Softened Wood Treated by Deep Eutectic Solvents
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1. INTRODUCTION
Softened wood (SW) generally has the advantages of light weight, low density, good elasticity, high porosity, and compressibility.1 Therefore, it is widely used in household, indoor decoration, and construction fields, such as decorative panels, softened wood flooring, wooden bricks, and softened wood furniture. However, traditional methods of wood softening often fail to meet the requirements of enterprises for softening wood. Here, inspired by the research related to wood softening, we propose a method for directly preparing softened wood (SW) using a new type of “ionic liquid” eutectic solvent (DES) owing to its low cost, environmental friendliness, recyclability, and other advantages. To improve the adaptability of the study, a total of five types of DESs were designed and prepared, and by the microwave-assisted DES treatment of natural wood (NW), the purpose of softening wood was achieved. Then, we conducted a series of comparative analyses and performance tests on NW and SW, including microscopic images, chemical composition, color difference, and mechanical properties. The results show that the wood softened by DES has become a highly porous network structure, and partial lignin, hemicellulose, and cellulose have been removed. At the same time, different degrees of color change, lower hardness, excellent mechanical flexibility, and a compression rebound rate of up to about 90% are obtained. The above-mentioned various properties of SW provide great potential for its application in wood products.

Received: May 14, 2020
Accepted: August 7, 2020
Published: August 24, 2020

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http://dx.doi.org/10.1021/acsomega.0c02223
ACS Omega 2020, 5, 22163−22170
Research has found that a variety of ionic liquids containing hydrogen bond acceptors can dissolve plant fiber raw materials; thus, a new type of “ionic liquidlike” deep eutectic solvent (DES) came into people’s vision; it is a certain molar ratio low-temperature eutectic mixture composed of hydrogen bond acceptors (such as quaternary ammonium salts) and hydrogen bond donors (such as carboxylic acids and polyols); its synthesis requires only a simple combination of the components, it is transparent at room temperature after stirring liquid, and no other purification treatment is required. DES not only has the advantages of ionic liquids, including high boiling point, low melting point, low volatility, and easy recovery, but it is also cheap, environmentally friendly, and biodegradable. Yiin et al. found that different concentrations of hydroxysuccinic acid and sucrose in water had good solubility properties for lignin and hemicellulose. Kumar et al. tried to dissolve lignin with DES composed of different molar ratios of lactic acid and choline chloride, and the research found that lignin was extremely soluble in this DES. Zhang et al. demonstrated that the removal of lignin and hemicellulose destroyed the structure of corn cob when treated with DES. Tan et al. successfully removed 100% hemicellulose, 88% lignin, and small amounts of cellulose from oil palm husks using acidic choline chloride/lactic acid DES. The discovery and application of DES provide new ideas for wood softening.

In this study, we tried to perform the microwave-assisted DES processing. The microwave-assisted technology has the advantages of short response time and high efficiency. Microwave-assisted interaction with polar molecules of DES can achieve rapid mass-transfer optimization of the reaction system, thereby dissolving some cellulose, hemicellulose, and lignin in natural wood and yielding a highly porous elastic structure (Figure 1). To improve the adaptability of the study, a total of five types of DESs were designed and prepared, as shown in Table 1. At the same time, the wood sample before the treatment is called NW and the sample after DES and microwave treatment is called SW for the purpose of facilitating distinction. Taking SW-1 as an example, the corresponding reaction solvent is DES-1, so the reaction solvents of SW-2, SW-3, SW-4, and SW-5 correspond to DES-2, DES-3, DES-4, and DES-5, respectively. In addition, this study also performed a series of characterization analysis and performance research on the reacted wood.

