Transparent Wood Developed by Impregnating Poplar with Epoxy Resin Assisted by Silane Coupling Agent

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Biodegradable transparent wood was fabricated by introducing epoxy resin E51 modified with the silane coupling agent (KH550) into bleached poplar veneer. The light transmittance of transparent wood was modulated by KH550 content. The silane coupling agent KH550 was able to change the compatibility of epoxy resin and wood substrate, thereby affecting the performance of transparent wood. In this study, the effect of KH550 on the properties of transparent wood and its mechanism were investigated. The light transmittance, tensile strength, and elongation at break of transparent wood showed an increasing and then decreasing trend with increased KH550 dosage. When the mass ratio of the coupling agent KH550 to the epoxy resin was 1:20, the transparent wood made by impregnating wood substrate with epoxy resin modified by KH550 had the best performance, with the fast degradation starting temperature of 338 °C, 81.07% light transmittance at λ = 780 nm, 59.92 MPa tensile strength, and 3.47% elongation at break. This work provides a new way for preparing high performance transparent wood.

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Keywords: Transparent wood; Epoxy resin; Silane coupling agent; Interfacial compatibility; Light transmittance

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INTRODUCTION

The serious harm to the environment caused by the extensive use of petroleum-based materials poses an urgent need for the development of environmentally friendly bio-based materials. Bio-based materials can replace non-renewable petroleum products and play an important role in maintaining environmental sustainability (Cai *et al*. 2021). As a natural and environmentally friendly material, wood plays an extremely important role in the field of materials because of its low price, easy handling, environmental protection, sustainability, and other advantages (Xiao *et al*. 2019). Transparent wood (TPW) is a new material with high light transmission and low haze, excellent toughness, low thermal conductivity, low density, and anisotropic optical and mechanical properties made from wood, and is being paid increasing attention (Montanari *et al*. 2019). The chromophoric structures in lignin absorb the light within the broad visible range, which contributes to opacity. By contrast, cellulose and hemicellulose, the main constituents of wood, are optically colorless due to their significantly low absorption in the visible range. However, the difference between the refractive index of the main components of wood (such as cellulose) and the refractive index of the air in the wood pores causes some light to be

scattered when light shines on natural wood (Yang *et al*. 2019). To study the morphological characteristics of wood microstructure, Fink first proposed the concept of transparent wood (Fink 1992). Through removing lignin, a light-absorbing component of wood, a nanocellulose porous template structure is produced. The next key issue in the preparation of transparent wood is the selection of resin. In order to prevent the refraction and scattering of light at the interface of different media, the impregnated resin should have high transparency, and its refractive index should match with that of wood (Li *et al*. 2016; Zhu *et al*. 2016a). In this case, most of the irradiated light shining on the wood can pass through, resulting in a wood that exhibits high light transmission. The resulting multifunctional transparent wood-polymer composite is defined as "transparent wood". The successful development of TPW has captured enormous interest in areas such as automotive windshields, transparent packaging, and biomedical devices, *etc.* (Zhu *et al*. 2016a; Cai *et al*. 2021).

The structural and functional properties of TPW are mainly influenced by both wood species and infiltrated polymer. To prepare transparent wood, removal of lignin to increase transparency is a necessary means (Li *et al*. 2018), which mainly includes oxidation, chemical modification, and enzymatic hydrolysis. Among them, the oxidizing agents used in most of the oxidation methods are hypochlorous acid (Gan *et al.* 2017) or sodium chlorite (Yaddanapudi *et al.* 2017). In chemical purification methods, alkaline sodium sulfite is mainly used to treat wood, which enhances the water solubility of lignin by introducing a sulfonic acid group on the side chain of the benzene ring, resulting in highly transparent wood (Bi *et al.* 2018). Enzymatic digestion, as an environmentally friendly method, can achieve wood decolorization (Cullis and Mansfield 2010), but the strategy of enzymatic digestion for the preparation of transparent wood remains to be explored. The selection of a suitable resin is another key issue in the preparation of transparent wood. The resins used for impregnating wood should have a high transparency and refractive index matching the wood to reduce refraction and scattering of light at the interface of different media (Zhu *et al.* 2016a). Currently, the resins commonly used to prepare transparent wood are polymethyl methacrylate (PMMA) (Li *et al.* 2017), epoxy resin (Zhu *et al.* 2016a), polyvinyl alcohol (PVA) (Mi *et al.* 2020), and polyvinyl pyrophthalate (PVP) (Zhu *et al.* 2016b).

