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Fabrication of transparent wood by impregnation of epoxy resin into modified balsa wood for application in energy saving materials

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ABSTRACT

Transparent wood, an innovative material, merges optical and mechanical properties for energy-efficient light transmission. Removing pigments like lignin is vital for enhancing light transmission and refractive properties, creating a robust framework. Polymers with suitable refractive indices are integrated into the pigment-treated wood to optimize light transmission while preserving refractive properties. This study introduces a method for producing transparent wood from balsa wood, prized for its lightness and stability, by stabilizing its structure in epoxy. FT-IR spectroscopy verifies stable structural bonds in the wood framework treated with lignin and epoxy composites. Optical analysis indicates light transmittance ranging from 43% - 70%, depending on the original wood thickness. Mechanical tests demonstrate transparent wood's superior elastic moduli compared to lignin and natural wood, indicating enhanced strength. SEM analysis illustrates epoxy filling the hollow tubes. These findings emphasize transparent wood's potential to supplant conventional materials in construction and solar energy panels, garnering interest from building materials and renewable energy sectors.

Introduction

Wood is a renewable resource, and an environmentally friendly material. It exhibits good mechanical properties including high strength, low thermal conductivity, and attractive biodegradability [1,2]. The natural composition of wood comprises cellulose, hemicellulose, and lignin, which are arranged in a multi-layered and hierarchical structure. Wood inherently possesses opacity and specific coloration due to chromophoric groups in lignin and other substances that strongly absorb visible light within the 380–780 nm wavelength range [4,5].. Pigments in wood, particularly lignin, constitute a significant portion of the wood tissue and can affect the material's light transmission and refraction. Lignin, acting as a binder for cellulose fibers, helps stabilize the natural wood structure. When lignin is removed, the cellulose-based material framework serves as the base for creating transparent wood [6]. Lignin removal from wood can be achieved through various methods. Scientists at the Vietnam Forest Industry Research Institute [7] have investigated the lignin removal process using $NaOH/H_2O_2$ on manglietia conifera wood and bodhi wood samples to produce transparent wood cores. To ensure stability and connectivity within the cellulose fibers, polymers with suitable refractive indices are selected to impregnate into the treated wood

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framework, forming a transparent wood composite product. Li et al. [8] demonstrated the fabrication of transparent wood through the impregnation of polymerized methyl methacrylate. Additionally, Li et al. used the H_2O_2/NaOH treatment method to remove lignin, followed by the infusion of polyvinyl alcohol (PVA) to fabricate transparent wood [9]. Thus, the fabrication process of transparent wood involves two primary stages: (i) the removal of lignin from natural wood and (ii) the impregnation of polymers with suitable refractive indices, resulting in transparent wood with specific light transmitting properties. The integration of a polymer matrix with modified wood creates a material that demonstrates high light transmission, enhanced mechanical properties compared to traditional glass, and reduced glare. These attributes make transparent wood a promising alternative to glass with potential applications as light-transmitting material in energy-saving buildings. Furthermore, this material can be used as wall or roof material to provide light, effectively reducing lighting energy consumption for buildings [10].

This study investigates the fabrication of transparent wood using natural balsa wood of varying thicknesses. The natural wood is modified to form a porous structure through the removal of lignin, followed by impregnation with epoxy at a specific ratio. The resulting transparent wood/epoxy samples are systematically analyzed and characterized in terms of their chemical structure, morphology, light transmission capability, and mechanical properties. The fabrication process enables control over the material's light transmission, haze, mechanical properties. Transparent wood demonstrates potential as a sustainable replacement for conventional glass in construction, contributing to the efficient utilization of natural resources.

Experimental and Research Methods

Materials and Chemicals

Balsa wood (Ochroma Pyramidale) with a density of 0.113g/cm³ was supplied by San Ho Timber Pte Ltd, Singapore. Sodium chlorite ($NaClO_2$, 80%) was obtained from Sigma Aldrich, while sodium acetate (CH3COONa.3H_2O, 98%) and acetone (CH_3COCH_3, 99.6%) were sourced from Xilong Company, China. Acetic acid (CH_3COOH, 99.5%) and ethanol (C_2H_5OH, 96%) were supplied by Duc Giang Chemicals Joint Stock Company, Vietnam. Low molecular weight bisphenol A epoxy resin and polyetheramine curing agent were sourced from JDiction Chemical Company.

Lignin Removal in Wood

Step 1: Wood Material Treatment. Commercial wood was cut along the grain from commercial wood into small pieces with a cross-section of 30x30 (mmxmm), and thicknesses ranging from 1 to 3 mm. The wood samples were dried at 100°C for 24 hours to remove moisture.