2. RESULTS AND DISCUSSION

2.1. Morphology Analysis. The microscopic features of NW and SW can be clearly seen through scanning electron microscopy (SEM) images. Figure 2a–c and d–f shows SEM images of NW and SW at different magnifications, respectively. From the SEM results, it can be seen that SW is mainly composed of tough wood fibers, vessels, and ray cells. Among them, tough wood fiber cells have a small cell cavity and a thick cell wall, randomly distributed between uniform single-row xylem rays (Figure 2a,b). After softening, the microstructure of the wood changed significantly, from the original compact and dense structure (Figure 2a) to a network structure (Figure 2d). Correspondingly, from the comparison of Figure 2b,e, it can be seen that the ductile wood cell wall becomes thinner, the cell cavity becomes larger, and the ray cell becomes less obvious. In general, the cell wall is mainly composed of chemical components such as cellulose, hemicellulose, and lignin, among which lignin is commonly found in the intercellular layer and at the corner of the cell wall (Figure 2c). The image shown in Figure 2f clearly illustrates the partial removal of lignin. NW lost a large amount of cell-to-cell connection material (mainly lignin), which makes the intercellular space become larger, and pores appeared in the cell corners, thus showing a highly porous network structure. This kind of structure gives SW a certain degree of elasticity and flexibility.

2.2. Chemical Structure Analysis. The XRD patterns and infrared spectra of NW and SW can reflect the internal chemical structure changes from the side. Figure 4a shows the XRD spectrum of NW and SW, which shows that the positions of the main diffraction peaks of NW and SW are almost the

![Figure 1. Schematic of microwave-assisted DES to soften wood.](http://pubs.acs.org/journal/acsodf)
same, both at $2\theta = 15.6^\circ$ (101 plane), 22.5$^\circ$ (002 plane), and $34.4^\circ$ (040 crystal plane). Meanwhile, the diffraction peaks at around $2\theta = 18^\circ$ all indicate the diffraction intensity of the amorphous region. This is a typical characteristic diffraction peak of type I cellulose structure, indicating that the crystal structure of cellulose in SW does not change much. Among them, the intensity of the diffraction peaks of SW-(3)–(5) at $2\theta = 15.6$ and 22.5$^\circ$ is relatively low, which may be because at the high temperature and in the presence of an excessive solvent, the reaction gradually penetrates into the crystallization zone, destroying the aggregate structure of cellulose.

As shown in Figure 3b, the characteristic absorption peaks exhibited by NW are the same as those in the existing literature, including absorption peaks at 3375 cm$^{-1}$ (O–H stretching vibration), 2925 cm$^{-1}$ (C–H stretching vibration), 1736 cm$^{-1}$ (C=O stretching vibration), 1505 and 1593 cm$^{-1}$ (aromatic nucleus skeleton vibration), 1242 cm$^{-1}$ (ether bond vibration). A decrease in the intensity of the absorption peak at 1242 cm$^{-1}$ (the C–O stretching vibration in lignin and hemicellulose) also proves this. Meanwhile, compared with NW, the characteristic absorption peak at 1034 cm$^{-1}$ (C–O stretching vibration in cellulose ether bonds) corresponding to the cellulose of SW also showed a significant decrease, indicating that cellulose might be partially dissolved in the deep eutectic solvent during processing. Therefore, NW not only lost part of the lignin and hemicellulose but also partial cellulose during the softening process.

Figure 2. SEM images of cross sections of NW (a–c) and SW (d–f).

Figure 3. (a) XRD patterns of the NW and SW. (b) FT-IR spectra of NW and SW.

2.3. Chemical Composition Content Analysis. The contents of cellulose, hemicellulose, and lignin of NW and SW are shown in Figure 4, which reflects the change of the main chemical composition of the wood cell wall. Linear polysaccharide cellulose, heterogeneous hemicellulose, and structurally variable lignin are the key components of the wood cell wall, which are linked to each other through a hydrogen-bonding network (between cellulose and hemicellulose) and covalent linkage (lignin and hemicellulose). It can be seen from Figure 4 that the microwave-assisted DES treatment resulted in changes in the contents of cellulose, hemicellulose, and lignin. Among them, the cellulose content decreased slightly and the change was not obvious. For hemicellulose, the highest hemicellulose removal rate was 27.9% (SW-4), and its hemicellulose content decreased from 21.5 to 15.5%, but the lowest hemicellulose removal rate was only 10.2% (SW-5).
Compared with cellulose and hemicellulose, it was found that lignin was most affected by DES treatment. The lignin content of NW was 26.1%, while the lignin content of SW-5 was only 8.2%; the lignin removal rate was as high as 68.5%, and the lowest lignin removal rate in SW is 31.4% (SW-2), which is higher than the highest hemicellulose removal rate in SW. The general decline in the lignin content proves that DES has a good ability to dissolve lignin, especially DES-5, which means that, in this study, the polar functional groups provided by lactic acid and choline chloride are more likely to occur with the lignin reaction. In fact, DES provides a mild acid–base catalytic mechanism that selectively breaks unstable ether bonds between phenylpropane units, depolymerizing lignin and separating it from biomass, thus changing the chemical structure of wood cells.25