Mechanical and optical properties are important indicators of transparent wood, which can generally be improved by optimizing the interface bonding between the wood template and the polymer. The main improvement paths are modified wood template, modified resin, or improved technology. Disparities between properties of wood polymers having hydrophilic groups and synthetic resins containing hydrophobic moieties may easily cause phase separation. In addition, although the liquid resin can completely penetrate the wood substrate, volume shrinkage occurs after polymerization and curing, resulting in peeling off and creating interfacial wind gaps with the wood substrate. The use of silane coupling agents containing both organic and inorganic groups into their molecule can improve the compatibility of the wood substrate with the resin and reduce the interfacial tension between the two, aiming to improve the overall properties of the material. Therefore, the epoxy resin with refractive index close to that of cellulose was selected as the filler resin. After modification by silane coupling agent KH550, it was filled into the wood substrate to prepare TPW. The effect of KH550 dosage on the performance of TPW and its mechanism were investigated to provide a reference for the preparation of high performance TPW.

EXPERIMENTAL

Materials

Tangential veneers of *Populus euramericana* were obtained from Hefei Yusen Wood Industry Co., Ltd. (Hefei, China). Silane coupling agent KH550 (γ-aminopropyl triethoxysilane $C_9H_{23}NO_3Si$) was provided by Shanghai Aladdin Biochemical Technology Co., Ltd. (Shanghai, China). Epoxy resin E51 and epoxy resin curing agent 593 were provided by Shanghai Aotun Chemical Technology Co., Ltd. (Shanghai, China). Anhydrous sodium sulfite (Na_2SO_3), sodium hydroxide (NaOH), and hydrogen peroxide (H_2O_2, 30 wt%) were purchased from Xilong Scientific Co., Ltd. (Shantou, China). Absolute ethyl alcohol (C_2H_6O, 99.7 wt%) and acetone were both obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

Preparation of the Sample

Specimens with the uniform size of 50 mm × 50 mm × 2 mm (length × width × thickness) were cut from tangential veneers of poplar wood. The defective places on the wood pieces were avoided during cutting. The wood samples were placed in the oven at 105 °C for 3 h until their mass no longer changed. Then, the wood samples were submerged in a mixed aqueous solution of Na_2SO_3 (5 wt%) and NaOH (10 wt%) at 90 °C for 16 h, followed by washing with distilled water to neutral pH. Subsequently, the washed wood samples were immersed in a 4 wt% H_2O_2 aqueous solution at 90 °C until the wood became completely white in appearance. As a result, the lignin-removed wood substrates were prepared. After washing with hot water to remove residual chemicals, the wood substrates were stored in anhydrous ethanol to improve the permeability of the wood cell walls. Next, KH550 was added to epoxy resin E51 as a modifying agent according to the mass ratios shown in Table 1. The epoxy resin modified with KH550 was obtained after heating and stirring at 40 ℃ for 4 h. To remove the ethanol from the samples, the lignin-removed wood samples were repeatedly washed three times with acetone and filtrated under vacuum for 2 min. The lignin-removed wood samples were impregnated in different modified epoxy resin impregnating solutions with a mass ratio of epoxy resin to curing agent of 4:1, with a vacuum of -0.08 MPa and a soaking time of 5 min. This procedure was repeated three times. The wood samples were removed and clamped with two clean glass plates for fixing and pressing. After the samples were cured at 30 ℃ for 14 h, transparent wood could be obtained. The names of transparent wood corresponded to the modified epoxy resin.

