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Article

Preliminary Study on the Physical and Mechanical Properties of Poplar Wood Modified by Waterborne Glucose Silicone Resin

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Abstract: In order to improve the performance of soft plantation wood, an environmentally friendly inorganic-organic hybrid wood modifier was developed. First, using urea and melamine as crosslinking agents, the waterborne glucose silicone resin (MUG) was prepared with glucose under the catalysis of inorganic acid and metal ions. Then MUG resin was diluted to 10% and 20% mass fraction, and compounded with sodium silicate (S) of 20% and 10% mass fraction, so the inorganic-organic hybrid $G_{10}S_{20}$ and $G_{20}S_{10}$ wood modifier were obtained respectively. Then plantation poplar wood (*Populus tomentosa*) were impregnated and modified with them. Their physical and mechanical properties were tested and compared with those of the wood treated with S of 20% mass fraction (S_{20}). Infrared analysis showed that amino resin characteristic structure (CO-NH-) existed in MUG resin. The resin has good permeability. Compared with S_{20} modified wood, the degree of shrinkage of $G_{10}S_{20}$ or $G_{20}S_{10}$ modified wood is reduced, their moisture absorption is reduced, and their dimensional stability is improved. Waterborne glucose silicone modifier can effectively improve the wood density, modulus of elasticity, modulus of rapture and compression strength. SEM analysis showed that the cell wall of $G_{20}S_{10}$ modified wood was significantly thicker than the untreated wood, and there were columnar and granular solid substances attached in some cell cavities, ducts and corners, etc. EDX showed that the number of Si elements on the cell wall was significantly increased compared with the control, indicating that the modifier effectively entered the wood cell wall. The waterborne glucose silicone resin can greatly improve the physical and mechanical properties of wood through organic-inorganic hybridization. It is a green, non-formaldehyde, eco-friendly, low cost, compound wood modifier with broad application prospects.

Keywords: poplar; glucose resin; sodium silicate; impregnation modification; wood properties

1. Introduction

Wood is a natural biomass material with a wide range of applications. Vigorously developing plantation wood can effectively alleviate the resource and environmental pressure caused by the sharp decline of natural forests worldwide [1]. Plantation wood has low density, poor quality and limited application range. Wood modification can improve its performance and product added value [2]. Impregnation treatment is a commonly used modification method for plantation wood. The main impregnations include low molecular weight urea-formaldehyde resin, phenolic resin, melamine-formaldehyde resin, melamine-urea-formaldehyde resin and other formaldehyde-based resins prepolymer, and reactive monomers such as furfural, dimethylol dihydroxy ethylene urea and acetic anhydride [3]. The release of small molecule volatiles such as formaldehyde during production and use will endanger human health and environmental safety. Low reactivity (dimethoxyacetaldehyde), strong pungent odor (glutaraldehyde), high cost, poor resin performance and other issues restrict the application of non-formaldehyde crosslinking agents [4]. Environmental protection and low cost are the inevitable trends in the development of resins for wood impregnation.

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Glucose is the most extensive biomass resource, and the aldehyde group on its molecule can partially or completely replace formaldehyde. Viswanathan et al. under the action of inorganic acid and metal ion catalysts, heating (50~200℃) sugars and urea, phenol or melamine, can generate soluble resin and organic carboxylic acid crosslinking and curing [5]. Adding a crosslinking agent can accelerate the decomposition of glucose into the active monomer 5-hydroxymethyl furfural (5-HMF) under hot acid conditions [6]. Glucose phenol resin synthesized with glucose as raw material, the bonding strength is 21.4MPa, the water resistance time is more than 30h, and the water resistance and bonding strength are good [7-8]. Manganese sulfate and nickel sulfate can effectively catalyze glucose/melamine resin and glucose/urea Synthesis of resin [9-10]. Sodium silicate is a soluble inorganic silicate, rich in resources, environmentally friendly and non-toxic. It can effectively improve wood hardness, strength, corrosion resistance and flame retardancy, etc. [11-12], but sodium silicate is alkaline and easy to absorb moisture. It will cause problems such as shrinkage of the treated material and non-lasting modification effect [13].

In this paper, synthetic resin of glucose and inorganic sodium silicate are compounded to prepare green and environmentally benign organic-inorganic hybrid wood impregnation modifier. The plantation poplar wood is impregnaed, and the properties of modified wood are tested and analyzed. This new wood impregnation modifier is of great significance.

