Research Article

In Polymerization of Environment Friendly Melamine-Urea-Glyoxal Resin in Rubber Wood for Improved Physical and Mechanical Properties

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In the study, we report that a safe and simple way for upgrading inferior rubber wood through the combined modification of environment-friendly MUG resin was synthesized from glyoxal, melamine, urea, and other additives. MUG-treated wood samples were prepared with six different MUG resin concentrations (5, 15, 25, 35, 45, and 55 wt %) into the wood matrix and then heated and polymerized to form a solid and hydrophobic MUG resin in the wood scaffold, and the physico-mechanical properties were evaluated. As the MUG resin concentration increased, the weight percent gain and density increased, water uptake and leachability decreased, and the antiswelling efficiency increased at first and then decreased. MUG-treated wood sample can be prepared when the MUG resin concentration was set as 25%, and the physical properties of treated wood was optimum. Scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy analysis showed that the MUG resin is widely distributed in the cell lumens and cell walls. With enhanced physico-mechanical properties, MUG-treated wood sample can be well used as a promising alternative to existing engineered wood products for structural applications.

1. Introduction

In recent years, the depletion of fossil resources and increasing attention to environment require more sustainable development in the process of finding renewable resources to provide energy for production and life. In this case, considering the renewability and sustainability of lignocellulosic biomass, it has broad application prospects and is expected to become a substitute for fossil resources. Among the available natural materials, wood has become one of the main candidate materials that has attracted more and more interest in academia and industry due to its unique inherent features. Structural wood has always been and has been a very versatile and important material with a wide range of uses due to its pleasing properties. At the same time, there are some disadvantages, such as high-water uptake, and physico-mechanical properties change with environmental factors [1]. The troublesome inherent properties of wood can be minimized by appropriate chemical treatment, such as the formation of wood polymer composites (WPC) [2]. The existence of hydrophilic hydroxyl groups in wood component is the main reason for the negative characteristics. Wood attracts water through hydrogen bonds, making it dimensionally unstable. The physico-mechanical properties of wood can be improved by using an appropriate chemical impregnation technology which can react with cell wall components.

Rubber trees (Hevea brasiliensis), which are indigenous to the Amazon valley in South America, have been widely cultivated in China since the latter half of the twentieth century [3]. Currently, they are primarily planted in Hainan, Yunnan, and other southern Chinese provinces, covering more than 1.18 million ha and providing rubber wood logs up to 2 million cubic meters each year. Due to the decline of rubber production capacity of rubber trees, the rubber plantations are renewed and the rubber wood can be recycled.
continuously. This will make rubber wood superior to some traditional tropical wood used in furniture and wood-based panel manufacturing. The latest progress in rubber wood plantation certification confirms its development [4]. However, the rubber wood has inferior dimensional stability compared to other woods and is prone to decay, warp deformation, and cracks. Modifying the rubber wood is a good method for improving its properties and achieving a greater range of potential use.

In order to improve the dimensional stability, water resistance, and mechanical properties of wood, it has made a number of chemical modification strategies, such as acetylation [5], furfurylation [6], resin impregnation [7], and silanization [8]. Although chemical modification can improve some properties of wood, most wood modifiers contain harmful chemicals or solvents, which may cause serious environmental and health problems during the treatment process and the use of wood products. For example, it is known that wood impregnated with phenol formaldehyde (PF) resin and melamine urea formaldehyde (MUF) resin will release harmful volatile organic compounds, such as formaldehyde, phenols, and other VOCs, thus limiting its indoor applications. Granting polymerization of vinyl monomers such as styrene and methyl methacrylate often involves the use of toxic organic solvents to bring chemicals into the wood structure [9]. Driven by eco-environmental protection and health issues, the development of nontoxic and green chemically modified wood methods is strongly pursued. Glyoxal is a dialdehyde with low volatility and nontoxicity, and it is usually used as a paper reinforcer in the pulp and paper industry, due to its two active merged aldehyde groups, which can be used to glyoxylate lignin in cell walls or crosslink with hydroxyl groups in cellulosic materials via hemiacetal or acetal reactions [10, 11]. In this context, glyoxal is used for wood modification, thereby improving the dimensional stability of the modified wood. However, it will reduce the mechanical properties of wood and prevent its use as load-bearing structural member [12, 13].