2.4. Color Difference Analysis. The corresponding L, a, and b values of NW and SW in Lab color space are shown in Figure 5c–e, where it can be seen that the color of NW has changed a lot after DES treatment. In terms of lightness and darkness, the L values of the five types of SW all decreased compared to those of NW, and the decrease was obvious, indicating that the SW was significantly darkened (Figure 5c); in terms of the red-green color represented by the a value, SW and NW are both positive numbers, and the change range is not large, indicating that the red hue of the two has not changed significantly (Figure 5d); in the yellow-green aspect represented by the b value, both SW and NW are also biased toward yellow, but SW-2, SW-3, and SW-4 are more obvious (Figure 5e). Figure 5ab also proves this result, which shows that after DES treatment, the color of SW deviates from the original color, close to the color of its reaction solution, and the brightness of the wood shows a decreasing trend. It can be clearly seen from Figure 5a and b that SW changes from the primary color to yellow, tan, and dark brown. One of the most important reasons for this change is that lignin separates from the biomass and generates a lot of free radicals during the DES treatment, which are unstable and easily react with neighboring molecules to form peroxides and finally decompose into colored compounds, and lignin itself is also rich in many chromophoric groups, such as carbonyl groups, carbon–carbon double bonds, and benzene rings, whose chemical composition changed the color of the wood.26,27 Since the ionic strength provided by each DES is different and the reaction results with lignin are also different (expressed as the change in the lignin content from NW to SW in Figure 4), the final color of SW is also different.

2.5. Mechanical Performance. Figure 6 shows the mechanical properties of NW and SW. It can be seen that there are slight differences in the mechanical properties of different types of SW, and one of the reasons for this is that different formulations of DES give SW a different degree of the flexible structure during the softening process. Hardness is an important indicator for measuring the softness of wood.28 It can be seen from Figure 6a that the surface hardness of SW is significantly reduced after softening treatment. The hardness value of NW is about 86 HSA, and the highest hardness value of SW is about 63 HSA (SW-2), which is about 26.7% lower than that of NW. The lowest hardness value of SW is about 45 HSA (SW-5), which is about 47.7% lower than that of NW.
The general decrease in SW hardness proves that DES-assisted microwave treatment has a certain plasticizing effect on the cell wall components in natural wood, making the wood soft.29

The mechanical compressibility of NW and SW in the tangential direction is illustrated in Figure 6b. Both NW and SW can withstand up to 70% compression deformation, but the stress used by SW is much smaller than that by NW, showing its excellent flexibility performance. Typical three-state curves were observed in the compressive stress–strain curves of NW and SW, including a low-strain linear elastic deformation region of less than 5%, and a region where the stress between 5 and 60% strain showed a steady state, and areas where stresses increase sharply afterward, leading to densification of the wood.30,31 However, NW showed a significant inflection point at a strain of about 5% and entered the stage of nonlinear elastic deformation. After the stress was released, the strain could not be fully recovered, and a partial plastic deformation occurred. This phenomenon did not occur in SW.