2. Materials and Methods

2.1 *Materials*

Fast-growing poplar (*Populus tomentosa*), with a moisture content of 9%-12%, and a density of air-dried wood of 0.436g/cm^3, the size of these specimens was 300mm (longitudinal) × 25mm (radical) × 25mm (tangential). Glucose ($C_6H_{12}O_6$) was purchased from Xilong Science Co., Ltd.; melamine ($C_3H_6N_6$), urea (CH_4N_2O) were obtained from Tianjin Fuchen Chemical Reagent Company; sodium silicate (Na_2SiO_3), itaconic acid ($C_5H_6O_4$), boric acid (H_3BO_3) were from Aladdin Reagent Co., Ltd.; Copper sulfate ($CuSO_4$), ammonium chloride (NH_4Cl) were purchased from Sinopharm Chemical Reagent Co., Ltd. The concentration of self-made sodium hydroxide and hydrochloric acid solution is 20% mass fraction.

2.2 *Preparation of waterborne glucose silicone resin*

Put 260g distilled water and 370g glucose in a three-necked flask, stir and raise the temperature to 60°C before adding 26g melamine. After it is dissolved, add an appropriate amount of acid catalyst copper sulfate and ammonium chloride to adjust the pH value to about 3, and then add 49g urea. Stir and heat up to 100°C and keep it until the color of the solution finally becomes reddish brown (the color of the solution changes as light blue-colorless and transparent-light yellow-orange-dark red-reddish brown during the whole process), adjust the pH value of the solution to 6.5-8.5. Cool down to 40°C, add 2wt% curing agent, stir for 1h and discharge to obtain waterborne glucose silicone resin (noted as MUG), with a solid content of about 52.6%, a viscosity of about 4.2 mPa·s , and a water solubility is higher than 9. Dilute the MUG resin solution to 10% or 20% by mass, and compound it with inorganic sodium silicate solution of 20% or 10% mass fraction to prepare $G_{10}S_{20}$ or $G_{20}S_{10}$ waterborne glucose silicone resin wood impregnation modifier; The S_{20} modifier is the inorganic sodium silicate aqueous solution of 20% mass fraction.

2.3 Wood Impregnation

Use vacuum-pressure impregnation tank (SBK-450B) to impregnate the specimens with modifiers S_{20}, $G_{10}S_{20}$ and $G_{20}S_{10}$ respectively, and the impregnation process is vacuuming (−0.09 MPa, 0.5h) → liquid injection → pressurization (1.0 MPa, 24h) → pressure relief. After being taken out, specimens are first air-dried to about 50% moisture content, then dried at gradually raised 103°C to oven dry, and S_{20}, $G_{10}S_{20}$ and $G_{20}S_{10}$ modified poplar wood were obtained respectively.

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2.4 Physical and mechanical properties

Weight percentage gain (WPG) and bulking effect (BE) are used to characterize the size and quality changes of wood before and after impregnation. The WPG and BE were calculated as the following formulas (1) and (2). According to the provisions of GB1934.2-2009, GB1931-2009 and LY/T 2490-2015, at 86% relative humidity, record the size and mass changes of the specimens from oven dry to moisture equilibrium, and their size (volumetric) swelling coefficients (A), equilibrium moisture content (EMC) and anti-swelling efficiency (ASE) were calculated according to the formulas (3), (4) and (5) respectively. Specimen size is 20mm (longitudinal) × 20mm (radical) × 20mm (tangential), 10 samples per group.

$$WPG\ (\%) = (m_1-m_0)/m_0\times100 \quad (1)$$

$$BE\ (\%) = (v_1-v_0)/v_0\times100 \quad (2).$$

Here, m_0 and m_1 are the oven-dry mass (g) of the wood before and after treatment, v_0 and v_1 are the oven-dry volume of the wood before and after treatment.

$$A\ (\%) = (C_1-C_0)/C_0\times100 \quad (3).$$

$$EMC\ (\%) = (m_2-m_1)/m_1\times100 \quad (4).$$

$$ASE\ (\%) = (A_0-A_1)/A_0\times100 \quad (5).$$

Where, C_0 and C_1 are the size (mm) / volume (mm^3) of the wood before and after moisture absorption, m_2 is the mass (g) of the moisture equilibrium specimen, A_0 and A_1 are the control and treated wood size (volume) swelling coefficients (%), respectively.