Table 1. Mass Ratios of KH550 to EP in the Samples of Transparent Wood

Sample	KH550	EP
KE0-100	0	100
KE5-100	5	100
KE10-100	10	100
KE15-100	15	100
KE20-100	20	100

Characterization of the Sample

The optical transmittance of all samples was measured with an UV/V-16/18 UV–vis spectrophotometer (Shanghai Yiheng Scientific Instrument Co., Ltd., Shanghai, China)

in the range of 380 nm to 780 nm at 20 nm intervals. The mechanical properties of the samples were tested along the longitudinal direction of the wood using an UTM4304 microcomputer control electronic universal mechanical testing machine (Shandong Kesheng Electronics Co., Ltd., Jinan, China) with the tensile speed of 2 mm/min (the length × width × thickness of samples was 50 mm × 10 mm × 2 mm). The results of mechanical properties were the average of 5 samples. Over the range of 400 to 4000 cm^{-1}, the chemical structures of all samples were collected using a TENSOR II infrared spectrometer (Bruker Corporation, Karlsruhe, Germany) through 32 scans at a resolution of 4 cm^{-1}. The thermal stability of the samples was measured at a heating rate of 10 °C/min in the range of 50 °C to 600 °C using a TGA/DSC 1/1100 thermal analyzer (Mettler-Toledo International Inc., Zurich, Switzerland) under nitrogen protection. The morphologies of wood and transparent wood samples were analyzed using a Hitachi S-4800 scanning electron microscope (SEM) (Hitachi, Ltd., Tokyo, Japan) at an accelerating voltage of 3.0 kV.

RESULTS AND DISCUSSION

Based on the difference between the cutting direction and the direction of the axis of wood trunk, the wood section can be divided into the longitudinal (L) section (parallel to the axis of the wood trunk) and the radial (R) section (perpendicular to the axis of the wood trunk). The TPW prepared from the L-section wood chip exhibited strong optical anisotropy and mechanical properties because of its anisotropic microstructure. The lignin-modified bleaching process was used to remove leachate and raw color masses from lignin while retaining a large amount of lignin, which was expected to be beneficial for establishing the structural stability of the material. The colored components were eliminated, and the wood becomes optically white. Next, the polymer (epoxy resin) that penetrates the cellulose-rich bleached wood substrate induced transparency and enabled a homogeneous interface within the wood microstructure, thus exhibiting a unique high light transmittance.

SEM Analysis

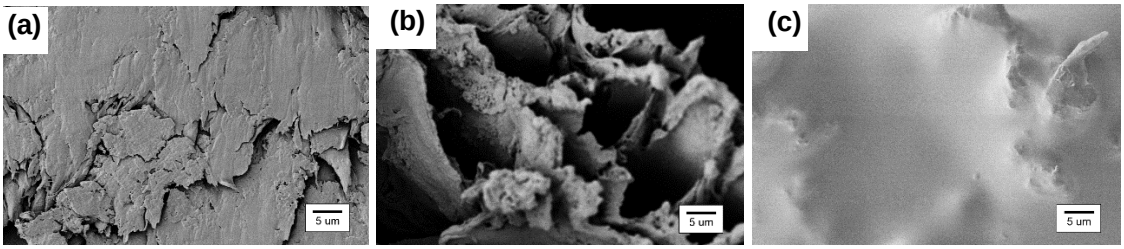

Fig. 1. SEM images of (a) original wood, (b) bleached wood, and (c) TPW (KE5-100)

Figure 1 shows scanning electron micrographs of original wood, bleached wood, and TPW (KE5-100). In addition to cellulose, hemicellulose, and lignin, the interior of the original wood was filled with a large amount of pectin material and appears dense, as shown in Fig. 1a. Figure 1b shows the cross-section of bleached wood. After delignification, the wood substrate showed a highly loose skeleton with many micropores at the middle plate (ML) and corners of the cell wall. The partial removal of lignin caused the cell walls of the bleached wood to become thinner and the cell lumen to become larger.

