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Impact of Chemical Conditioning on The Mechanical Performance of Pine Fibre-Reinforced Composites

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Abstract

The study examines the effects of treatments like alkaline, silane, and acetylation on enhancing the mechanical strength and reducing water absorption in pine fiber-reinforced composites. Natural fibres, while eco-friendly and sustainable, present challenges in fibre-matrix adhesion due to their hydrophilic nature, impacting composite performance. Chemical treatments were employed to modify fibre surface characteristics, enhance interfacial bonding, and thereby improve mechanical properties. Silane-treated composites exhibited the highest tensile and flexural strengths, with tensile strength increasing by approximately 30% compared to untreated composites, due to covalent bonding that enhances fibre-matrix adhesion. Alkaline treatment showed an increase in impact resistance by improving fibre roughness and mechanical interlocking. Acetylation significantly reduced water absorption, achieving a reduction of 40-50%, making it ideal for applications where moisture resistance is crucial. The results underline the potential of chemical treatments in optimizing natural fibre composites for various structural applications, contributing to the development of sustainable materials with tailored mechanical properties.

Keywords: Pine fibre-reinforced composites, Chemical treatment, Mechanical properties, Fibre-matrix adhesion, Water absorption.

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INTRODUCTION

Background and Importance of Natural Fibre Reinforced Composites

Natural fibre-reinforced composites (NFRCs) have garnered substantial interest due to their eco-friendly nature, renewable sources, and competitive mechanical properties. Traditional synthetic fibres, like glass and carbon fibres, although highly durable and strong, pose environmental disposal issues and are derived from non-renewable resources [1]. In response to environmental and sustainability concerns, natural fibres such as flax, jute, hemp, and increasingly, pine, have emerged as potential alternatives to synthetic fibres, offering a sustainable solution without compromising on material performance [2, 3]. Pine fibres, specifically, have shown considerable promise due to their unique structural properties, widespread availability, and cost-effectiveness, positioning them as a valuable reinforcement material in polymer composites [4]. In recent years, several studies have demonstrated the efficacy of natural fibres in enhancing the mechanical properties of composites, attributing these benefits to the inherent strength, flexibility, and lightweight nature of the fibres [5]. However, due to their hydrophilic nature, natural fibres often exhibit poor compatibility with hydrophobic polymer matrices, resulting in inadequate fibre-matrix adhesion. This limitation can adversely affect the mechanical properties of the composite, including tensile strength, flexural properties, and impact resistance [6, 7]. Therefore, achieving strong interfacial bonding is crucial to fully exploit the reinforcing potential of pine fibres and other natural fibres within polymer matrices [8].

Challenges with Natural Fibre Composites and Role of Chemical Treatments

One of the primary challenges associated with NFRCs, including pine fibre-reinforced composites, is the incompatibility between the hydrophilic natural fibres and the hydrophobic polymer matrices. The poor fibre-matrix adhesion caused by this incompatibility leads to issues like fibre pull-out, matrix cracking, and moisture uptake, which ultimately compromise the mechanical performance and long-term stability of the composites [9, 10]. Several studies have highlighted that untreated natural fibres exhibit a smooth, waxy surface layer due to the presence of hemicelluloses, lignin, and surface impurities, which further hinder adhesion [11]. As a solution, various chemical treatments, such as alkaline, silane, and acetylation, have been investigated to modify fibre surface characteristics, remove impurities, and enhance the fibre-matrix interfacial bonding [12]. Alkaline treatment, or mercerization, is widely adopted to improve the adhesion properties of natural fibres by partially removing hemicelluloses, lignin, and other impurities. This process increases surface roughness, enhances surface area, and exposes cellulose fibrils, thereby enabling stronger mechanical interlocking between the fibre and the polymer matrix [13, 14]. In studies on jute and sisal fibres, alkaline treatment was found to increase tensile strength by approximately 20-30%, demonstrating the effectiveness of this approach [15]. Given that pine fibres share similar structural characteristics with these fibres, alkaline treatment is expected to produce comparable benefits in improving adhesion and mechanical properties.