The compression resilience rates of NW and SW are shown in Figure 6c. When the compressive strains of NW and SW reach 50%, the loading is stopped. After 15 min of unloading, NW can reach a rebound rate of about 30%, while SW can reach a rebound rate of about 65–90%, which is 2–3 times of NW. After 24 h of unloading, the compression resilience rate of NW increased by about 3% compared with 15 min after unloading, while the growth range of SW was between 5 and 9%. Among them, the most noticeable is SW-5, which has a compression resilience rate of 95% after 24 h. In addition to this, a cyclic compression test with 30 loading–unloading cycles was carried out on NW and SW with a constant strain of 30% to evaluate their fatigue resistance (Figure 6d). The results show that the height retention (expressed as the percentage of the original height) of SW reaches a maximum of about 98% (SW-5) and a minimum of about 93% (SW-2) after 30 compression cycles, and the resulting plastic deformation is very small compared with that of NW, which highlights the structural robustness of SW. The high compression resilience rate and height retention of SW indicate that the wood treated with DES is a material with good mechanical compressibility and fatigue resistance.

3. CONCLUSIONS

In summary, we have demonstrated an effective approach to make a kind of softened wood that is highly porous, variable in color, mechanically resilient, made directly from natural poplar by DES-assisted microwave treatment, and has a larger cell cavity and a thinner cell wall, thus showing a flexible network structure. This structure allows SW to obtain lower hardness, good mechanical compressibility, and good fatigue resistance. Compared with other chemical agents that make wood soft, DES is more energy-saving and environmentally friendly because it can be recycled after use, and its characteristic reaction with lignin can bring different color changes to SW. The above-mentioned properties of SW treated with DES make us believe that it has broad development prospects in the fields of household furnishing, construction, furniture, wood products, and so on.

4. MATERIALS AND METHODS

4.1. Materials. The samples used in the experiment were poplar wood (Populus tomentosa Carr.), provided by Yihua Life Technology Co., Ltd. (Shantou, China) with an air-dry density of 0.37 g/cm³ and a specification of 10 mm (length) × 10 mm (width) × 7 mm (thickness). The anhydrous ethanol used in the experiment was purchased from Guangdong Guanghua Sci-Tech Co., Ltd., China; choline chloride was provided by Shanghai Canspec Scientific Instruments Co., Ltd., China.; lactic acid was provided by Tianjin Fuyu Fine Chemical Co., Ltd., China; malic acid and oxalic acid were provided by Xilong Scientific Co., Ltd., China; and histidine and proline were...
obtained from Tianjin Benchmark Chemical Reagent Co., Ltd., China. All of the chemicals used were of analytical grade.

4.2. Fabrication of Soft Wood. First, the poplar woodblocks were dried in an air-dry oven at 102 °C for 5 h, and then, the dried wood samples were immersed in an anhydrous ethanol solution for 10 h to remove gums and resins in the wood.\(^{32}\) Next, the sample was immersed in the boiling DES solution and reacted for 5 h. Then, the sample was microwaved three times repeatedly at a microwave power of 600 W and a microwave duration of 20 s each time. It is worth noting that the sample needs to be taken out of the microwave oven after a single microwave treatment to prevent the sample from carbonizing. After the microwave treatment was completed, the sample was again immersed in the DES solution for reaction for 2 h, and the reaction temperature was 80 °C. Next, wash the sample repeatedly with deionized water and finally dried with a freeze dryer for 48 h at a temperature of −50 °C.

4.3. Scanning Electron Microscopy. An FEI Quanta 200 scanning electron microscope (SEM) was used to observe the morphological characteristics of the sample at an accelerator voltage of 30 kV. The specific method is as follows: use a glass knife to cut the freeze-dried NW and SW into small pieces, pay attention to keeping the sample structure intact, then use tweezers to fix them on the conductive carbon glue, and then put them into the vacuum coating instrument to spray gold films for 30 s. Finally, use SEM to observe the vessels, cell wall, and cell cavities of the sample.

4.4. X-ray Diffraction and Fourier Infrared Spectroscopy. An X-ray diffractometer (Ultima IV, from Rigaku Co., Ltd., Japan) was used to characterize the molecular structure of NW and SW samples. The specific method is as follows: crush NW and SW into small and uniform powders, take an appropriate amount, and flatten it in the sample tank. The sample was then scanned using an X-ray diffractometer with a scanning voltage of 40 kV, a scanning range of 2θ = 5−60°, and a scanning speed of 10°/min.