The cellulose network structure remained stable in the bleached wood substrate, as expected, indicating that a large amount of lignin was retained in the microstructure of the wood. Figure 1c presents the perfect impregnation of the epoxy resin into the lumen and void of the wood cell wall, indicating that the interfacial compatibility between the epoxy resin and the wood substrate was remarkably improved after modification by the coupling agent KH550. This modification effectively reduced the light scattering and improved the optical properties of the transparent wood.

FTIR Analysis

Figure 2 shows the FTIR spectra of bleached wood, KE0-100, KE5-100, and KE20-100. Although most of the lignin was removed from the bleached wood, characteristic peaks characterizing the wood still appeared on the IR spectra of bleached wood, such as the stretching vibration absorbance peaks of O-H and hydrogen bonds at 3340 cm^{-1}, the symmetric stretching vibration absorbance peak of C-H in cellulose at 2890 cm^{-1}, the stretching vibration absorbance peak of acetyl groups in hemicellulose at 1732 cm^{-1}, and the stretching vibration absorbance peaks of benzene skeleton in lignin at 1592 cm^{-1} and 1506 cm^{-1}. The characteristic peaks at 3340 cm^{-1} and 2890 cm^{-1} did not change noticeably, indicating that the structure of cellulose in the wood substrate was preserved after bleaching.

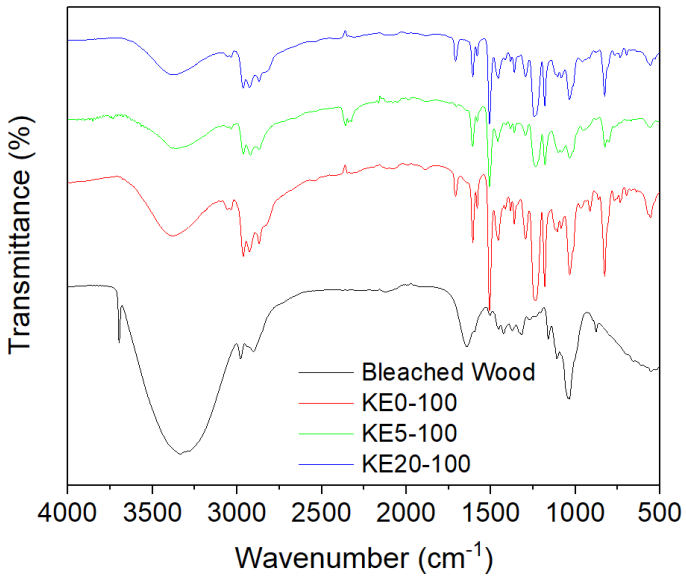

Fig. 2. FTIR spectra of bleached wood, KE0-100, KE5-100, and KE20-100

After impregnation with epoxy resin, the corresponding characteristic absorbance peaks appeared in the spectrum of transparent wood KE0-100, such as the stretching vibration absorbance peak of C-H in CH_2 and C-H at 2920 cm^{-1} and 2850 cm^{-1}, the stretching vibration absorbance peak of C=C in aromatic ring at 1606 cm^{-1}, the stretching vibration absorbance peak of C-C in aromatic ring at 1508 cm^{-1}, and the stretching vibration absorbance peak of C-O-C in ethers linkage at 1180 and 1033 cm^{-1}. This also indicates that epoxy resin had been impregnated into the pores of the wood substrate. The FTIR spectra of KE5-100 and KE20-100 confirmed KH550 successfully coupled wood to epoxy resin. The characteristic peaks of the stretching vibration of –C=C– at 1510 cm^{-1} and the symmetric stretching vibration and asymmetric vibration of C–O–C at 830 cm^{-1} and 1202

cm^{-1} in the spectrum of transparent wood KE5-100 and KE20-100 indicate that KH550 achieved the penetration cross-linking of epoxy resin and wood substrates. It can be seen that by introducing reactive groups using KH-550, the epoxy resin and the wood template were able to successfully couple and generate forces that improved the compatibility between components of transparent wood.