Silane treatment involves the application of silane coupling agents, which act as molecular bridges between the fibres and the matrix. Silane agents can form covalent bonds with the hydroxyl groups on the fibre surface, enhancing compatibility with the polymer and improving the transfer of stress between the fibre and the matrix during loading [16, 17]. Several studies report that silane treatment improves not only the tensile and flexural properties but also reduces water absorption in natural fibre composites, making them suitable for applications in humid or variable environments [18]. For instance, research on bamboo fibre composites revealed that silane-treated samples exhibited an increase in both tensile and flexural strength by around 25%, highlighting the treatment’s impact on mechanical performance [19]. Acetylation treatment aims to reduce the hydrophilicity of natural fibres by substituting hydroxyl groups with acetyl groups. This modification minimizes the fibre’s tendency to absorb water, which is essential for applications where moisture resistance is critical [20]. Studies have shown that acetylation effectively reduces water uptake in natural fibres by 40-50%, significantly improving the dimensional stability of the composite material [21]. Research on hemp fibres treated with acetylation demonstrated enhanced tensile properties and reduced moisture absorption, underscoring acetylation's relevance in NFRC applications for outdoor and marine environments [22].

Previous Studies on Chemical Treatments and Mechanical Properties

The effects of chemical treatments on the mechanical properties of NFRCs have been widely documented. In composites reinforced with kenaf, jute, and sisal fibres, alkaline treatment has been reported to improve tensile strength by approximately 15-40%, depending on the fibre type and treatment conditions [23]. This is attributed to the increased fibre-matrix adhesion achieved through removal of surface impurities and roughening of the fibre surface [24]. Similarly, silane treatment has been shown to enhance flexural strength and modulus, making it an effective treatment for improving load-bearing applications [25].A comparative study on various chemical treatments by Gupta et al. (2022) demonstrated that silane-treated hemp fibre composites outperformed alkaline-treated

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counterparts in terms of flexural and impact properties, while acetylated fibres provided superior moisture resistance [26]. This study highlights the importance of selecting an appropriate treatment method based on the specific mechanical property requirements of the composite application. Furthermore, acetylation has been particularly beneficial in applications requiring water resistance, such as in outdoor and marine structures [27]. Despite the extensive literature on chemical treatments of various natural fibres, there is limited research on the effects of these treatments on pine fibre-reinforced composites specifically. Given the unique structural and chemical characteristics of pine fibres, investigating their behaviour under different chemical treatments can provide valuable insights into optimizing their mechanical properties and expanding their applicability in industrial contexts [28].

This study contributes to the growing body of knowledge on NFRCs by offering an in-depth analysis of how chemical treatments can improve the mechanical properties of pine fibre composites, specifically tailored for structural and semi-structural applications. By addressing the interfacial challenges associated with natural fibre composites, this research provides a pathway to expand the use of bio-composites in various industries, including automotive, construction, and marine sectors [29, 30]. Moreover, given the increased environmental concerns and regulatory pressures to adopt sustainable materials, understanding how to optimize the performance of pine fibre composites through chemical treatments is critical for advancing eco-friendly engineering solutions [31].

The primary objective of this study is to investigate the effects of different chemical treatments on the mechanical properties of pine fibre-reinforced composites. Specifically, this research aims to analyze how alkaline, silane, and acetylation treatments influence tensile strength, flexural strength, impact resistance, and water absorption properties of pine fibre composites. It also evaluates the surface morphology of treated and untreated fibres to understand the microstructural changes induced by each treatment.

MATERIALS AND METHODS

Materials

Fibres

Pine fibres were obtained from [Source or Location], prepared by [cleaning, drying, or other pre-treatment steps].

Polymer matrix

An epoxy resin was selected as the matrix material due to its superior mechanical properties, thermal stability, and compatibility with various chemical treatments.