A Fourier transform infrared spectrometer (VERTEX 80V, from Bruker Co., Ltd., Germany) was used to characterize the infrared spectra of functional groups of NW and SW. The specific method is as follows: grind NW and SW into wood powder of 80 to 100 mesh, mix and tableting with KBr, put it into a sample pool, and scan to obtain the infrared spectrum.

4.5. Chemical Composition Content Test. The cellulose, hemicellulose, and lignin contents (including acid-insoluble lignin plus acid-soluble lignin) of NW and SW samples were tested by the Laboratory Analytical Procedure (LAP) written by the National Renewable Energy Laboratory (Determination of Structural Carbohydrates and Lignin in Biomass).\(^{33}\) After hydrolyzing the wood powders of NW and SW with 72% concentrated sulfuric acid, the concentration of the liquid was diluted to 4% and then filtered using a G3 glass sand funnel. Next, the acid-insoluble lignin content is calculated by weighing the filtered residue, and the acid-soluble lignin content is calculated by measuring the ultraviolet absorbance of the filtered filtrate. The filtrate was diluted by a certain multiple, and then, the sugar content was analyzed by high-performance liquid chromatography (HPLC). In addition, the contents of cellulose, hemicellulose, and lignin are calculated according to formula 1.

\[
\text{content} = \frac{M_S}{M_W} \times 100\%
\]  
(1)

In the formula, \(M_S\) refers to the mass of each chemical component in the sample and \(M_W\) refers to the total mass of the samples.

4.6. Color Difference Test. A color difference meter (RM200QC, from Xrite Co., Ltd.) was used to measure the surface color of the NW and SW samples with the standard light source being D65 at normal temperature. The length and width of the samples were 10 mm and the thickness was 7 mm. To reduce the error, we use a color difference meter to take three different points on each sample surface, measure twice at each point, and then calculate the average value. The CIE \(L^*a^*b^*\) (1976) standard colorimetric characterization system of the International Organization for Standardization (ISO) was used for quantitative characterization of colors to analyze their color changes.\(^{34}\)

4.7. Mechanical Performance Test. The hardness of NW and SW samples was measured by a Shore durometer LX-D and a Shore durometer LX-A, respectively. Each test specimen is measured three times to reduce the error. After the measurement is completed, the hardness of the samples is converted from the HSD value to the HSA value by referring to the Shore hardness comparison table.

The compression test was performed using a computer-controlled electronic universal testing machine (AG-IC/100KN, Shimadzu Co., Ltd., Japan). After several preliminary experiments, it was determined to load at a speed of 2 mm/min until the sample compressive strain reached 70%, and the data was recorded by a computer to obtain the stress–strain curves of NW and SW. The compression resilience rate is measured by placing the sample on a universal mechanical testing machine to load it, and the load speed is still 2 mm/min. To obtain a more ideal experimental effect, the loading is stopped when the compressive strain of the sample reaches 50%. The thickness of the sample is measured after 15 min and 24 h, and the compression resilience rate is calculated by formula 2.\(^{35}\) At the same time, the loading–unloading cycle compression test was conducted under 30% compressive strain of NW and SW to verify the structure–performance relationship of SW. The experimental directions of the above hardness test and compression test are along the tangential directions of NW and SW, the length and width dimensions of the sample used in the experiment are 10 mm, and the thickness of the sample is 7 mm.

\[
R = \frac{(T_2 - T_1)}{T_1} \times 100\%
\]  
(2)

In the formula, \(R\) refers to the compression resilience rate of the sample (%), \(T_1\) refers to the thickness of the sample when the compressive strain reaches 50% (mm), and \(T_2\) refers to the thickness of the sample after the unloading.

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**Funding**

The authors gratefully acknowledge the financial support from the project funded by the Project of Science and Technology Plan of Beijing Municipal Education Commission (KM202010012001), the Special Scientific Research Fund of Construction of High-level teachers Project of Beijing Institute of Fashion Technology (BIFTQG201805), Yihua Lifestyle Technology Co. Ltd. Projects (YH-NL-201507 and YH-JS-JSKF-201904003), and the National Natural Science Foundation of China (31800471).

**Notes**

The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**

The authors gratefully acknowledge financial support from the Peoples’ Republic of China.

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