

Development and Evaluation of Sodium Hydroxide-Treated Jujube Pit Reinforced Epoxy Composites

M. Amala Justus Selvam ^{1*}, Joseph Benny Kudiyirican ², T.M. Inbamalar ³

Abstract

The automotive sector has experienced a tremendous transformation, propelled by the increasing demand for materials that provide a balance between mechanical performance, economic efficiency, and environmental sustainability. In accordance with worldwide sustainability goals, the trend has moved toward the utilization of lightweight, recyclable, and biodegradable materials in modern vehicle architecture. Natural composite materials, which are derived from renewable resources such as seeds, leaves, and tree barks, have gained considerable attention due to their ability to replace conventional synthetic reinforcements. Among them, jujube pit fibres represent a potential, underexplored alternative. The current study deals with the fabrication of jujube pit-fibre reinforced epoxy composites through the hand-layup technique. To enhance the interfacial bonding and mechanical properties, the natural fibers were treated with sodium hydroxide solution. A thorough experimental study is carried out to investigate the tensile strength, flexural properties, and impact resistance of the treated and untreated samples. This work emphasizes the ability of sodium hydroxide-treated jujube pits as an environmentally friendly and cost-effective reinforcement for high-performance polymer composites with potential application in the automotive and allied industries for structural and semi-structural applications. The findings offer a compelling solution for decreasing reliance on synthetic fibres and enhancing the usage of renewable and sustainable materials in automotive and structural applications.

Keywords: Natural fibre composites, jujube pits, sustainable materials, sodium hydroxide treatment and epoxy composite

INTRODUCTION

Natural fibres have been widely replacing synthetic fibres in many engineering applications in recent years. Natural fibre-reinforced polymers are gaining popularity for sustainability development. Industrial awareness of environmental issues motivates these sectors to develop innovative materials made from natural, renewable, or reusable resources [1]. One of the most important parts found in the

cylinder head of an internal combustion engine is the inlet valve, along with the seat insert. Its primary function is to regulate the engine's intake of working fluid so that the combustion chamber and manifold are properly sealed [19]. Polymer industries are now showing more interest in natural composites, especially bio resources. In particular, Nylon 6 reinforced at different weight percentages (5%, 15%, and 25%) with silicon carbide (SiC) and boron carbide (B4C) is the focus of this research. This small selection of materials might not accurately reflect the wider range of polymer matrix composites that are offered by the industry, which could limit the findings application to other composite materials [20]. Because of their natural availability, wider resources, and cost effectiveness, these polymers are widely adopted by many industries. Furthermore, manufacturers are

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embracing this technology in order to fulfil consumer demands, which encompass a rising requirement for applications with superior performance and a heightened awareness of environmental issues associated with traditional materials. The utilization of natural based composite resources has had a notable influence on the polymer composite sector due to their ability to be converted into products that are more environmentally sustainable [2]. When weighed against other naturally occurring fibers now in widespread use like jute, coir, and sisal, jujube pits have certain advantages in cost-effectiveness and environmental sustainability. For one, the pits are reasonably inexpensive and renewable materials that minimize waste resulting from fruit processing. Moreover, alkaline treatment can greatly enhance the mechanical characteristics of jujube pits, therefore these could be regarded as a major substitute for conventional natural fibers in lightweight composite uses. However, further studies are required for comprehensive benchmarking of their performances in diverse engineering applications.

In order to strike a balance between the fibre's performance and its cost, hybrid fibres are often used. The same fibres can be modified to have a wide range of chemical compositions, densities, and mechanical qualities. Therefore, a fibre can be cost-effectively designed to meet the requisite specifications. A composite having different elastic characteristics in the main stress directions can be created, for instance, by combining carbon fibres and aramid in the weft and warp arrangement [3]. The physical and chemical qualities of natural fibres differ significantly with age, geographical location, and age. Statistical analyses of strength are crucial in the design and evaluation of composites made from natural fibres, as these differences must be taken into account. Hydrophobic and anisotropic, natural fibres are the best choice for a variety of applications. These characteristics increase the difficulty of designing and producing bio composites. In order to maximize the bond fibre-matrix, it is necessary to chemically treat natural fibres to regulate their water content. Precision tests and statistical evaluations should be used to objectively assess the vital properties of bio-composites, through the goal of providing trustworthy input parameters for computer modelling and simulation of components built from such materials [4].