In this study, in view of the fact that inferior rubber wood cannot meet the requirements of engineering materials, low volatility, nontoxic glyoxal, melamine, urea, and other additives were used to synthesize environmentally and friendly MUG resin, and rubber wood was impregnated. The overall performance of rubber wood are in terms of dimensional stability, and mechanical properties. Upon cured in wood, the formed polymer crosslinked structures within wood, thereby improving the performance of the wood. The resin structure and properties were analyzed, and the physico-mechanical properties of the resulting wood were investigated.

2. Material and Methods

2.1. Raw Materials. For this study, rubber wood (Hevea brasiliensis) was obtained from Ledong county, Hainan Province, China. Rubber wood samples were cut from the sapwood of the trunk and dried in an oven below 103°C before use. The glyoxal (G, 40% wt%) and additive used were of analytical grade of China National Pharmaceutical Group Corporation. Solid melamine (M) and urea (U) were industrial reagents, which were used provided by Chian Lanyi Chemical Reagent Company. Add aqueous solutions of sodium hydroxide (NaOH, 30 wt%) during the resin synthesis process to adjust the pH.

2.2. Preparation of Impregnation Solutions (MUG). The water-based impregnation solution (MUG resin) consisted of glyoxal, melamine, urea, and additives. MUG resin was prepared at a glyoxal/melamine/urea/additive molar ratio of 1:0.08:0.6:0.5. Appropriate amounts of glyoxal (G, 30 wt%) and additive were added to a three-necked flask and stirred, making it a weak acid with 30% NaOH. Subsequently, melamine (M) and urea (U) were added, stirred, and heated to 70°C. The synthetic technology of MUG resin is under weak acidity condition at 70°C for 3 h. The reaction mixture was made with weak alkaline and cooled at room temperature; MUG resin solution was acquired.

2.3. Preparation of MUG-Treated Wood. Before impregnation, all rubber wood samples were dried in an oven at 103°C for 12 h, in which the weight and dimension of rubber wood samples were measured. The MUG resin was immersed into the rubber wood samples in a customized chamber using a full cell process, firstly, for 30 min of vacuum treatment, and then, for 6 h of pressure treatment (1.0 MPa). After impregnation, wipe the samples with tissue paper to remove excessive solutions on the wood surface. In order to prevent the solution from evaporating during the curing process, the rubber wood samples were wrapped in aluminum foil and cured in an oven at 103°C for 4 h to allow the resin to fully polymerize in the wood matrix. After that, the aluminum foil was removed. First, the samples were dried at 80°C for 3 h, and finally, at 103°C until it reached a dry state. The weight percent gain and dimension stability were calculated according to the oven dry weight and size of rubber wood samples before and after impregnation.

2.4. Characterization. The stability of synthesized MUG resin was mainly evaluated by its change in viscosity and appearance over a period time according the GB/T 14074-2006. The MUG resin was stored at room temperature, and the viscosity was measured, and its appearance was assessed at regular intervals. If there was no change in viscosity and appearance before and after storage, the MUG resin was considered to be stable. About 1.000 g of MUG resin was poured into a disposable aluminum dish and then dried in an oven at 120°C for 2 h. The nonvolatile solid content was determined by measuring the weight of the MUG resin before and after drying. The average of three parallel specimens was presented. The viscosity of the MUG resin was measured at 25°C with an NDJ-1 rotary viscometer. A gel permeation chromatography (GPC) (Shimadzu LC20, Japan), a system equipped with a refractive index detector (RID-20), was used to measure the molecular weight (MW) of the resin. Fourier transform infrared (FTIR) spectra were recorded on a Nicolet IS10 spectrometer (Thermo Nicolet, USA) over the wavenumber range of
4000–400 cm$^{-1}$ at a resolution of 0.4 cm$^{-1}$. Scrape the surface of the sample with a dimension of 20 mm × 20 mm × 20 mm (radial × tangential × longitudinal), and the crosssections and longitudinal sections were observed by stereo microscope (Olympus SZX10, Japan). Field emission scanning electron microscope (FE-SEM) (Hitachi, SU8020) combined with an energy dispersive X-ray (EDX) detector was used to characterize the morphology of the samples.