Measure the mechanical properties of the modified wood according to the GB/T1933-2009, GB/T1936.1-2009, GB/T1936.2-2009 and GB/T1935-2009 standards, including the modulus of rupture (MOR), modulus of elasticity (MOE), and compression strength (CS) respectively.

2.5 Fourier Transform Infrared Spectroscopy (FTIR) Analysis of MUG

The MUG resin was cured in an oven at 120°C for 2 hours, and the cured resin was pulverized and sieved with a pulverizer to produce 160-200 mesh powder. Glucose and urea powder were taken as the control and FTIR spectrometer (Nicolet 6700, Nicolet, USA) was used. The scanning range was 4000-400cm^{-1} with a resolution of 4cm^{-1} and 32 scans.

2.6 Micromorphology Characteristic

Use a slide-away microtome to cut the middle slice of the 5mm×5mm×3mm sample, fix it on the metal stage with conductive glue, spray gold, and observe the modifier distribution in the wood with a scanning electron microscope (S4800, Hitachi, Japan), observe the changes and distribution of Si before and after the treatment by the built-in energy spectrometer (EDX).

3. Results and Discussion

3.1. Infrared spectroscopy (FTIR) analysis of resin

Fig. 1(a) (b) is the infrared spectrum of the resin. 3600-3100 cm^{-1} is the coupling vibration peak of -NH and -OH [14]; the absorption peaks of 2926cm^{-1}, 2937 cm^{-1}, and 2885cm^{-1} are attributed to the stretching vibration of -CH_2- [15-16], 1667cm^{-1} is the stretching vibration peak of the urea amide carbonyl group; 1630cm^{-1} is the characteristic peak of amide absorption band II, reflecting the in-plane bending vibration of the N-H bond of urea CO-NH_2 [17]; 1552cm^{-1} is the coupling of the in-plane bending vibration of the N-H bond in CO-NH- and the stretching vibration of the C-N bond [17-18]; 1421cm^{-1} is the CH bending vibration in -N-CH_2- and -CH_2-O- [18]; 1120cm^{-1}. The absorption peak at 1029cm^{-1} is attributed to the asymmetric stretching vibration of -C-O [16]. Comparing the reaction raw material and the resin spectrum, it can be seen that the flexural vibration of the amide carbonyl group and the N-H bond at 1667 cm^{-1} at 1630 cm^{-1} of urea weakened, and the characteristic peak of

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CO-NH- amino resin appeared at 1552 cm^{-1}. It indicates the formation of secondary amino groups and the cross-linking reaction between urea and glucose.

Glucose can be acidified and hydrolyzed to form 5-HMF at high temperature [19-20]. As shown in Figure 1(c), it is first isomerized to fructose, and then further dehydrated to form 5-HMF, which contains aldehyde groups, hydroxymethyl and furan rings, and is chemically active. Inferred from infrared analysis, 5-HMF can polymerize with urea to form secondary amino groups, as shown in the red mark in Fig. 1(d). Correspondingly, Fig. 1(e) shows the color change of the resin reaction. As the resin is further cross-linked, the molecular weight increases and the resin color becomes darker.

