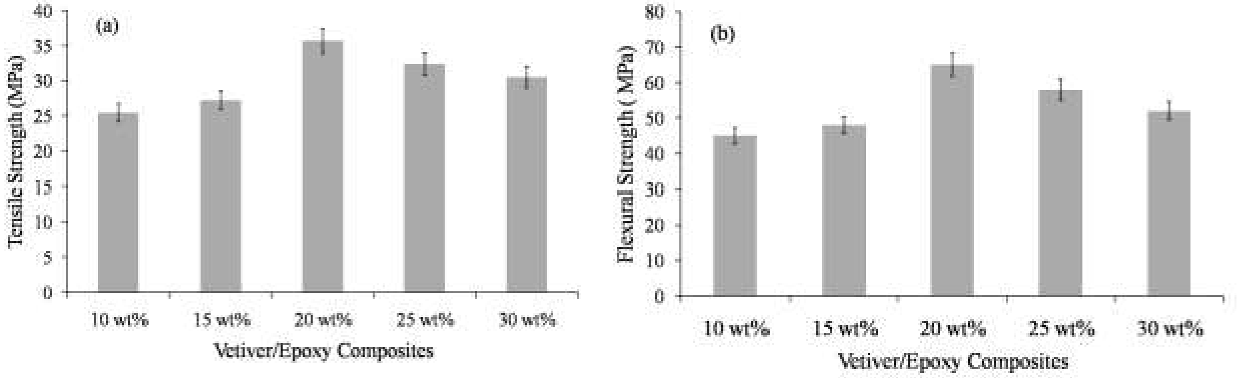

Figure 3.Mechanical properties of fabricated composites.

Fourier transform infrared (FTIR) spectroscopy:-

The FTIR spectra of both untreated and treated composites are presented in Figure 4. The spectra reveal characteristic stretching and vibrational peaks at wavenumbers of 815.5, 1020.5, 1562.5, 2812, 2875, and 3430.5 cm^{-1}. The broad peak observed at 3430.5 cm^{-1} corresponds to O–H stretching associated with hydroxyl (–OH) groups [18,19], and this peak remains present in both composites. Similarly, the band at 2875 cm^{-1}, observed in both cases, is attributed to C–H stretching vibrations [18,20].However, notable differences are observed in the treated VF/epoxy composite. A new peak appears at 815.5 cm^{-1}, which is absent in the untreated composite, indicating chemical modification of the vetiver fibers. Additionally, a peak at 2812.5 cm^{-1} is observed in the treated composite, while changes in peak intensity and structure suggest alterations in the chemical composition due to treatment.

Furthermore, the peak at 1020.5 cm^{-1}, corresponding to C–H wagging [19], exhibits increased intensity in the treated composite compared to the untreated one. These variations confirm that the chemical treatment has modified the fiber surface, leading to changes in the chemical structure and improved interaction between thefiber and the epoxy matrix.

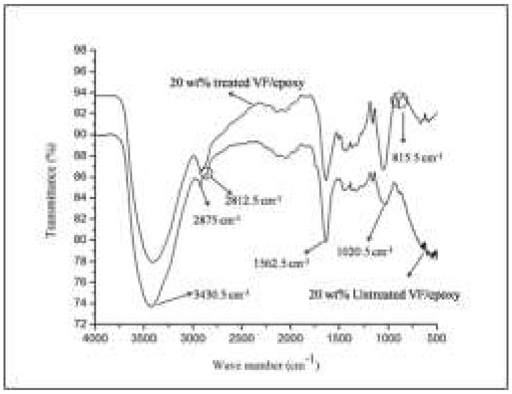

Figure 4. FTIR graph of vetiver/epoxy composite

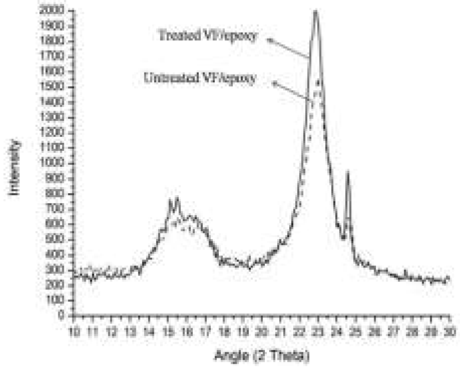

Figure 5. XRD graph of vetiver/epoxy composite.