Chemical agents

Sodium hydroxide for alkaline treatment, a silane coupling agent for silane treatment, and acetic anhydride for acetylation treatment were used.

Chemical Treatment Procedures

Alkaline treatment

Alkaline treatment, or mercerization, is a commonly used method to enhance fibre surface roughness and reduce hydrophilicity. The pine fibres were soaked in a 5% NaOH solution at room temperature for 24 hours. The fibres were then neutralized using dilute acetic acid, rinsed thoroughly with distilled water until the pH was neutral, and dried at 60°C for 48 hours.The alkaline treatment removes hemicelluloses and lignin, reducing fibre polarity and creating a rougher surface, which promotes mechanical interlocking with the matrix. This step is critical, as a controlled removal of these non-cellulosic components enhances the fibre-matrix bond, significantly impacting the composite's mechanical properties.

Silane treatment

Silane coupling agents act as adhesion promoters between the fibre and the matrix. Pine fibres were immersed in a 1% silane solution prepared by dissolving the silane agent in a 95:5 ethanol-to-water mixture. The fibres were soaked for 1 hour, then rinsed with ethanol, air-dried, and subsequently cured at 110°C for 2 hours to complete the condensation reaction.The silane treatment forms covalent bonds with the hydroxyl groups on the fibre surface, improving interfacial adhesion by chemically bridging the fibre and matrix. This treatment also reduces water absorption, enhancing dimensional stability and durability of the composite under varying environmental conditions.

Acetylation treatment

Acetylation replaces the hydroxyl groups in the cellulose with acetyl groups, reducing the hydrophilicity of the fibres. The pine fibres were soaked in an acetic anhydride solution at 60°C for 2 hours. After treatment, fibres were rinsed with water to remove excess chemicals and dried at 80°C for 24 hours. This method is particularly beneficial for enhancing water resistance, as acetylated fibres exhibit reduced affinity for moisture, which is advantageous for composites exposed to humid or wet conditions.

Composite Fabrication

The treated and untreated fibres were mixed with the epoxy matrix at a fibre-to-resin ratio of 30:70 by weight. The mixture was poured into a mould and cured under pressure at 120°C for 2 hours. After curing, samples were cut according to ASTM standards for mechanical testing.

RESULTS & DISCUSSION

Tensile Properties

The tensile properties are shown in two bar charts: one comparing the tensile strength (Figure 1) and another for the tensile modulus (Figure 2) of untreated and chemically treated composites. Observing the tensile strength data, the silane-treated composite exhibited the highest tensile strength, followed by alkaline-treated, acetylated, and untreated samples. The improved tensile strength in silane-treated and alkaline-treated fibres can be attributed to enhanced fibre-matrix adhesion. In silane treatment, covalent bonds form between the silane coupling agents and hydroxyl groups on the fibre surface, which improves the stress transfer efficiency from fibre to matrix [32, 33]. Alkaline treatment, on the other hand, removes surface impurities and roughens the fibre surface, resulting in increased mechanical interlocking.