The properties of natural fibre reinforced polymer composites can vary greatly depending on the fibre type, fibre source, and fibre structure of the natural fibres used in the composite. In order to determine the impact of chemical treatments, research was conducted on the thermal and mechanical properties of natural fibre reinforcing a wider plastic composites. The weaker mechanical qualities, higher water absorption, and reduced fire resistance of reinforced polymer all contributed to a reduction in their usefulness. Therefore, it is unavoidable to treat natural fibres differently to achieve the desired results. Frequently, these modifications centre on the integration of efficient groups capable of undergoing reactions with the fibre structures, hence modifying their composition. The compatibility issues arising from the interaction between the fibre and polymer matrix have been significantly ameliorated through the use of fibre modifications. These changes effectively diminish the inherent moisture absorption capacity of natural fibres[5].

Typically, natural fibres necessitate many treatments in order to address the aforementioned limitations, namely for the purpose of improving the bond between the fibre and the compounds. Several methods, such as treating the material with a water repellent, using a bonding agent, or subjecting it to heat, have been detailed. These procedures aim to affect the surface morphological and topological features of the fibres, as well as their roughness and water absorption index. Consequently, there has been a documented increase in research and technology endeavors aimed at enhancing the quality of crops and the performance of fibres from both technical and economic perspectives. The objective of these efforts is to offer novel solutions and applications [6]. Enhancements in the tensile characteristics of composites can also be attained by means of chemical addition on the fibres. Plasma treatment enhances inter bonding through two mechanisms: augmentation of hydrophobicity on fibre surfaces and amplification of surface roughness. The results of untreated bio-composites and composites derived from fibres treated with a sodium hydroxide solution were evaluated, revealing that the treated composites exhibited reduced hardness but increased tensile strength[7]. Alkali treatment reduces the fibres' ability to absorb moisture, resulting in composites with higher flexural modulus, strength, and stiffness. The enhanced adhesion and bonding strength between the chemicals and fibres seen in these

studied fibres alter the mechanical properties of composites. [8]. This paper used hand layup techniques to create a natural composite out of jujube pits and epoxy resin. To enhance the properties, alkaline treatment was done, and the results of the tensile test, flexural test, and impact strength test were compared with untreated composites.

MATERIALS AND METHODS

Consumption-grade fibre is present in jujube date kernels. After being extracted from the tree, the dates' seeds were subsequently cleaned with distilled water. Subsequently, the seeds undergo a drying process. The pits undergo fragmentation, resulting in smaller fragments, among which a small amount is deemed edible.

The fibre obtained from jujube pits is considered a natural fibre, as it is derived from the jujube pits process seen in the Figure 1. During the production process, jujube fruits are harvested from the jujube tree and subsequently subjected to a water-based washing procedure. The fruits that have been gathered are divided into two components: the pits and the layers. The excavations have been desiccated and afterwards subjected to treatment with a solution containing 1% sodium hydroxide (NaOH). The aforementioned procedure persists in distilled water for a duration of 2 to 3 hours. The ratio between the number of pits and the amount of water is 2:3. In the conducted studies, the removal of pollutants is achieved through the process of clearing them in the pits. Subsequently, the treated pits undergo a process of desiccation and are subsequently rinsed with water to ensure cleanliness. Once more, the treated pits undergo a drying process, whereas the untreated pits are subjected to a cleaning procedure using distilled water, followed by drying. The shells in both the treated and untreated pits were broken into small pieces and ground into flour. The purpose of the broken pieces and flour mill is to produce fibre powder, which is afterwards separated and sieved into particles of uniform size [9,10].

The hardener serves as a curative agent for epoxy or fiberglass materials. Epoxy resin necessitates the presence of a hardener in order to commence the curing process. This hardener, sometimes referred to as a catalyst, is responsible for inducing the hardening of the adhesive upon its combination with the resin. The final features and compatibility of the epoxy coating for a given environment are determined by the precise selection and combination of the epoxy and hardener components [11,12]. The seeds undergo a treatment process (Figure 2) involving immersion in a solution consisting of 1% sodium hydroxide (NaOH) and 99% JP mixed with water for a duration of 2 hours. The ratio of seeds to water is 2:3. The process of removing contaminants from seeds is essential for their maintenance. The seeds that have undergone treatment are subjected to a drying process. Once again, the seeds were rinsed thoroughly using distilled water. The treated seeds undergo a drying process once again. The seeds that have not undergone any treatment are subjected to a cleaning process using distilled water, followed by a drying procedure. The pits undergo fragmentation, resulting in smaller fragments, among which a limited part is deemed suitable for consumption [13,14].