TG Analysis

The thermal behaviors of the original wood, bleached wood, KE0-100, and KE5-100 in the nitrogen atmosphere were investigated by thermogravimetric analysis (TGA/DSC 1/1100; Mettler-Toledo International Inc., Zurich, Switzerland) with a N_2 flow rate of 20 mL/min and a heating rate of 10 °C/min. The weight loss of the samples at different temperatures was recorded and analyzed to characterize the thermal stability of the samples in the temperature range of 50 to 600 °C. As shown in Fig. 3, the samples exhibited different degradation patterns at high temperatures because of their different polymer composition. Up to 250 °C, the mass loss of the original wood and the bleached wood was 9% and 22.7%, respectively. Rapid mass loss of original wood occurred between 270 and 395 °C. The data in Table 2 show that the bleaching treatment caused degradation of most of the hemicellulose in the wood, making the starting temperature of the bleached wood higher than that of the original wood during the rapid mass loss phase. The increased cellulose content in the wood substrate after bleaching resulted in a lower thermal degradation onset temperature of bleached wood. When heated to 600 °C, the mass loss was 78.7% and 74.46% for original wood and bleached wood, respectively. Due to the modification and enhancement of epoxy resin in wood, the starting decomposition temperature of the bleached wood impregnated with the epoxy resin during the rapid mass loss phase has increased considerably. The coupling reaction of KH550 facilitates the chemical bond between epoxy resin and wood substrate, thus improving the thermal stability of KE5-100 at high temperature. When heated to 600 °C, the thermal weight loss of KE5-100 was lower than that of KE0-100, which is the illustration of this effect.

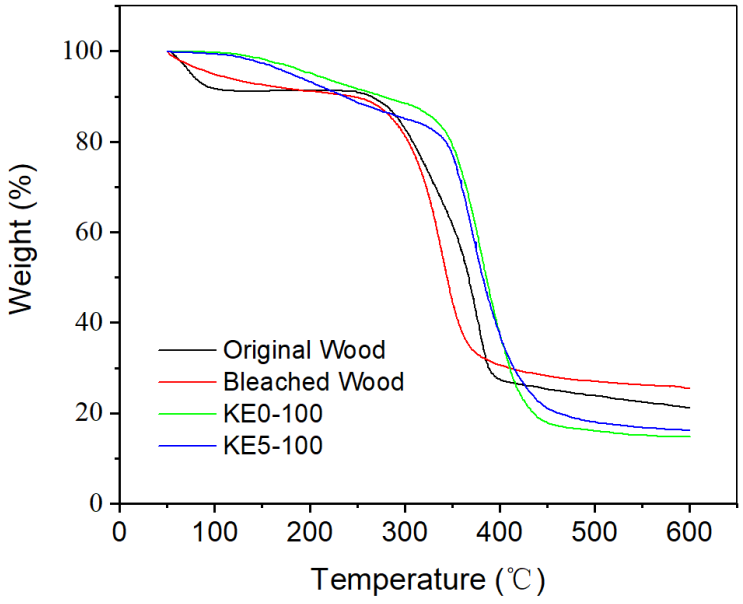

Fig. 3. TG curves of original wood, bleached wood, KE0-100, and KE5-100

Table 2. Thermal Properties of the Samples

Sample	Mass loss rate at 250 °C	Main weight loss temperature range	Mass loss rate at 600 °C
Original Wood	9%	270 °C – 395 °C	78.7%
Bleached Wood	22.7%	294 °C – 372 °C	74.21%
KE0-100	8.3%	338 °C - 450 °C	85.2%
KE5-100	11.31%	338 °C - 450 °C	83.75%