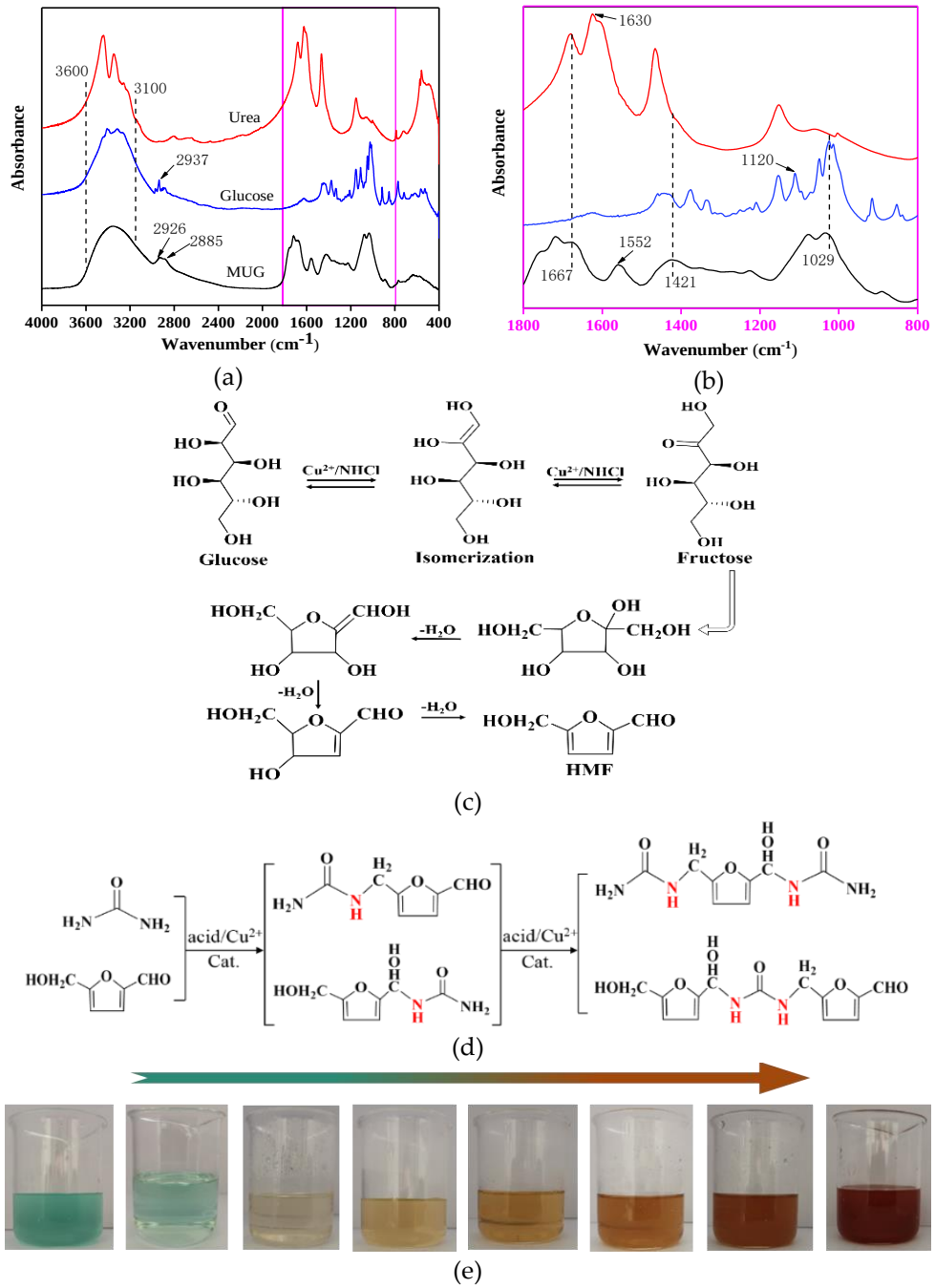

Fig. 1 Schematic illustration of: (a), (b) Infrared spectrum image of resin, (c) The reaction process of glucose isomerization-dehydration to form 5-HMF, (d) Synthetic mechanism of linear MUG resin, (e) Color change during resin synthesis.

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3.2. Physical properties of wood

The WPG is an index for evaluating the permeability of the modifier. From Table 1, the WPG of S_{20}, $G_{10}S_{20}$ and $G_{20}S_{10}$ treated wood are 25.63, 43.23 and 55.79%, respectively. Among them, $G_{10}S_{20}$ and $G_{20}S_{10}$ have the same solid content, but the WPG of $G_{20}S_{10}$ treated wood increases by 29% compared with $G_{10}S_{20}$, indicating that the higher the MUG resin content in the GS modifier, the better the penetration and fixation of the modifier, indicating that the crosslinking and curing of MUG resin in the wood can effectively promote fixation of sodium silicate solution in wood.

The bulking effect (BE) represents the swelling effect of the modifier on the wood cell wall. The modifier enters the non-crystalline area of the wood cell wall, increases the distance between the microfibrils and the fibrils in the cell wall, and expands the volume of the wood [21].