2.5. Physico-Mechanical Properties. The physico-mechanical properties of wood samples were tested according to China National Standard Testing Methods for Physical and Mechanical Properties of Wood (GB/T 1929-2009). The weight percent gain (WPG), density ($\rho$), water uptake (WA), leachability ($L$), and antiswelling efficiency (ASE) were measured on 10 replicates of cubic samples with a size of 20 mm × 20 mm × 20 mm (radial × tangential × longitudinal). The WPG and ASE due to impregnation were determined by comparison of the dry weight and dimension of the control sample and the treated sample, respectively. For the determination of ASE, both the control and treated samples in the oven-dried state were soaked in water for 20 days. The volume of the samples before and after water immersion were measured to determine the volume swelling coefficient. Therefore, ASE was calculated by comparing the volume swelling coefficients of the control and treated samples.

Density ($\rho$) of the samples were measured as an index of wood properties. The control and treated samples were dried at 103°C until a dry state was achieved. Density was calculated by weight and volume of samples which were measured before and after oven-dried.

Water uptake (WA) of the wood samples were measured as an index of water repellency. The oven-dried samples were soaked in water for a total of 20 days. After a certain time interval, the samples were taken out of the beaker and wiped the surface with tissue paper to remove excess water, and then, the weight was measured. The WA of samples was calculated based on the initial dry weight and final wet weight after soaking in water.

The modulus of elasticity (MOE), modulus of rupture (MOR), and compressive strength (CS) parallel to grain were measured to evaluate the mechanical properties of the wood samples. Wood samples with dimensions of 20 mm × 20 mm × 300 mm (radial × tangential × longitudinal) were used for MOE and MOR tests and 20 mm × 20 mm × 30 mm (radial × tangential × longitudinal) were used for CS test. Using a universal testing machine (Model AG-2000A model manufactured by Shimadzu, Japan), MOE and MOR were measured under static three-point bending. Ten wood samples were measured for each of the above mechanical tests.

For the determination of leaching ($L$) test, treated samples in the oven-dried state were soak in water. The samples were taken out of the beaker and wiped the surface with tissue paper to remove excess water after 20 d and then dried at 103°C until a dry state was achieved. The $L$ of samples were calculated based on the dry weight of control sample, and initial dry weight and final dry weight before and after water soaking.

### 3. Results and Discussion

The manufacturing process of MUG-treated wood sample is illustrated in Figure 1. Natural wood has a unique cellular structure with many open channels along the cell growth direction. Water and other liquid can flow in the wood through the channels. The cell wall is a natural fiber composite material, composed of hard cellulose microfibrils embedded in a soft matrix of hemicellulose and lignin. Due to the hygroscopicity of amorphous cellulose and hemicellulose, the cell wall of wood will swell or shrink upon with the change of moisture content, resulting in a change in wood dimensions. The dimensional instability related to moisture is particularly obvious for the rubber wood. On the other hand, due to its natural porous structure including cell lumen and nanopores in the cell wall, additional materials can be incorporated to enhance performance. After being introduced into the wood under vacuum and pressure conditions, MUG resin can penetrate the pore structure of wood. After high-temperature curing, a hydrophobic dark brown polymer with a crosslinked structure is formed inside the wood, thereby improving the strength and reducing the water absorption rate of wood.

#### 3.1. Characterization of Resin

3.1.1. Properties of Resin. The light-yellow liquid MUG resin was synthesized under slightly acid condition with a pH of 5–6, and the properties are summarized in Table 1. The synthesized resins can remain stable for at least 30 days or more at room temperature. As shown in Table 1, the viscosity and nonvolatile solid content of MUG resin were 3.16 mPa·s and 47.62%, respectively. And the pH and water tolerance of MUG resin were 8.0 and 100%, respectively.