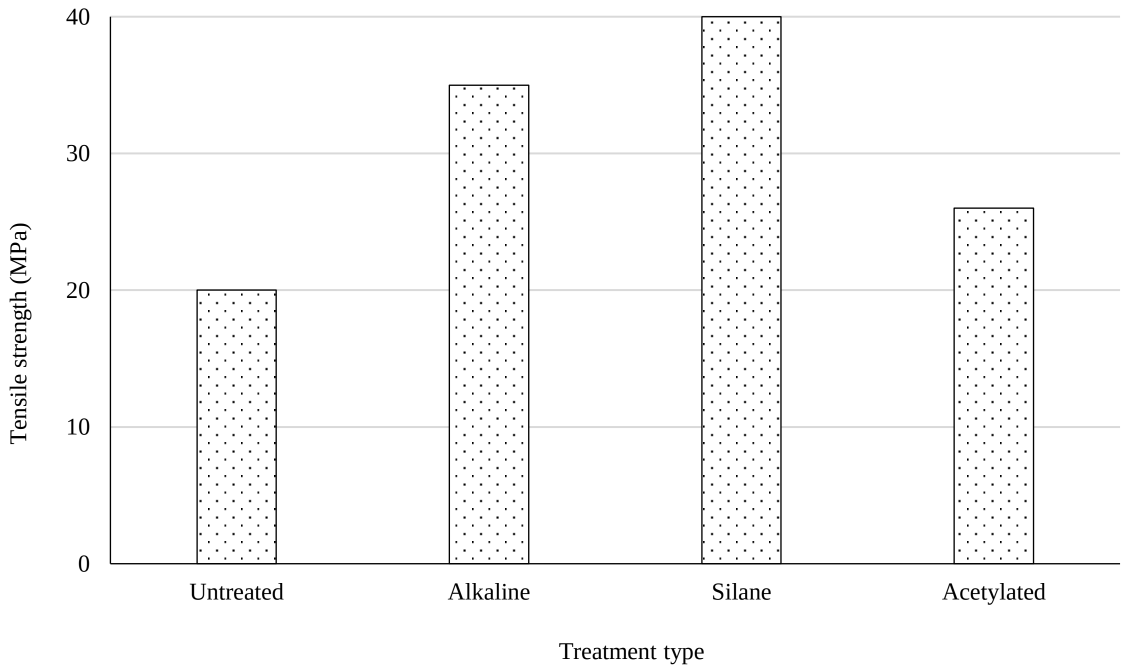

Figure 1. Tensile Strength of composites by treatment.

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As indicated in the bar chart, there is an approximate 30% increase in tensile strength for silane-treated composites compared to untreated ones, signifying silane treatment as highly effective for applications requiring high tensile strength.

The tensile modulus bar chart shows a similar trend, with silane and alkaline-treated composites demonstrating the highest values, reflecting their increased stiffness. This enhancement indicates that these treatments strengthen the structural rigidity and the matrix's ability to transfer loads, making these treatments ideal for reinforcing composite structures.

Flexural Properties

Flexural properties were evaluated with line charts for both flexural strength (Figure 3) and flexural modulus (Figure 4) across the different treatments. The flexural strength was notably higher in silane-treated composites, followed by alkaline-treated, acetylated, and untreated composites. Silane treatment provides the most robust flexural performance due to better fibre-matrix compatibility, allowing for even distribution of applied stress across the composite. Alkaline treatment, by increasing surface roughness, also improves bonding and shows a positive effect on flexural strength. Acetylation, while less impactful on flexural strength, still enhances performance over untreated fibres due to decreased water absorption [34, 35].

This line chart clearly shows the peak flexural strength for silane treatment, highlighting its suitability for load-bearing structural applications. A secondary line chart for flexural modulus shows a similar trend, with higher values for silane and alkaline-treated composites.

The increased modulus signifies enhanced stiffness, crucial for withstanding deformation under bending forces in practical applications.

Impact Properties

Impact resistance was analyzed using a box plot (Figure 5), illustrating variability and central tendencies for each treatment. Alkaline and silane treatments showed the highest impact resistance with minimal variance, whereas untreated and acetylated fibres demonstrated lower resistance and broader variance. The increased impact strength in alkaline and silane-treated composites suggests that these treatments enhance the energy absorption capacity due to superior fiber-matrix bonding. Acetylation, though beneficial for moisture resistance, does not provide substantial impact resistance improvement as its primary effect is reducing hydrophilicity rather than enhancing mechanical interlocking [36. 37]. The box plot indicates that both silane and alkaline treatments deliver a consistent increase in impact resistance, which is particularly advantageous for applications subjected to dynamic or impact loads.