Step 2: Preparation of Lignin Treatment Solution. The lignin treatment solution was prepared from a 3.5 wt% $NaClO_2$ solution and a 0.006M CH_3COONa / 0.1M CH_3COOH solution with a 1:1 volume ratio, having a pH of ~4.5 [11].

Step 3: Lignin Removal. The dried balsa wood samples were immersed in the lignin treatment solution at a specific ratio, heated to 80°C, and maintained at this temperature for 6–10 hours.

Step 4: Sample Washing. After lignin treatment, the wood samples were washed three times with hot distilled water to remove residual chemicals from the wood surface. Subsequently, the samples underwent further treatment to remove impurities through two steps: (i) washing with 96% ethanol solution, and (ii) washing in a 1:1 volume ratio ethanol/acetone solution. Finally, the samples were kept stable in an acetone solution.

Fabrication of Transparent wood

After lignin treatment, the wood exhibited a porous structure resulting from the substantial removal of lignin. These wood samples were placed in a vacuum system and dried for 30 minutes.

Epoxy resin was prepared by mixing bisphenol A and polyetheramine curing agent in a 3:1 volume ratio, followed by stirring until a homogeneous mixture was obtained. To fabricate transparent wood, the epoxy mixture was poured into a mold containing the lignin-treated wood samples at a ratio of 35 wt% relative to the original wood material and maintained under vacuum for 30 minutes to ensure through impregnation. Subsequently, the samples were removed, sandwiched between two silicone sheets, and dried at 70°C for 24 hours, yielding the transparent wood product. The process diagram for creating transparent wood is shown in Figure 1.

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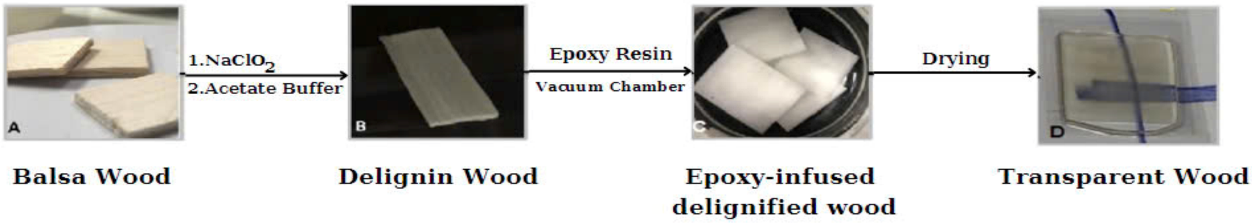

Figure 1. Transparent wood fabrication process

The symbols for the wood samples in the experimental process are summarized as follows:

- Balsa wood
- Delignifield wood
- Transparent wood

*X-1mm, -2mm, -3mm indicates the thickness of the raw wood or corresponding modified wood samples.

Product Characterization Analysis

The characteristics of the wood during the modification process were analyzed using various characterization methods. The infrared spectroscopy method for determining bonds in the wood structure before and after treatment was analyzed using Fourier-transform infrared spectroscopy on an (IRAffinity-1, Shimadzu) device. The FTIR spectra were recorded using the KBr method, with a measurement range from 4000 - 500 cm^{-1}. Light transmittance was determined using a Shimadzu UV-2600 spectrophotometer with a wavelength range from 300 - 800 nm. The surface structure of the samples was determined using FE-SEM: Regulus 8100 (Hitachi). Tensile strength and elastic modulus were evaluated according to ASTM D638 standards on a Zwick Z2.5 (Germany) measuring machine.

Results and discussion

Analysis of Light Transmittance and Transparent wood

In this study, the material used was balsa wood. The raw characterized by its light-yellow color underwent two stages: (i) lignin treatment using $NaClO_2$ solution in CH_3COONa/CH_3COOH solution, (ii) epoxy impregnation to create transparent wood. After the lignin removal process, the wood sample exhibited a cloudy white appearance, indicating the successful removal of lignin and chromophoric groups. This process resulted in a porous structure characteristic of lignin-free wood. The transparent wood/epoxy sample was obtained following the epoxy impregnation stage. The final product demonstrated enhanced light transmission properties compared to natural wood, as shown in Figure 2.

Figure 2. Images of Transparent wood sample (a) fabricated from balsa wood (b)

It can be observed that lignin is the binding agent between wood fibers and the determinant of the natural wood color. From the raw wood sample with a yellow color, when lignin is removed, the wood has a bright white color. When epoxy is impregnated into the wood fibers, it fills the pores left by lignin, creating a transparent wood product. These observations are clearly confirmed when analyzing the light transmittance of the wood samples (Figure 3).