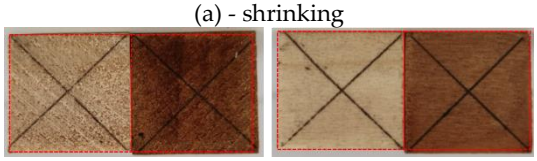
Table 1 Weight percent gain , bulking effect and density of the modified wood

Groups	WPG (%)	BE (%)	Density(g·cm^{-3})
Control	--	--	0.385±0.01
S_{20}	25.63±0.29	-14.90±0.65	0.529±0.01
$G_{10}S_{20}$	43.23±1.22	-0.55±2.37	0.522±0.10
$G_{20}S_{10}$	55.79±2.89	7.84±0.69	0.515±0.10

Note: The values in the table are the average ± standard deviation, the same below.

The BE of S_{20}, $G_{10}S_{20}$ and $G_{20}S_{10}$ treated wood are -14.90, -0.55 and 7.84%, respectively. The mass percentage of S_{20} is small (20%), but the shrinkage of the treated wood is 14.9%. It shows that pure sodium silicate solution treatment will cause serious shrinkage of wood (Fig 2a), which is caused by its alkaline dissolution on wood hemicellulose and other components [22]. $G_{10}S_{20}$ treatment wood shrinks slightly, while $G_{20}S_{10}$ has a significant swelling effect on wood cell walls (Fig 2b), indicating that MUG resin can effectively inhibit wood shrinkage caused by inorganic sodium silicate, but as the content of MUG resin increases, the cell wall is fully swelled, The filling and fixing effect of the modifier will cause the volumetric expansion of the treated material, and the compatibilization rate of the modified wood increases with increasing MUG resin content. Density is an important index to evaluate wood mechanical properties. From Table 1, the densities of S_{20}, $G_{10}S_{20}$ and $G_{20}S_{10}$ treated specimens are 0.529, 0.522, 0.515g·cm^{-3}, respectively, all higher than the untreated wood. The highest density of S_{20} treated wood is caused by its severe shrinkage and smaller volume.

(a) - shrinking



(b) - expansion

Fig. 2 (a) - S_{20} modified material size shrinkage drawing,
(b) - $G_{20}S_{10}$ modified material size expansion drawing.

3.3 Hygroscopicity and dimensional stability of wood

The EMC of different modified wood at 86% relative humidity is shown in Figure 3. The EMC of S_{20} and $G_{10}S_{20}$ treated wood is 34.2% and 19.8%, which are 96.5% and 13.7% higher than that of the control respectively. This is due to the strong hygroscopicity of inorganic sodium silicate, which makes the modified specimen easy to absorb moisture. The EMC of $G_{10}S_{20}$ and $G_{20}S_{10}$ treated wood is 42.1% and 65.7% lower than that of S_{20} treated wood respectively, indicating that MUG resin can seal the hygroscopic groups of sodium silicate and wood to a certain extent and reduce its hygroscopicity. The EMC of the $G_{20}S_{10}$ treated wood is 11.7%, which is 32.7% lower than that of the control, indicating

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that as the MUG resin content increases to a certain extent, the moisture absorption problem of the treated wood can be effectively solved.