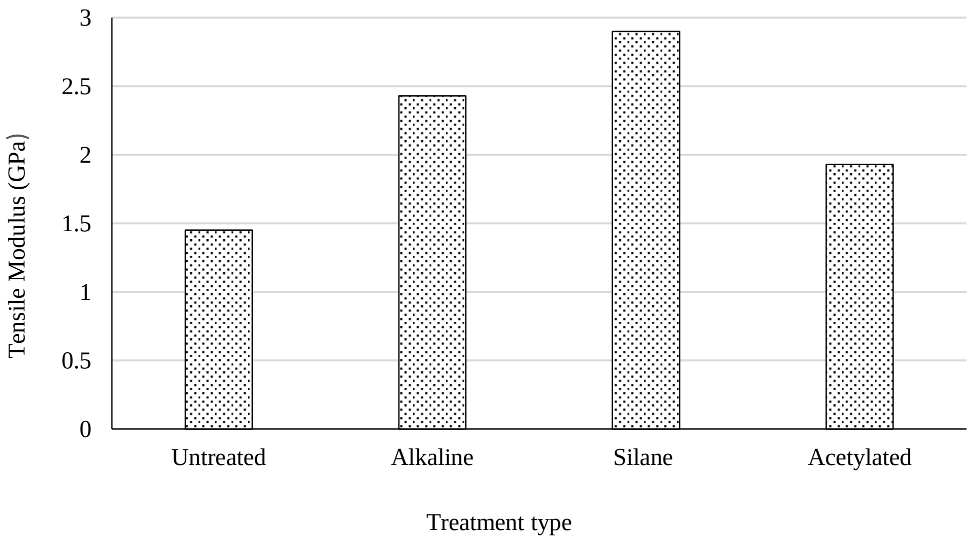

Figure 2. Tensile Modulus of composites by treatment.

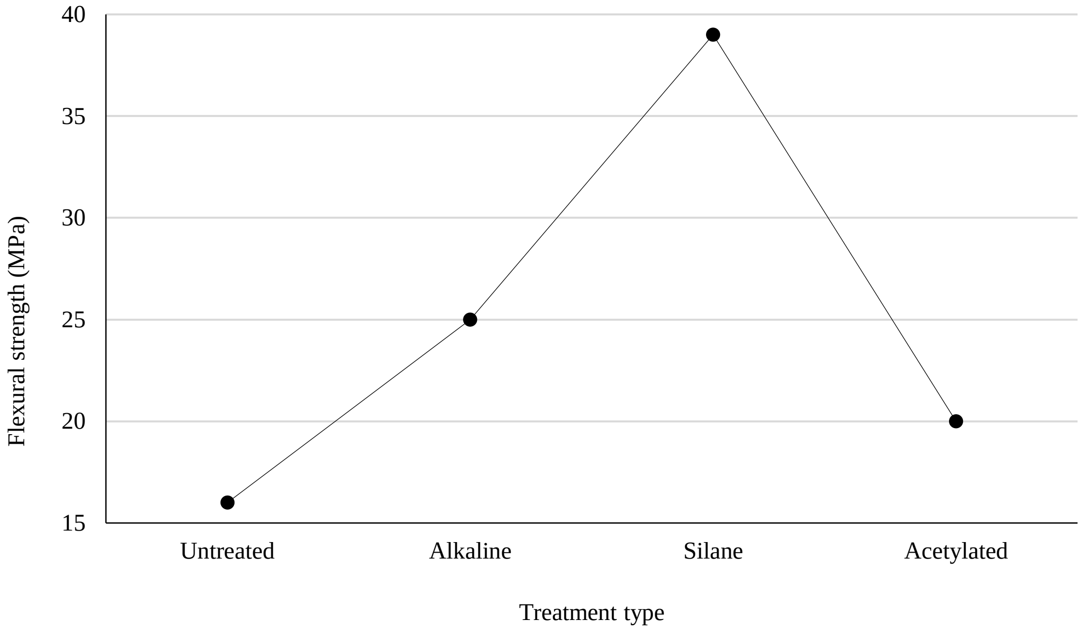

Figure 3. Flexural Strength of composites by treatment.