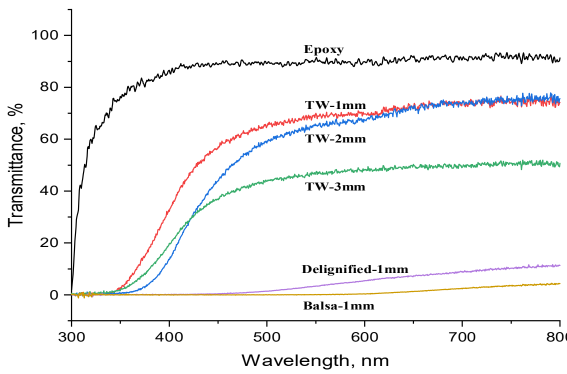

Figure 3. Light transmittance of balsa wood, delignified wood, Transparent wood (TW), and epoxy samples.

As observed in Figure 3, the light transmittance of the transparent wood samples is significantly higher than that of raw balsa wood across a broad wavelength range from 300 - 800 nm, the is. Due to its dense

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structure, raw balsa wood exhibits minimal light transmittance, attributed to the tightly interwoven cellulose, lignin, and hemicellulose, which absorb light. Following lignin-removal, the wood sample appear visually brighter and white compared to natural wood. However, the light transmittance of the lignin-removed wood sample (delignified 1mm) is very low in the 300-500 nm wavelength range and increases to 8% in the 500-800 nm wavelength range. This limited transmittance is due to air filling the hollow tubes and pores left by lignin, rendering the wood sample opaque. When analyzing the light transmittance of the epoxy-impregnated wood samples, it can be observed that the light transmittance gradually decreases with increasing thickness of the wood samples. The epoxy-impregnated wood samples show light transmittance starting to appear early from the 350nm wavelength region and increasing significantly from 500 nm. The epoxy-impregnated wood samples with thicknesses of 1mm and 2mm exhibit a light transmittance of 70%, which is substantially higher than that of sample with a thickness of 3mm, showing 45% of transmittance. These results indicates that the lignin treatment method is highly effective for thin wood samples of 1-2 mm. However, wood samples with thicknesses exceeding 3 mm require a combination of methods to ensure deeper treatment.

The light transmittance analysis of the transparent wood samples highlights the effectiveness of lignin removal using $NaClO_2$ in CH_3COONa/CH_3COOH. The light transmittance of the material can be adjusted based on changes in fabrication conditions and the removal of lignin from natural wood. This allows for control of the brightness of the transparent wood, which is also an advantage compared to conventional glass. While conventional glass exhibits relatively high light transmittance (80-90%) depending on the composition [12] its brightness is challenging to adjust, often resulting in excessive glare. On the other hand, glass is brittle and easily broken, which limits its practical usability.

FT-IR Infrared Spectroscopy Analysis

The results of the infrared spectroscopy analysis of raw wood, lignin-treated wood, and transparent wood/epoxy samples are summarized in Figure 4. The infrared spectra reveal peaks at 3332 cm^{-1} and 2927 cm^{-1}, which are characteristic of the stretching vibrations of -OH and C-H bonds. These bonds are present as single bonds in the structure of cellulose and hemicellulose. Notably, the peaks at 1509 cm^{-1} and 1598 cm^{-1} observed in the raw wood sample (Figure 4a) corresponding to C=C bond vibrations in the aromatic ring of lignin, are faintly present in the spectra of lignin-removed wood samples (Figure 4b, c, d). Additionally, the peak at 1242 cm^{-1}, indicative of the stretching vibration of the C-O bond in the O-C (aromatic ring) structure of the balsa sample, is absent in the spectra of lignin-removed wood samples.

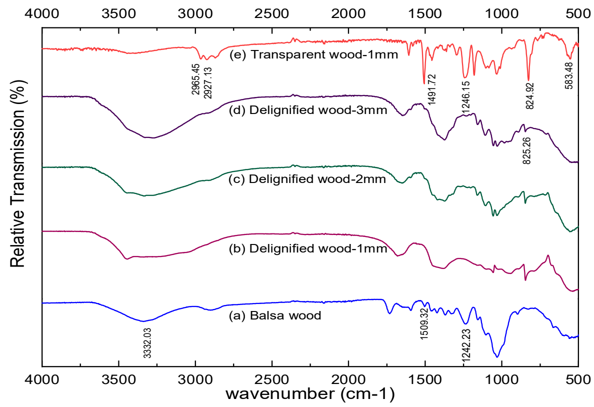

Figure 4. FT-IR spectra of the raw wood sample and modified wood samples (a) balsa wood, (b,c,d) delignified wood 1mm, 2mm, 3mm thick, (e) Transparent wood/epoxy 1mm thick.