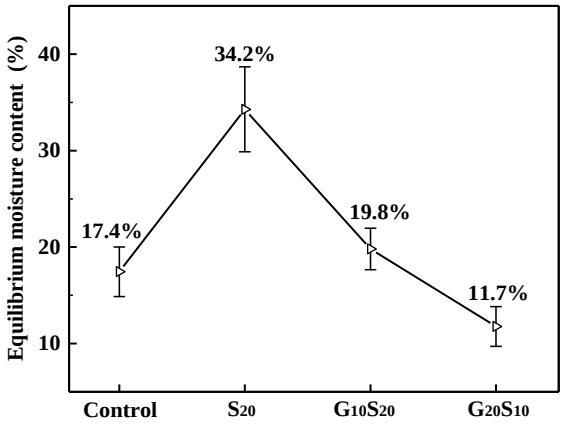

Fig.3 Equilibrium moisture content of the modified wood

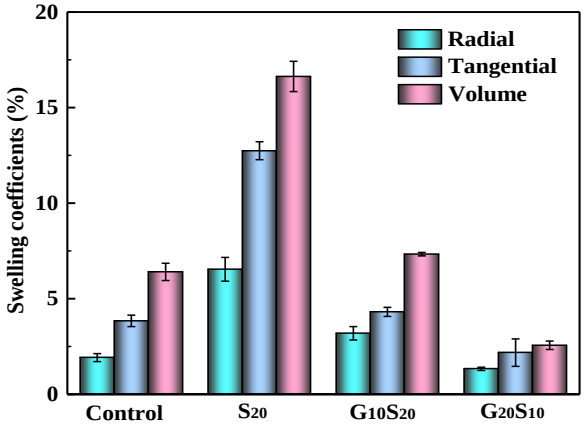

Fig.4 Swelling rate of the modified wood

The swelling rate of modified wood at 86% relative humidity is shown in Fig. 4. The radial, tangential and volume swelling rates are all in order as S_{20} > $G_{10}S_{20}$ > W >$G_{20}S_{10}$, and all is radial < tangential < volume, which is consistent with the heterogeneous nature of the structure of wood itself. Among the modified wood, $G_{20}S_{10}$ has the smallest swelling rate, indicating that its size is the most stable. The ASE test results (Table 2) show that the radial, tangential and volume ASE of the modified wood are all in order as $G_{20}S_{10}$ > $G_{10}S_{20}$ > S_{20}, and the dimensional stability of S_{20} and $G_{10}S_{20}$ is lower than that of the control. The radial, tangential and volume ASE of $G_{20}S_{10}$ are 30.8%, 43.3%, and 60% respectively, and its dimensional stability is significantly improved compared with the control. More MUG resin in the compound modifier, higher the dimensional stability of the modified wood.

Table 2 Anti-swelling efficiency (%) of the modified wood

Group	Radial	Tangential	Volume
Control	--	--	--
S_{20}	-241.11±32.26	-232.05±12.14	-221.71±12.33
$G_{10}S_{20}$	-66.40±18.07	-12.33±6.29	-14.45±1.37
$G_{20}S_{10}$	30.85±4.10	43.30±18.86	60.01±3.37

Results show that there is much waterborne glucose silicone resin impregnated into wood. On one hand, the dried and solidified glucose resin result the physical package of the sodium silicate particles, which hinders the moisture absorption path of sodium silicate; on the other hand, the glucose resin prepolymer can interact with certain wood cell wall components. Some hydroxyl groups are cross-linked and the hygroscopic groups are reduced; in addition, the cross-linked structure formed by curing organic resin and inorganic silicate fills the wood voids, prevents moisture from entering, and improves wood dimensional stability.

3.4 Mechanical properties of wood

The cellulose in the wood gives it strength and rigidity, the lignin guarantees its hardness and toughness, and the cellulose microfibrils in the internal structure play a role in supporting the wood. When the wood is stressed, the weakest part of the fibril connection shows relative slippage, which causes the destruction of the wood structure [23]. Fast-growing poplar has a short growth cycle, high content of colloidal wood fibers and is not completely wrapped by lignin. When a load is applied, relative slippage between wood filaments is easy to occur, and the mechanical properties of the material are poor [24]. From Table 3, the MOE, MOR, and CS of modified wood is all significantly higher than that of the control. Among the modified wood, $G_{10}S_{20}$ has the highest MOE, MOR and CS, which increases by 72.2, 50.8 and 113.6% respectively compared with the control. Their MOE and

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CS is in order as S_{20} < $G_{20}S_{10}$ < $G_{10}S_{20}$, and their MOR is in order as $G_{20}S_{10}$ < S_{20}< $G_{10}S_{20}$. Compared with S_{20}, the $G_{20}S_{10}$ has slightly lower MOR, because inorganic silicon is a rigid substance. The content of inorganic silicon in S_{20} modifier is more, its mechanical support effect is more obvious, and it contributes more to the bending strength of wood.