3.1.2. GPC Analysis. Figure 2 shows the GPC chromatogram of MUG resins. The species with the large molecular weight was detected first and then followed by oligomer with the smaller molecular weight species in resin. The number-averaged molecular weight ($M_n$), weight-average molecular weight ($M_w$), polydispersity index (PDI), and the peak area portions of the GPC peaks of the MUG resin are shown in Table 2. As shown in Figure 2, retention time at 17.8
minutes was the low molecular weight (LMW) portion, whereas the other peak at 16.1 minutes was stated as high molecular weight (HMW) portion. The $M_n$ and $M_w$ values of MUG resin were relatively small, 334 g/mol and 355 g/mol, respectively, indicating that the resin contains a small amount of polymer materials. The PDI was 1.06, indicating that the molecular of the MUG resin has good uniformity. The addition reaction of glyoxal to melamine is easier and more complete than that of urea [14]. The primary addition reaction attached glyoxal molecules to the nitrogen of the amino group to form methylol compounds, which then condensed to form high molecular weight intermediate. Since a melamine molecule has three amino groups, hexahydroxy methylmelamine can be formed because hydrogen atoms may be replaced by melamine [15, 16]. The molecular weight of MUG resin is almost in the range of 290~470 g/mol, indicating that MUG resin can be enter the cell wall of wood [17].

3.1.3. FTIR Analysis. The chemical reactions involved in the synthesis of MUG resin are rather complex. In order to further study the chemical bonds that may be formed between the reactants, the FTIR spectra of MUG resin were done and are shown in Figure 3 [13, 18–22]. The strong and wide absorption band near 3250 cm$^{-1}$ is attributed to the superposition of the stretching vibration of N-H and O-H. Owing to the influence of the p-$\pi$ conjugation effect between the -NH$_2$ and C=O in the MUG resin, the stretching vibration peak of C=O shifts to lower wavenumber of 1690 cm$^{-1}$. The band at 2940 cm$^{-1}$ is related to the C-H asymmetric stretching vibration in CH$_2$. The band at 1640 cm$^{-1}$ is related to C=O stretching vibration. Due to the influence of hydrogen bond, the adsorption peak of C=O shifts to a lower wavenumber [11]. The peak at 1450 cm$^{-1}$ is attributed to the in-plane vibration of the triazine ring and the C-N stretching vibration in methyl. In addition, there is absorption band at 1380 cm$^{-1}$, which is attributed to the C-H bending vibration. When the free amino group of hydrogen is consumed, there is influence of the conjugation effect between the N-H in the amino group and C=O in the amide. The absorption peak at 1240 cm$^{-1}$ is related to C-N stretching vibration. The absorption peak at 1020 cm$^{-1}$ indicates that melamine, urea, and glyoxal have reacted to form a C-O-C ether bond.

3.2. Physical Properties of Treated Wood

3.2.1. The Weight Percent Gain, Density, Water Uptake, and Leachability of Control and Treated Wood. Since the hydrophobic MUG resin was incorporated into the wood, it is expected that the physico-mechanical properties of the treated wood will be improved. As shown in Table 3, the WPG and density of the treated wood samples increased as the concentration of the MUG resin solution increased. The highest WPG and density were 72.02% and 0.90 g/cm$^3$ for 55% MUG resin-treated wood samples. The higher the WPG, the more MUG resin that enters the wood. When there was a large amount of resin inside the wood, the toughness may decrease of the treated wood due to the
brittleness of the resin. Density of the wood sample was measured as an index of wood properties, and it is directly related to the WPG. Table 3 shows the density of control and treated wood samples. Compared with the control rubber wood, the density of the treated wood (55% MUG resin) increased from 0.62 g/cm³ to 0.90 g/cm³, a significant increase of 45.16%. From the perspective of density alone, the treated wood can be well used in floor and furniture manufacturing.