For the transparent wood/epoxy sample (Figure 4e), the peaks at 1491 cm^{-1}, characteristic of =C-H vibration, 1246 cm^{-1}, corresponding to the stretching vibration of the aliphatic ether C-O-C, and 825 cm-1, attributed to the out-of-plane deformation vibration of the benzene ring with para substituents. The epoxy structure contains ether groups and C=C double bonds, originating from the presence of vinyl monomers, which are incorporated into the wood matrix through polymerization. The appearance of these peaks indicates that epoxy has successfully bonded into the wood framework, resulting in the formation of the transparent wood/epoxy composite.

Scanning Electron Microscopy (SEM) Analysis

The surface morphology of the wood sample before and after treatment was analyzed using SEM images. As observed in Figure 5, the porosity of the natural wood samples in the cross-sectional surface with 1mm and 2mm thicknesses changes during the modification process. The hollow tubes of the natural wood structure are clearly visible, featuring thick and smooth walls as shown in Figures 5a and 5b, corresponding to the 1mm and 2mm wood sample thicknesses, respectively. Following lignin removal, the wood's microstructure is not disrupted, and the tube walls remain stable within the structural framework (Figures 5c, d). Basically, minimal differences are observed in the cell wall structure between the 1mm and 2mm wood samples. After epoxy impregnation, the transparent wood samples are successfully formed. The SEM images (Figures 5e and 5f) reveal good interfacial contact between the epoxy and wood cell walls. The epoxy effectively fills the hollow tubes left by lignin removal, resulting in a stable cross-sectional surface with no visible cracks.