Optical Characteristics

Figure 4 shows the optical properties of the transparent wood samples and the bleached wood. The digital photographs of the samples in Fig. 4a show that the light transmittance of transparent wood was influenced by the degree of binding of the wood substrate to the epoxy resin. Figure 4b shows the variation of light transmittance with wavelength for the samples in the range of 380 to 780 nm. It is clear that the light transmittance of the blenched wood was extremely low, which was 12.2% at λ = 780 nm. The transmittance of TPW samples showed an increasing trend as the wavelength increased from 380 to 780 nm. Compared to bleached wood, TPW filled with epoxy resin exhibited a noticeable increase in light transmittance. The light transmittance of the bleached wood and the transparent wood KE0-100 at λ = 560 nm was about 9.33% and 52.02%, respectively. Because of the large difference in refractive index among holocellulose (μ = 1.53), lignin (μ = 1.61), and air (μ = 1) in the bleached wood, light was scattered inside the wood. When the epoxy resin (μ = 1.51 to 1.56) with refractive index matching the wood (μ = 1.51 to 1.56) was impregnated into the wood substrate, the light transmittance of the wood could be noticeably enhanced.

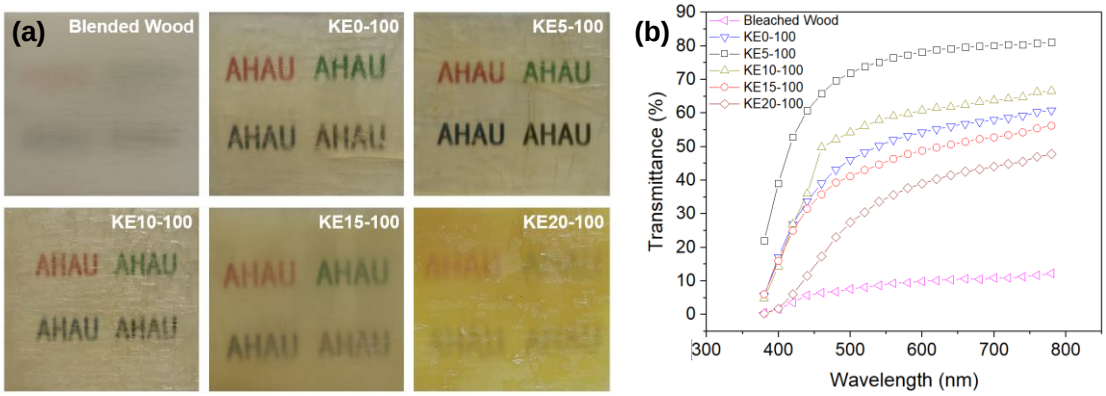

Fig. 4. (a) Digital photograph, and (b) visible light transmittance of the Bleached Wood, KE0-100, KE5-100, KE10-100, KE15-100, and KE20-100

Compared with the KH550-modified transparent wood, such as KE5-100 and KE10-100, the transparent wood KE0-100 without KH550 modification had a low light transmittance of 60.81% at λ = 780 nm. Among the transparent woods modified with KH550, the KE5-100 showed the highest light transmittance at λ = 780 nm with 81.07%. It can be seen that the interfacial compatibility of epoxy resin and wood substrate was effectively improved after modification by silane coupling agent KH550, which enhanced the bonding effect between epoxy resin and wood substrate and reduced the scattering of light in transparent wood. It is worth mentioning that the light transmittance of transparent woods modified with KH550, KE15-100, and KE20-100 was lower than that of KE0-100.

This indicates that too much or too little of the coupling agent KH550 would lead to incomplete grafting reactions, which eventually caused a decrease in the light transmittance of transparent woods.

Mechanical Properties

The mechanical properties are important indicators that need to be paid attention to in the application of transparent wood. The tensile strength and elongation at break of the bleached wood and the transparent wood (KE0-100, KE5-100, KE10-100, KE15-100, and KE20-100) along the fiber direction are shown in Fig. 5. Clearly, the mechanical properties were all better than those of the bleached wood under the enhancement of epoxy resin. At the same time, it can be seen that the value of the mechanical properties of transparent wood (KE0-100) were all lower than those of TPW modified by KH550. In the process of lignin modification, the mechanical properties of bleached wood decreased due to the mechanical loss caused by the removal of hemicellulose and lignin. The impregnation of wood substrate with epoxy resin improved the anisotropy of the wood itself, resulting in a more homogeneous material with increased strength and toughness.