Water uptake of control and treated wood samples were measured to evaluate the water repellency upon water soaking. As shown in Table 3, the water uptake and leachability of the treated wood samples increased as the WPG increased. The control wood showed a weight gain of 105.05% after being soaked in water for 20 days, while the water uptake of the 55% MUG resin-treated wood was significantly reduced by 63% compared with the control wood, indicating improved water repellency of wood due to MUG resin incorporation. On the one hand, the hydrophobic MUG resin absorbed into the wood can occupy the space for water. On the other hand, there may be crosslink effect between MUG resin and hydroxyl groups in the wood, resulting in a decrease in the number of hydroxyl groups, thereby decreasing the water absorption of the wood. In order to maintain the durability of the modification, it is necessary to fix the resin inside the wood so that such will be treated with a small leachability. The leachability of the MUG resin-treated wood was measured for 20 days. As shown in Figure 4, the leachability rate of modified wood decreased slowly when the resin concentration was greater than 25%. Therefore, it can reduce the production cost with 25% MUG resin of treated wood.

3.2.2. The ASE of Control and Treated Wood. The stabilizing effect of incorporating MUG resin is mainly reflected in the dimensional changes of the treated wood, as measured by ASE of the radial, tangential, and volume. The ASE of the treated wood are shown in Figure 5. A large ASE value indicates a higher dimensional stability. In the 35% concentration range, the ASE of treated wood increased as the MUG resin concentration increased. The treated wood shows an ASE value of radial, tangential, and volume as high as 57.6%, 49.9%, and 62.6%, respectively, which means that impregnation will greatly limit the swelling of wood. Considering their small molecules, the MUG resin can enter the cell wall and then cured after high temperature. It is expected that the formed hydrophobic MUG resin will bulk the cell wall of wood, resulting in reduced water uptake of the cell wall.
wall, thereby improving dimensional stability of the MUG resin-treated wood. In fact, the MUG resin which enters the cell wall improves the dimensional stability of the wood, while the MUG resin deposited in the cell lumen has almost no effect. Thus, the modification results in reduced ASE for treated wood when the resin concentration was greater than 35%. When the resin concentration was 25%, the ASE values of radial, tangential, and volume were 56.1%, 49.1%, and 61.6%, respectively, which were very different from that of the modified wood with 35% MUG resin concentration.

3.3. Macrostructure and Microstructure of the Rubber Wood

3.3.1. Macrostructure of Control and Treated Wood. Figures 6 and 7 compare the changes in the structure of control and 25% MUG resin-treated wood transverse and radial sections. As shown in Figure 6(a), rubber wood is diffused porous wood, and some of the pore is filled with tylosis. There are many longitudinal parenchyma and obviously ray distinct to the naked eye. Photomicrographs for 25% MUG resin-treated wood at the transverse section is shown in Figure 6(b). The yellow MUG resin is deposited in the pore after curing. As shown in Figure 7(a), there are obvious ray markings at the radial section. The pore is arranged neatly and included milky rubber of control wood. There was a yellow transparent cured MUG resin for 25% MUG resin-treated wood at the radial section (Figure 7(b)).

3.3.2. Microstructure of Control and Treated Wood. SEM-EDX mapping was conducted to visualize the distribution of MUG resin in the wood and its elemental composition (Figure 8). As shown in Figure 8, it mainly contains carbon (C), oxygen (O), and nitrogen (N) elements, and three elements distributed uniformly along the contour of the inner wall of the cell lumen and cell wall. Carbon (C) and oxygen (O) elements come from rubber wood sample and MUG resin, while nitrogen (N) mainly comes from MUG resin. There were many nitrogen (N) in the cell wall of the MUG-treated wood sample, which indicates that MUG resin was not only deposited in the cell lumens but also stably exists in the cell wall.

3.4. Mechanical Properties of Treated Wood

3.4.1. Bending Properties. Bending performance of control wood and MUG resin-treated wood samples are presented in Figure 9. 25% MUG resin-treated wood and 35% MUG resin-treated wood showed improved modulus of elasticity.
(MOE) and modulus of rupture (MOR) values compared to the control wood. An increase of the MOE and MOR by 12.4% and 19.5% were achieved after treatment with 25% MUG resin, for 35% MUG resin treatment enhancement amounted to 16.5% and 20.7%, respectively. The MUG resin content increased from 25% to 35%, and the MOR of the MUG resin-treated wood increased slightly, but the difference between them was not significant. The incorporated

Figure 8: (a) SEM image of MUG-treated wood and EDX mapping showing the distribution of elements (b) carbon, (c) oxygen, and (d) nitrogen.