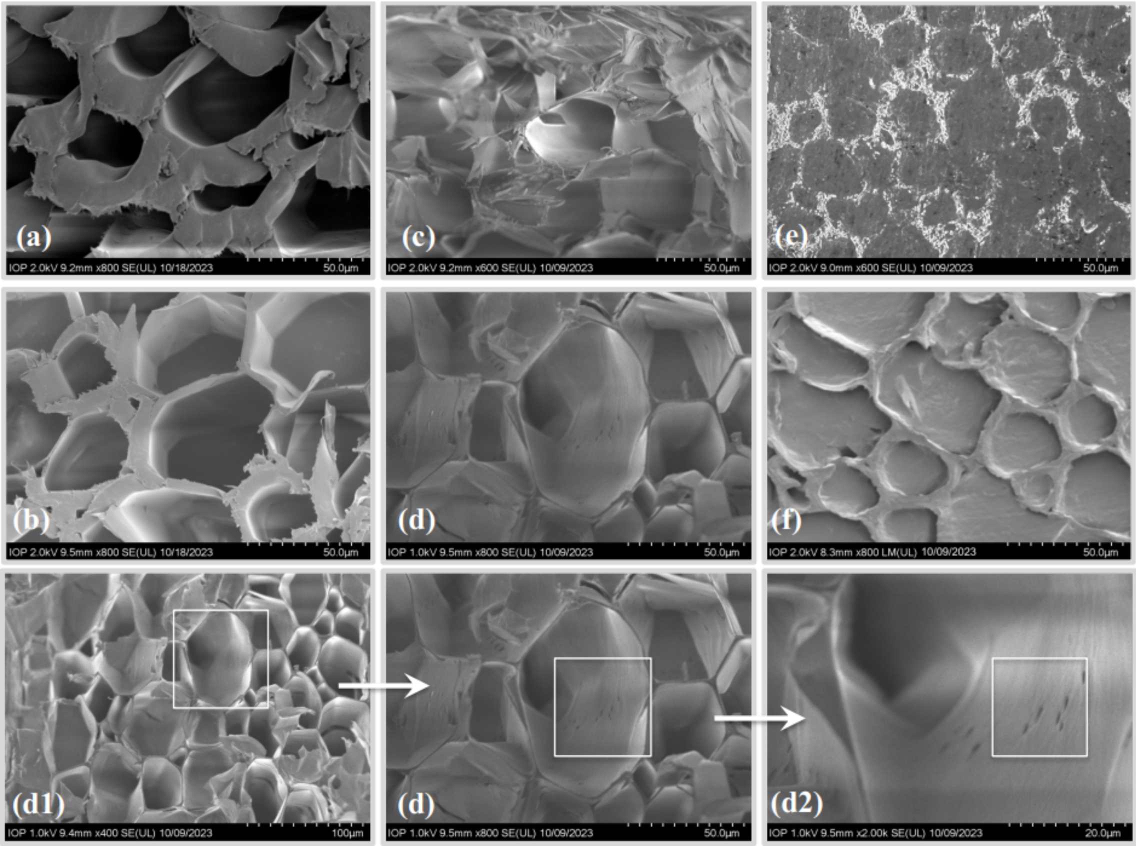

Figure 5. SEM images of wood samples (a) balsa wood 1mm, (b) balsa wood 2mm, (c) delignified wood 1mm, (d) delignified wood 2mm, (e) Transparent wood 1mm, (f) Transparent wood 2mm; delignified wood samples at magnifications (d1) 400x, (d) 800x, (d2) 2000x

As a result, the SEM image analysis of the wood samples during the fabrication of transparent wood highlights the removal of lignin, which creates small voids in the structure. This is evident in Figure 5 (d, d1, d2), showing magnified SEM images of the lignin-removed wood sample. The tube walls of the lignin-removed wood sample. The tube wall of the lignin-removed wood exhibit small pores, and surface of the tube wall forms thin fibers due to lignin removal. This makes the wood structure more porous, and the rough tube wall surface ensures that epoxy is filled tightly, creating an interwoven and robust structure.

Mechanical Strength

Mechanical properties are an important factor affecting the widespread application of materials. These properties are significantly influenced by the type of wood, including factors such as density, cellulose content, cell structure, and the anisotropic nature of the wood structure. In this study, the mechanical properties of the material were determined by the elastic modulus of the product. The results are summarized in Figure 6.

It can be observed that transparent wood demonstrates significantly higher strength with an elastic modulus of 2.4 GPa compared to lignin-removed wood with an elastic modulus of 2.05 GPa, balsa wood with 1.65 GPa, and epoxy resin with 2.1 GPa. The low elastic modulus of balsa wood is attributed to weak hydrogen bonds within its structure. Lignin-removed wood has a lower elastic modulus than transparent wood due to the absence of strong hydrogen bonds linking the cell walls. the mechanical properties of wood are primarily determined by the

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amount of cellulose capable of forming hydrogen bonds within the cell walls. During the lignin removal process, substantial voids are created as lignin is eliminated. Following epoxy impregnation, the epoxy interacts with the wood to form hydrogen bonds. This process significantly enhances the elastic modulus of transparent wood due to the strong hydrogen bonds established between the cell walls of the wood through the interaction between the polymer and the hydroxyl groups on cellulose. Therefore, the intermolecular bonding between cellulose and epoxy considerably improves the strength and stiffness of transparent wood, making it a promising material with potential applications across various fields.

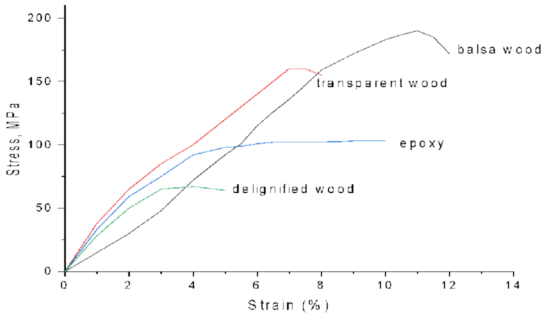

Figure 6. Relationship between stress and strain of samples (a) balsa wood, (b) delignified wood, (c) Transparent wood, and (d) epoxy

Conclusion

The results of this study indicate the successful fabrication of a transparent wood composite material based on epoxy/balsa wood. The process involved lignin removal with $NaClO_2$ in CH_3COONa/CH_3COOH solution, followed by epoxy impregnation under vacuum conditions. The transparent wood achieved a light transmittance of 70% for wood samples with thicknesses of 1mm and 2mm in the wavelength range of 500–780 nm. The lignin treatment process, lasting 6–8 hours allowed for the stabilization of the cellulose fiber structure in the modified wood. However, wood samples with thicknesses of 3 mm require a more intensive lignin treatment to prevent sample cracking.

The epoxy/balsa transparent wood composite exhibits higher tensile strength compared to natural wood. Based on its light transmittance and mechanical properties, the epoxy/transparent wood is promising material with potential application as replacement for conventional glass in areas such as smart windows and decorative materials. Furthermore, the use of transparent wood in construction projects offers energy-saving benefits by optimizing light transmission and reducing energy consumption.

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