X-ray diffraction (XRD):-

The X-ray diffraction (XRD) patterns of both untreated and treated composites are presented in Figure 5. The diffraction patterns exhibit two प्रमुख peaks located near $2\theta \approx 16°$ and $2\theta \approx 22°$, which correspond to the cellulose crystallographic planes I_{101} and I_{002}, respectively.For the untreated VF/epoxy composite, these peaks are observed at 15.84° and 22.42°. In contrast, the treated VF/epoxy composite shows a slight shift of these peaks toward lower angles. This shift can be attributed to the chemical modification of the vetiver fibers, which alters their crystalline structure and, consequently, affects the overall structural characteristics of the composite.The observed variation in peak position indicates that the chemical treatment has influenced the crystallinity and molecular arrangement of the fibers, thereby contributing to changes in the composite properties.

Scanning electron microscope (SEM):-

Scanning electron micrographs of the tensile-fractured surfaces were examined to evaluate fiber–matrix interaction and fracture behavior. SEM analysis was carried out on both treated and untreated VF/epoxy composites at 20 wt% fiber loading. The corresponding micrographs are presented in Figure 6(a, b).Figure 6(a), representing the treated composite, shows improved interfacial bonding between the fibers and the epoxy matrix, which correlates with its enhanced mechanical properties. In contrast, Figure 6(b), corresponding to the untreated composite, exhibits noticeable fiber pull-out along with the presence of voids. These features indicate weak interfacial adhesion between the fiber and matrix, which adversely affects the mechanical performance of the composite.

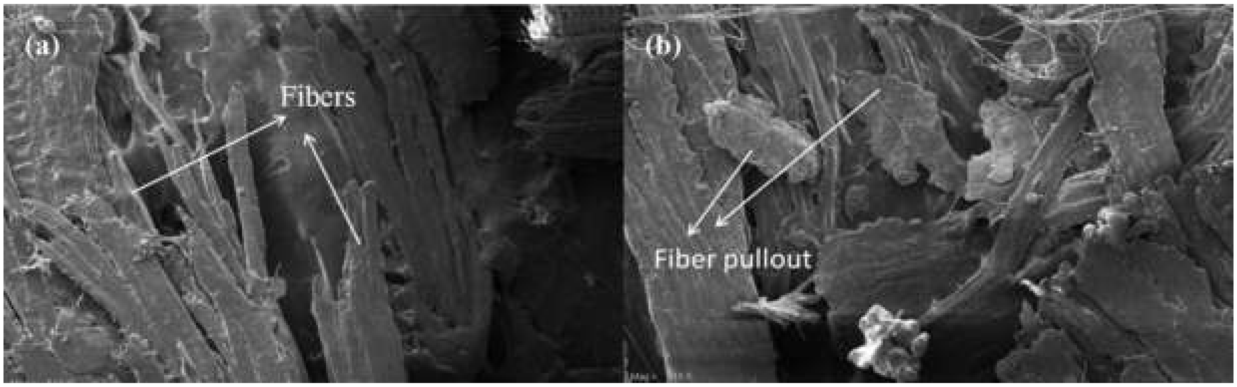

Figure 6. SEM of (a) 20 wt% treated (b) 20 wt% untreated VF/epoxy composite

Conclusions:-

In the present study, natural fiber-reinforced polymer composites were successfully fabricated by incorporating untreated and benzoylation-treated vetiver (VF) fibers into an epoxy matrix. The mechanical properties and chemical characteristics of the composites were systematically evaluated and compared with those of untreated fiber composites.The results indicate that chemical treatment of vetiver fibers significantly enhances the mechanical performance of the composites. The optimum performance was achieved at a fiber loading of 20 wt%, yielding tensile strength of 35.67 MPa, flexural strength of 65.61 MPa, and impact strength of 17 kJ/m². However, beyond this optimal fiber content, a decline in mechanical properties was observed, primarily due to reduced fiber–matrix adhesion and increased fiber agglomeration.Overall, vetiver fiber-reinforced epoxy composites demonstrate promising potential as sustainable and high-performance materials, particularly for packaging applications, owing to their improved mechanical properties.

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