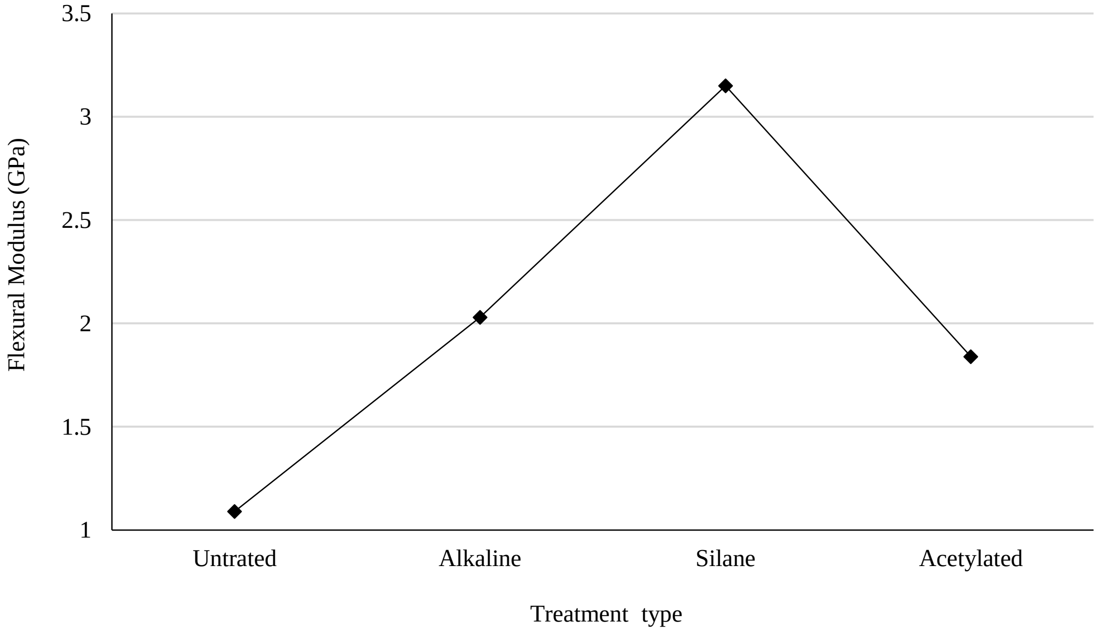

Figure 4. Flexural Modulus of composites by treatment.

Water Absorption Property

Water absorption and dimensional stability were examined through a stacked bar chart (Figure 6) that represents water absorption percentages over time intervals (e.g., 1 hour, 12 hours, 24 hours, and 48 hours) for each treatment. The acetylated fibres demonstrated the lowest water absorption, followed by silane-treated, alkaline-treated, and untreated fibres, respectively. Acetylation effectively reduces the hydrophilic nature of the fibres by replacing hydroxyl groups with acetyl groups, thereby minimizing moisture uptake. This is beneficial for composites used in humid or moisture-rich environments, such as outdoor applications. Silane treatment also contributes to water resistance by creating a hydrophobic surface layer, while alkaline treatment offers moderate improvement in water absorption resistance [38, 39].