Figure 9: Bending properties and stress-strain curves of grain of control wood, 25% MUG-treated wood, and 35% MUG-treated wood.
MUG resin is located in cell lumen and cell wall, thus decreasing the MOR and MOE. The stress-strain (load-deflection) curve of the samples is given in Figure 9(b). The slope of the load-displacement indicated the modulus. The greater the slope, the greater the modulus, and the greater the rigidity; furthermore, the bending deformation hardly occurs in wood. The load-displacement curve shows a little difference of displacement before failure between the control and treated wood.

Figure 10 shows the fracture morphology of control and treated wood on broken bending samples. The control wood showed an irregular fracture surface with jagged fracture line.
(Figure 10(a)). The brittleness increased likely due to brittle fracture at high crosslinking density and short crosslinking formed between the components of MUG resin and wood. 25% MUG-treated wood showed a large fracture surface and jagged fractures with some smooth surface (Figure 10(b)). For sample treated by 35% MUG resin, the fracture morphology (Figure 10(c)) did not change very much, and fracture surface was smoother and denser than that of 25% MUG-treated wood, indicating a decrease in brittleness. Basically, 25% and 35% MUG treatment improved the elasticity of the wood, which showed that it could be applied to load-bearing structure.

3.4.2. Compressive Properties. Figure 11(a) shows that the compressive strength parallel to grain direction of 25% MUG-treated wood and 35% MUG-treated wood was 121.5 MPa and 129.8 MPa, respectively, and higher than that of control wood. An increase of the compressive by 19.2% was achieved after treatment with 25% MUG resin, for 35% MUG resin treatment enhancement amounted to 27.4%, respectively. The compressive process of control wood and treated wood include an elastic phase, plastic phase, yield phase, and failure phase (Figure 11(b)). During the elastic phase, the load and displacement increase proportionally. In the plastic phase, the slope of the load-displacement gradually decreased until it reaches the maximum loading. During the yield phase, the load remained basically constant and the displacement increased sharply. The 35% MUG-treated wood lasted for a long time at this stage, indicating that it is stable under longer compression. In the failure phase, the displacement remained constant, and the load decreased sharply, and the wood samples were destroyed.

3.5. Comparison with Different Treatment of Rubber Wood. Table 4 provides a comparison of physical and mechanical properties from different experimental studies of rubber wood. Under similar weight percent gain conditions, the mechanical properties of the UF resin, MUF resin, and furfurylation-treated wood were worse than 25% MUG-treated wood. However, it is known that wood impregnated with UF and MUF resins will release harmful volatile organic compounds. As shown in Table 4, the comparison of MOE between horizontally glued laminated wood and 25% MUG-treated wood have no significant difference. The failure of the laminated beam mainly extends along the adhesive layer. The strength properties of 25% MUG-treated wood compare favorably with the laminated veneer lumber (LVL) made from rubber wood, which is used as door, window frames, and flooring.

4. Conclusion

This study successfully synthesized and applied environment-friendly MUG resin to treat rubber wood. Experiment and analysis results show that as the MUG resin concentration increased, the weight percent gain and density increased, water uptake and leachability decreased, and the antiswelling efficiency increased at first and then decreased. Due to the hydrophobic MUG resin absorbed into the wood and fills the pores after curing, thus, the water uptake decreased. Furthermore, the MUG-treated wood sample possesses significantly improved dimensional stability compared to the control wood. The MUG-treated wood can be prepared when the MUG resin concentration was set as 25% and 35%, and the physical properties of treated wood was optimum. The incorporated MUG resin also helps to strengthen the wood scaffold, thereby improving overall mechanical properties. With good physical and mechanical properties, the MUG-treated wood can be well used as a promising alternative to existing engineered wood products for flooring, furniture manufacturing, and building construction. Further research should be conducted to study the combination of MUG resin and wood components in MUG-treated wood and investigate new technologies that can be used to increase resistance to thermal degradation and biodegradability, while maintaining the physical and mechanical properties of the treated wood.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.
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