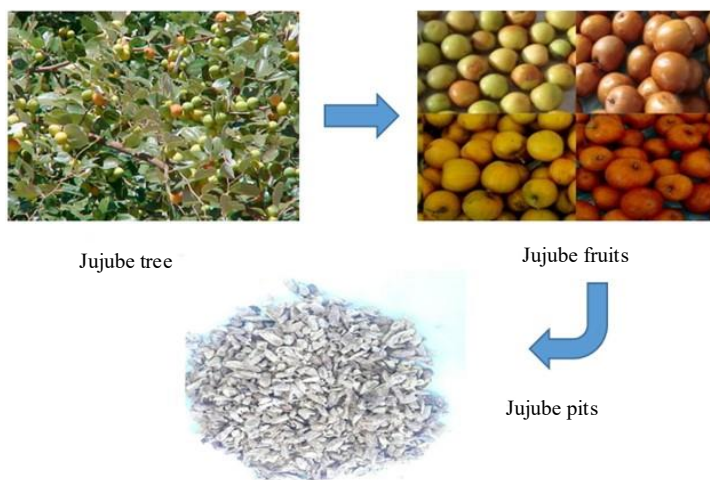


Figure 1. Jujube pits process.



Figure 2. Jujube pits treatment process.

The sieving method is a commonly used and straightforward approach. The approach is commonly employed for particle size analysis. The sieve diameter utilized in this project adheres to the specifications set by the International Standards Organisation (ISO), measuring 320 microns. The jujube date pits powder and epoxy resin were combined in a 3:7 ratio, with 30% of the mixture consisting of jujube date pits powder and 70% consisting of epoxy resin. These components were thoroughly mixed together to create the desired final product with the appropriate dimensions. The composite materials utilized in this study were manufactured using the hand layup procedure. The base plate is securely affixed within the frame to facilitate the construction process of natural fibre compounds. These compounds consist of a combination comprising 70% resin hardener and the remaining portion is composed of natural fibres. The composite of resin and hardener is introduced into the designated mould. The prepared natural fibres are introduced into the resin hardener mixture in a random manner, ensuring a complete absence of gaps. The roller is rotated within the mould. Once again, the mould is filled in a systematic way with the subsequent layer, while the fibres are poured in a random manner. The aforementioned procedure is executed in a concurrent manner until the desired vertical dimension of the mould is achieved. The lid is affixed to the upper portion of the frame in order to uniformly distribute the load throughout the mould. The apparatus is stored in a dry location for a duration of 24 hours. After a period of 24 hours, the mould is removed from the pattern, so completing the production process of the natural fibre compounds.

RESULTS AND DISCUSSION

The manual hand layup procedure is employed in the preparation of composite overlays. Two plane glasses were selected for the experiment, and a layer of fax was put to both the top and bottom surfaces of the glass. The epoxy resin and hardener mixture is fully applied, incorporating the addition of Jujube pits powder. A mixture consisting of 30% epoxy resin and 70% jujube powder was poured between two galas surfaces and subsequently crushed. The specimens are produced through the grinding of date pits. The specimens are prepared at the Indian Institute of Technology Chennai. The specimens are prepared in accordance with the ASTM-D 638 standard Figure 3 and Figure 4.

The present study employs the manual hand layup method for the preparation of composite laminates. Two plane glasses were selected for the experiment, and a layer of fax was put to both the top and bottom surfaces of each glass. The LY556 epoxy glue and HY951 hardener mixture has been fully applied. The specimens are produced using ground date pits. The combination laminate composite is fabricated through the sequential arrangement of individual fibre layers. The initial layer consists of a combination of ground date pits fibre and an epoxy and resin mixture that is applied. The specimen was prepared according to the ASTM-D 638 standard. The fabrication of the fibre composite material was ultimately completed, and in accordance with ASTM standards, three identical specimens were made for each test and afterwards subjected to the respective tests[15]. According to the ASTM D638 standard, the dimensions of the components used in the samples are $300 \times 25 \text{ mm}^3$. The experimental procedure involves subjecting the specimen to a controlled mechanical load until it reaches its point of failure. The range of stiffness exhibited by composite materials is demonstrated within the aforementioned context.