Table 3 Mechanical properties of the modified wood

Group	MOE (GPa)	MOR (MPa)	CS (MPa)
Control	10.57±0.61	92.17±4.12	58.02±4.79
S_{20}	16.20±0.55	131.87±2.98	119.17±8.98
$G_{10}S_{20}$	18.21±0.91	139.33±2.51	123.94±2.18
$G_{20}S_{10}$	18.02±0.22	121.52±2.05	122.20±4.54

The compound of sodium silicate and glucose resin can further improve the bending resistance of wood. The reason may be [11, 25]: the inorganic sodium silicate deposited on the cell cavity and cell wall can increase the resistance of the wood to external loads ability; MUG resin reacts with the active groups on the cell wall and wraps around the cell wall microfibrils, inhibiting the free sliding of the microfibrils; the modifier inflates the cell wall micropores to reduce the moisture content of the cell wall and weaken the plasticizing effect of water on microfibrils. It can be seen from Table 1 that the weight gain rate of $G_{20}S_{10}$ treated wood is higher than that of $G_{10}S_{20}$ under the same modifier solid content. It shows that the organic-inorganic synergistic effect produced by the compound of sodium silicate solution and glucose resin makes it easier for the modifier to penetrate the wood, and the mechanical properties of the wood are improved to a greater extent.

3.5 Microstructure Analysis

The porous hierarchical structure of wood helps it absorb modified substances and improve the performance of wood. Using SEM and EDX to investigate the internal structure changes before and after wood modification, and to explore the distribution and combination of MUG resin and sodium silicate in wood. The further analysis of the modification mechanism lays the foundation.

It can be seen from Figure 5 that the wood fiber cells, ray cells and duct tissues of the control are all in a hollow state, and the energy spectrum scan shows that a small amount of Si is distributed in the cell wall of the control (Figure A, a); while the cell wall of the $G_{20}S_{10}$ treated wood is obviously thickened, in some cell cavities, ducts and cell corners, etc., columnar and granular solid substances are attached or filled (the red arrows in Figure B and C); compared with the control, the amount of Si elements in the cell walls and ducts of the $G_{20}S_{10}$ treated wood increases significantly (Figure a, b). It shows that the waterborne glucose silicone resin modifier can effectively enter the characteristic pores of poplar wood cell walls, cell cavities and ducts. After solidification, it can cross-link with the cell wall substances, wrap around the microfibrils, and deposit and fill in the wood voids. It effectively increases the ability of wood to resist external forces and reduces the moisture adsorption sites and moving channels inside the wood. The SEM-EDX analysis results are consistent with the experimental results of mechanical properties and dimensional stability. The filling of wood pores and crosslinking with cell wall substances by the water-based glucose silicone resin modifier are the main factors to improve the physical and mechanical properties of wood.

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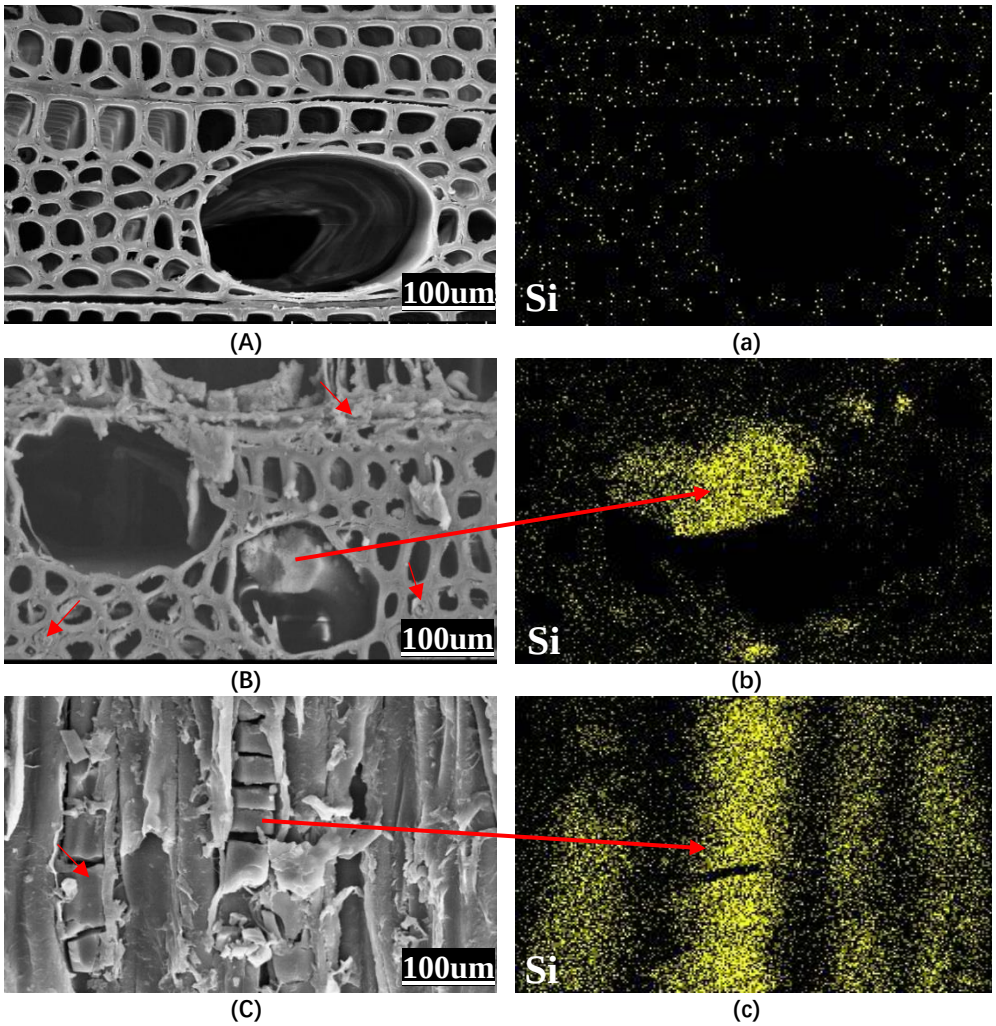