The introduction of silane coupling agent KH550 was able to improve the compatibility between wood fibers and epoxy resin, which helps the penetration and cross-linking of epoxy resin in the wood substrate, thus noticeably improving the mechanical properties of transparent wood. The right amount of KH550 was able to improve the interfacial compatibility of epoxy resin and wood substrate. When too little KH550 was added, the coupling agent will not be completely covered on the surface of the wood substrate, making it difficult to form a good layer of coupling molecules. Excessive amounts of KH550 can in turn lead to too many coupling agent molecules covering the wood substrate surface, thus affecting the interfacial structure of the wood substrate and epoxy resin. Overall, the overall mechanical properties of transparent wood KE5-100 were the best, which makes it great potential applications in the fields of construction materials, solar cells, and magnetic materials.

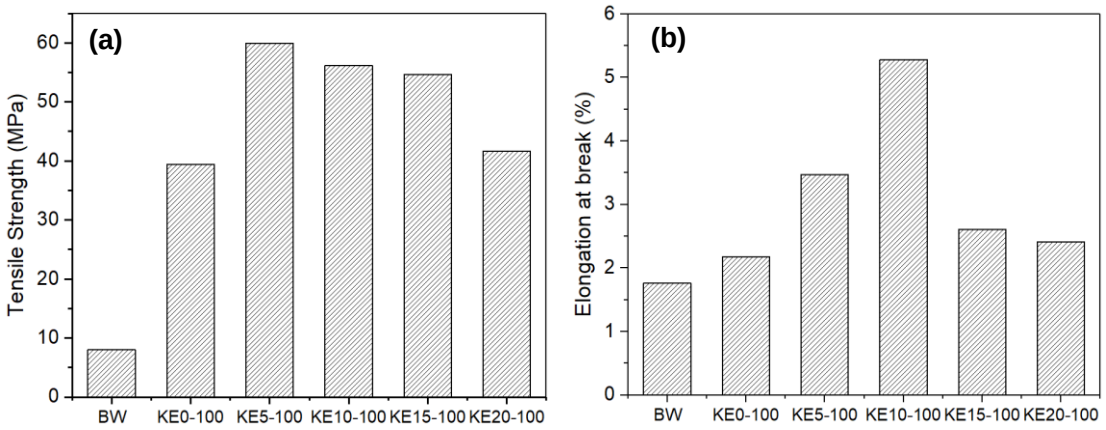

Fig. 5. The tensile strength (a) and elongation at break (b) of the Bleached Wood (BW), KE0-100, KE5-100, KE10-100, KE15-100, and KE20-100

CONCLUSIONS

1. In this study, transparent wood was produced by vacuum impregnation of delignified wood substrate with silane-modified epoxy resin.
2. Vacuum impregnation was able to effectively increase the content of epoxy resin in the wood substrate. The Fourier transform infrared (FTIR) spectra of the transparent wood showed that the silane coupling agent KH550 successfully achieved the coupling of the wood substrate and the epoxy resin. The scanning electron microscopy (SEM) analysis showed that most of the pores between the epoxy resin and the wood substrate were eliminated under the coupling effect of KH550, thus enhancing the interfacial compatibility between the two.
3. The light transmittance, mechanical properties, and thermal stability of the transparent wood were related to the coupling agent KH550. When the mass ratio of silane coupling agent KH550 to the epoxy resin was 1:20, the transparent wood made by impregnating wood substrate with epoxy resin modified by KH550 had the best performance. Compared with the fast degradation starting temperature of 270 °C of original wood and bleached wood, the fast degradation starting temperature of KE5-100 increased to 338 °C, with a noticeable improvement in thermal stability. Among all transparent wood samples, KE5-100 exhibited the highest light transmittance with the light transmittance of 81.07% at λ = 780 nm. The tensile strength and elongation at break of KE5-100 were 59.92 MPa and 3.47%, respectively.
4. Strategy for the preparation of large-scale high-performance transparent wood has been provided in this work. The preparation process of transparent wood is simple and convenient. However, the preparation of large-area transparent wood needs further study.

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