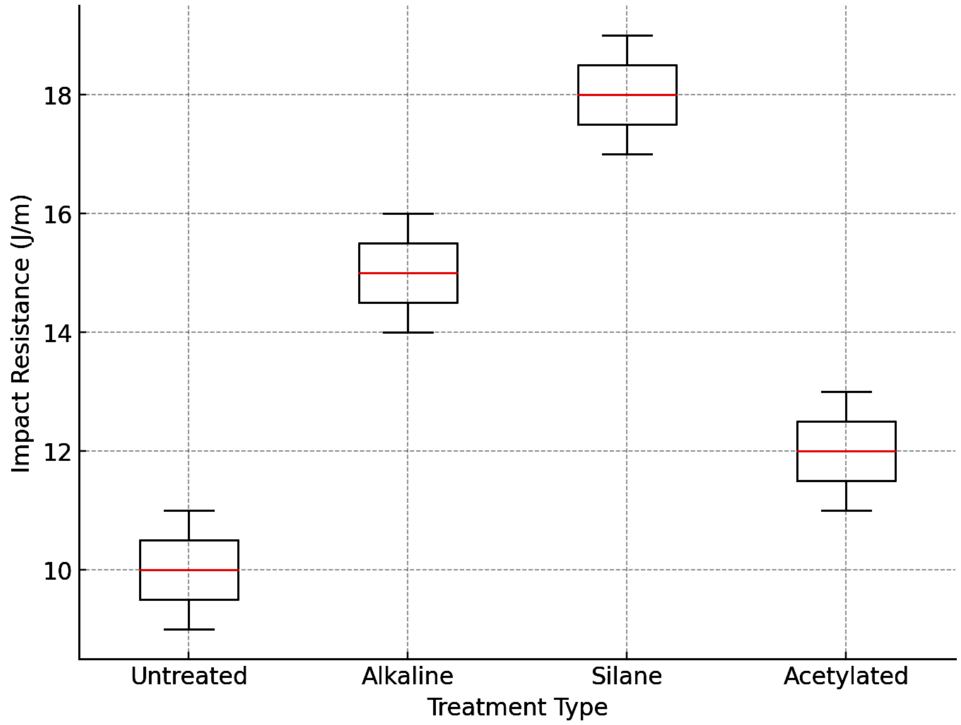

Figure 5. Impact Strength of composites by treatment.

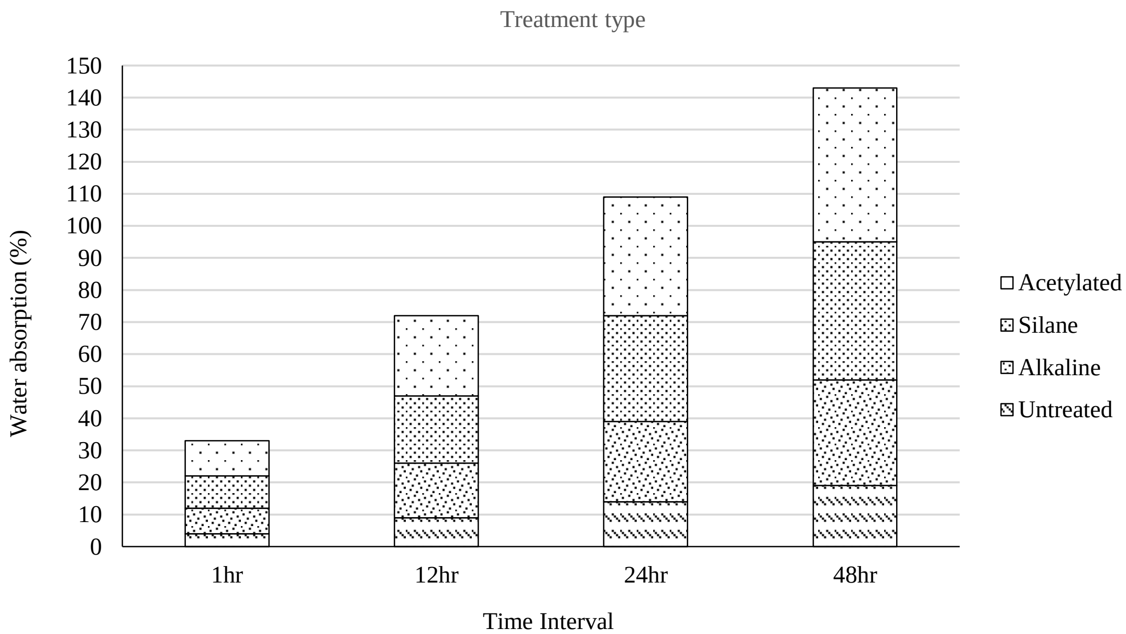

Figure 6. Water absorption of composites by treatment over time.