Figure5 SEM-EDX image of the modified wood (A, a-cross section and silicon energy spectrum diagram of the untreated wood; B, b-cross section and silicon energy spectrum diagram of $G_{20}S_{10}$ treated wood; C, c-longitudinal section and silicon energy spectrum of $G_{20}S_{10}$ treated wood)

4. Conclusions

In this study, glucose resin (MUG) was prepared using glucose as the main raw material, urea and melamine as cross-linking agents, and was catalyzed by inorganic acid and metal ions, and then compounded with Na_2SiO_3 solution to prepare a waterborne glucose-silicone resin composite modifier. By impregnating fast-growing poplar wood under vacuum pressure, the physical and mechanical properties of poplar wood are significantly improved. The research results are as follows:

(1) Infrared analysis found that the secondary amino structure was formed in the MUG resin, indicating the presence of a prepolymer structure in the resin; the MUG resin has good permeability, and the composite package and consolidation of Na_2SiO_3 by the MUG resin achieves the effect of the wood cell wall expansion and restriction, significantly reduces the shrinkage and moisture absorption of the Na_2SiO_3 modified wood, and greatly improves the dimensional stability of the modified wood.

(2) The mechanical properties, such as MOE, MOR, CS, of all modified wood are significantly improved compared to the untreated. Among the S_{20}, $G_{10}S_{20}$ and $G_{20}S_{10}$ modified wood, the $G_{10}S_{20}$ has the highest strength, and its MOE, MOR and CS increase by 72.2, 50.8 and 113.6% respectively compared with the control. Waterborne glucose silicone resin compound modifier has the advantages of strong inorganic silicon rigidity, high hardness and good permeability of MUG resin. It can fully penetrate into the wood tissue and exert the synergistic effect of organic-inorganic hybridization to significantly improve the mechanical properties of the treated material.

(3) SEM-EDX analysis shows that the waterborne glucose silicone resin can effectively enter the inherent pores of poplar wood cell walls, cell cavities and ducts; compared with the untreated, the

cell wall of the $G_{20}S_{10}$ treated wood is significantly thicker. It penetrates or adheres to the cell wall and undergoes polycondensation, cross-linking and solidification to form a wood-organic-inorganic consolidation and filling system. It is the physical filling and chemical cross-linking of the compound modifier that makes the mechanical properties and dimensional stability of the modified wood significantly improved.

The preparation of glucose resin compounded with inorganic sodium silicate to obtain an organic-inorganic hybrid compound modifier for wood impregnation is a green wood modification. Glucose as the main raw material is of renewable resources. The waterborne glucose silicone resin is an ecological and eco-friendly, low cost, compound wood modifier. It has broad application prospects and is worthy of in-depth research for early promotion and application.

Author Contributions: Conceptualization, W.L.; methodology, Q.L. and H.D.; software, Q.L.; validation, W.L.; formal analysis, Q.L.; data curation, H.D.; writing—original draft preparation, Q.L.; writing—review and editing, Q.L. and W.L.; supervision, W.L.; project administration, W.L. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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