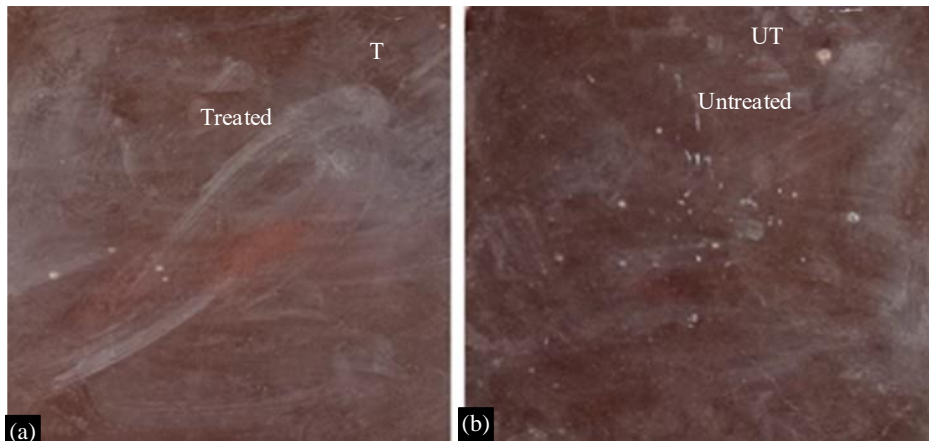


Figure 3. Composite plates (a) Treated (b) Untreated



Figure 4. Jujube fibre samples.

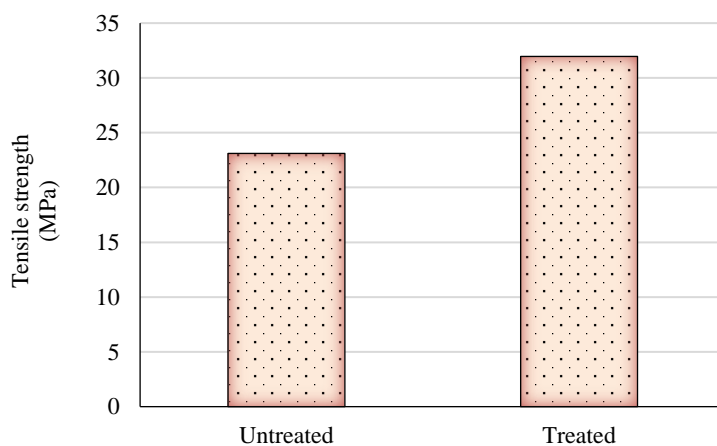


Figure 5. Comparison of tensile strength.

The data presented in this study illustrates the different levels of elasticity observed in materials that have undergone treatment compared to those that have not. The tensile strength of the treated composite specimen is observed to be much higher when matched to the untreated compound specimen as presented in Figure 5 [16].

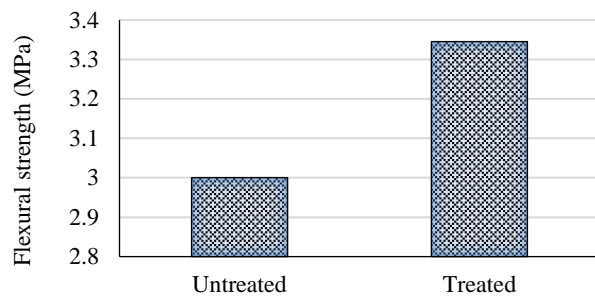


Figure 6. Comparison of flexural strength.

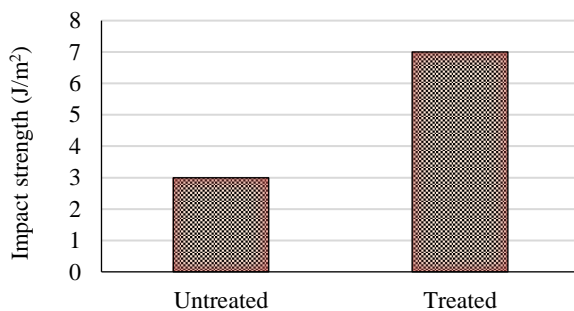


Figure 7. Comparison of impact strength.

The flexural specimens have been prepared in accordance with the ASTM-D790 standard. The 3-point flexure test is widely recognized as the predominant method for evaluating the flexural properties of composite materials shown in Fig. 6. The results show the variation in flexural strength between treated and untreated composite samples at maximum loading circumstances. When compared to the untreated composite's flexural strength of 3 MPa, the treated composite's value of 3.345 MPa is a significant improvement. [17]

According to the ASTM-D256 standard, the test pieces for the impact test have been made to the measurements as shown in Figure 7. As part of the testing process, the samples fixed into the testing machine and hit by the pendulum until it breaks. By employing the Charpy impact test, it is possible to efficiently estimate the energy required to fracture the material. This estimation may be utilized to assess both the durability of the material and its yield strength[18].The previous section presents the range of impact strength exhibited by the composite materials. This study demonstrates the diverse range of impact strength through the utilisation of distinct composite cases. The provided composite sample exhibits a diminished level of Impact strength when composed of untreated Jujube pits. Conversely, the individual specimen demonstrates a heightened level of Impact strength when composed of treated Jujube pits.