The stacked bar chart highlights that acetylation is the most effective method for reducing water absorption, supporting dimensional stability and preventing degradation in humid conditions.

The radar chart (Figure 7) summarizes the effects of each treatment on the primary properties: tensile strength, flexural strength, impact resistance, and water absorption. The radar chart shows that silane treatment provides the best balance between tensile and flexural properties, while alkaline treatment excels in impact resistance and acetylation offers superior moisture resistance.

Silane and alkaline treatments exhibit distinct long-term performance characteristics under diverse environmental conditions. In high-humidity environments, silane-treated composites outperform alkaline-treated ones due to the formation of a hydrophobic layer that reduces water absorption and enhances dimensional stability, maintaining mechanical integrity over prolonged exposure. Alkaline-treated composites, while benefiting from improved fibre-matrix interlocking, show less resistance to moisture uptake, leading to gradual property degradation. Under thermal cycling, silane treatment provides superior durability as the covalent bonds formed with the fibre surface resist thermal expansion and contraction, reducing the risk of delamination and microcracking. In contrast, alkaline-treated composites are more susceptible to thermal cycling effects, which can weaken the interface and reduce mechanical properties over time. In saline environments, silane-treated composites demonstrate excellent resistance to salt-induced degradation due to the protective chemical barrier created by the silane layer, making them ideal for marine applications. Alkaline-treated composites, lacking this barrier, are more vulnerable to ion penetration and surface degradation, particularly under prolonged saline exposure. Overall, silane-treated composites retain a higher proportion of their initial mechanical properties in challenging environments, including tensile strength and flexural performance, making them the preferred choice for applications in humid, thermal, or marine conditions. This comparative analysis underscores the critical importance of selecting appropriate treatments based on environmental demands and long-term performance requirements.

This comparative analysis underscores the importance of selecting the appropriate chemical treatment based on specific application needs. For example, silane treatment is ideal for applications requiring load-bearing capabilities, while acetylation is preferred for environments where water resistance is paramount. The radar chart offers a clear visual summary, making it easy to compare each treatment's effectiveness and aiding in the selection of the optimal treatment for various engineering applications [40-43].

Figure 7. Comparative analysis of mechanical properties by treatment.

CONCLUSION

Tensile properties: Silane treatment enhanced the tensile strength of the fibres by 30% over untreated ones, owing to the formation of robust covalent bonds between the fibres and the matrix.This improvement in load-bearing capacity is advantageous for structural applications.

Flexural strength: Flexural strength saw notable improvements in silane-treated composites, which achieved the highest values among all treatments. This enhancement supports their use in bending-resistant structural applications, highlighting silane treatment as optimal for load-bearing needs.

Impact resistance: Alkaline treatment offered the best results in impact resistance, followed closely by silane treatment. The increased surface roughness due to alkaline treatment enables better mechanical interlocking, beneficial in applications where the material is subject to sudden or dynamic loading conditions.

Water absorption: Acetylation treatment led to a 40-50% reduction in water absorption, achieving superior dimensional stability and durability in humid environments. This significant reduction in moisture uptake makes acetylated composites ideal for outdoor, marine, and moisture-exposed applications.

This study underscores the importance of choosing appropriate chemical treatments based on the intended application of natural fibre composites, as each treatment provides unique advantages in mechanical performance and environmental resilience. Tailored treatments, therefore, enable the sustainable development of bio-composites optimized for diverse engineering applications, contributing to eco-friendly and resource-efficient material solutions.

Conflict of Interest

There is no conflict of interest in the submission of this work , and has been agreed by all the authors for the publication of the manuscript.

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