The impact of water osmosis is of paramount importance when the material, upon its utilisation for various applications, comes into contact with water. The consideration of water ingestion limit is an additional fundamental component to be taken into account when assessing the influence of water on composite materials. The composite guides utilized for the clamminess maintenance test were initially subjected to air drying at room temperature. Subsequently, the aforementioned composite models were immersed in purified water at ambient temperature for a duration of approximately five days. During regular intervals, the models were removed from the water and cleaned using filter paper to remove surface water. Subsequently, they were weighed using an automated adjustment with a precision of 0.01 mg. The models were submerged in water in order to facilitate the ongoing maintenance process until the saturation threshold was reached. The weighing process was conducted within a 30-second timeframe in order to prevent errors caused by evaporation[9].

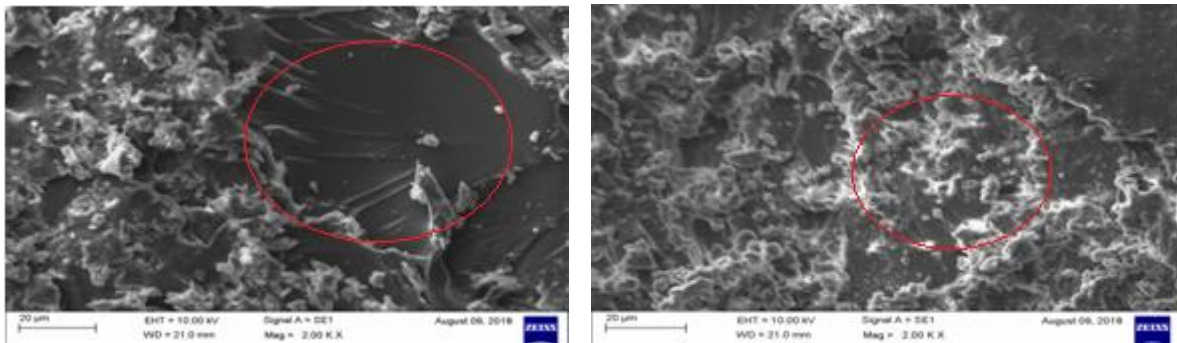


Figure 8. SEM analysis of jujube pits.

To analyse the microstructure of the jujube pits samples, scanning electron microscopy (SEM) images were obtained for both the treated and untreated samples. Based on the electron micrograph depicted in Fig. 8, the captured images pertain to treated jujube pits. Subsequently, an image analysis was conducted to ascertain the enhancement in strength of the treated jujube pits and the extent of their favorable interaction with the epoxy matrix. Furthermore, the investigation aimed to elucidate the consequential impact on the mechanical properties. As depicted in Figure 8, the observed phenomenon led to an increased probability of pressure redistribution from the framework to the fibre, thereby leading to an enhancement in the supportive efficacy. The deficient mechanical properties of untreated jujube pits, as depicted in Figure 7, can be attributed to the insufficient interaction between the jujube pits and the epoxy matrix [7].

CONCLUSIONS

The conducted investigations provide evidence that the mechanical features of Jujube pit fibre polymer composites, specifically their tensile, flexural, and impact properties, exhibit enhancement with treatment, as compared to the mechanical capabilities of untreated composites. This study thoroughly investigates the effects of chemical treatments on enhancing the interfacial matrix-fibre adhesion in fibre polymer composites, leading to notable improvements in their physical properties. The composite frameworks utilized in this assessment exhibited rough surfaces, characterized by a significant level of debonding and an inadequate bonding of jujube pit particles. These properties were deemed crucial for the purpose of this inquiry. A notable finding indicates that the jujube pits that underwent treatment exhibited a decrease in water absorption when compared to those that were not treated. Insufficient interfacial adhesion led to a rise in the occurrence of little gaps on the liquid's surface, thereby leading to an increase in water absorption. Future study can be focused on advanced techniques of manufacturing, such as resin transfer molding or compression molding to improve the scalability and process efficiency of jujube pit-reinforced composites for industrial uses.

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