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Potential of glycerol as a bio-based wood adhesive

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Abstract

The development of bio-based adhesives, to the extent possible without harmful chemicals derived from fossil resources, is very important for the sustainable use of wood-based materials. In this study, we investigated the possibility of glycerol as a bio-based adhesive by manufacturing wood-based moldings. The raw materials used were glycerol and wood powder of Japanese cedar (*Cryptomeria japonica*). The moldings were manufactured under hot-pressed at 160–220 °C and 4 MPa for 10 min. The glycerol content varied from 0 to 30 wt%. Color differences in the resulting molding increased with increasing glycerol content and hot-press temperature, suggesting that chemical changes occur during hot pressing in the presence of glycerol. The best bending properties of the moldings were obtained under manufacturing conditions of 15 wt% glycerol and 220 °C. In the water resistance test using boiling water, weight gain and thickness swelling of moldings were inhibited with increasing glycerol content. The moldings manufactured at hot-press temperatures above 200 °C developed water resistance. Ether linkages were detected by Fourier transform infrared spectroscopy and heteronuclear single quantum coherence nuclear magnetic resonance spectroscopy. Our results demonstrated that good adhesiveness is achieved by ether linkages resulting from the chemical reaction between glycerol and wood powder.

Keywords Glycerol, Bio-based adhesive, Wood-based molding, Wood powder

Introduction

Wood-based materials are considered part of the effort to global warming because they are composed of carbon–neutral lignocellulosic biomass such as wood. Currently, however, synthetic resin adhesives derived from fossil resources are essential when manufacturing wood-based materials in wood industry. In addition, the adhesives generally contain harmful chemical substances such as formaldehyde. Recently, bio-based adhesives using bio-substances derived from plants and animals have gradually attracted attention as an alternative to synthetic resin adhesives. Basically, bio-based adhesives are classified into saccharide-based, protein-based, aromatic-based, oil-based and others. Although various bio-based adhesives have been studied [1–4], utilization of some chemicals derived from fossil resources are generally required to obtain good adhesiveness.

We have been researching bio-based adhesives using citric acid [5–8] and sucrose [9–11], which eliminate to the extent possible the use harmful chemicals derived from fossil resources. They have demonstrated good adhesiveness with high water resistance under suitable adhesion conditions. However, compared to synthetic resin adhesives, many problems (e.g., high heating temperature, long heating time) remain to be solved for practical application. Therefore, further exploration of

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novel bio-substances with good adhesiveness for wood is needed to expand the possibility of bio-based adhesives.

In this study, we focused on glycerol, a simple triol compound. It is a colorless, non-toxic, and water-soluble compound, widely used as a sweetener in the food industry, and as a humectant in pharmaceutical formulations. Glycerol is generally obtained from plant and animal sources, with typical plant sources including soybeans or palm; it is also a known byproduct of biodiesel production [12]. Actually, it has been used and/or researched as an organic substrate to produce high-value chemicals [13, 14]. In the field of adhesive, it is being investigated as a polyol in bio-polyurethanes [15, 16]. There are also research on bio-based wood adhesives composed of citric acid and glycerol [17–19]. In wood, it has been investigated as a chemical for improving physical properties [20, 21]. Recently, it was tried to use as a solvent for liquefaction of lignocellulose materials [22, 23]. Thus, glycerol has been used as an adhesive raw material and a chemical for wood, but the adhesiveness of glycerol itself for wood has not been clarified. Therefore, the potential of glycerol itself as a bio-based wood adhesive was investigated by manufacturing wood-based moldings using wood powder.

Materials and methods

Preparation of wood-based molding

Wood powder from Japanese cedar (*Cryptomeria japonica*) of less than 150 µm and glycerol (Nacalai Tesque, Inc., Kyoto, Japan) were used as raw materials. Wood powder was first dried in an oven at 105 °C for 15 h. The moisture content of wood powder was almost 0%. Glycerol was mixed with distilled water in an aluminum cup. The concentration of the solution was adjusted to 16.7% (for example, 2 g of glycerol was added to 10 g of distilled water). A certain amount of dried wood powder was mixed with the glycerol aqueous solution, then the mixture was dried in an oven at 105 °C for 24 h to evaporate the water. The glycerol content varied from 0 to 30 wt%, as shown in Table 1. The dried mixture was ground to a fine powder with a mortar. A cylindrical metal mold with an inner diameter of 7 cm and a square metal mold with an inner dimension of 10 cm were used to prepare wood-based moldings for water resistance and bending test, respectively. The weight of the dried mixture powder for cylindrical and square molds was 10 g and 35 g, respectively. The powder was poured into the metal molding, which was then hot-pressed by machine (ANF-50, Shinto Metal Industries, Ltd., Osaka, Japan). The press temperature varied from 160 to 220 °C, and the press pressure and time were 4 MPa and 10 min, respectively. Details of the preparation conditions are summarized in Table 1. The obtained samples were conditioned in a desiccator at 20 °C and 60 ± 5% relative humidity for 1 week.

Table 1 Preparation of molding with glycerol

Glycerol content (wt%)	Hot-press temperature (°C)	Pressure (MPa)	Time (min)
0, 5, 10, 15, 20, 30	200	4	10
15	160, 180, 200, 220		

Color measurement

The difference in color of the specimens cut from a square molding made with different glycerol contents was measured using a color meter (CM-2600d, Konica Minolta Japan, Inc. Tokyo, Japan), according to the CIELAB color system. The sensor head was 8 mm in diameter, and measurements were performed under a D65 light source at an angle of 10 degrees. For each manufacture condition, five specimens were prepared and measured at the same location, and the mean and standard deviation were calculated. A molding prepared using wood powder alone and manufactured at 200 °C, 4 MPa, and 10 min was used as the reference value. CIELAB color parameters (L^*, a^*, b^*) were used to calculate the color change. The color difference (ΔE^*_{ab}) was calculated using the following formula,

$$\Delta E*_{\mathrm{ab}} = \left[(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2\right]^{1/2},$$

where ΔL^* is the lightness difference and Δa^* and Δb^* are the chroma differences based on the wood powder-only molding.

Bending test

An 80 × 10 × 3 to 4 mm specimen was cut from a square molding. The density was measured, and a three-point bending test was performed using a universal bending test machine (AGX-V, Shimadzu Corporation, Kyoto, Japan). In reference to the Japanese Industrial Standards (JIS) A1408 [24], the span was set at 60 mm, and the loading speed was 2 mm/min. The specific modulus of rupture (sMOR) and specific modulus of elasticity (sMOE) were calculated by dividing MOR and MOE by the density of each sample. Five specimens were tested for each condition and the mean and standard deviation were calculated.

Water resistance test

The discoid moldings underwent boiling treatment for 4 h. The moldings were subsequently dried in a vacuum oven at 60 °C for 15 h. The weight and thickness of the specimens were measured before and after each process.

Thickness was obtained by averaging the measurements of three points taken on the sample using a micrometer.

Fourier transform infrared spectroscopy (FT-IR) measurement

Moldings were crushed to powder by a mixer and then screened with 100 mesh sieves. The powder obtained was put into a vacuum dryer at 60 °C for 15 h. All infrared spectra of dried powder were obtained with a Fourier transform infrared spectrophotometer (FT/IR-4200, JASCO Corporation, Tokyo, Japan) using the KBr disk method, and recorded with an average of 32 scans at a resolution of 4 cm^{-1}.

Nuclear magnetic resonance (NMR) measurement

Sample preparation was carried out based on previous research [25, 26]. The moldings were first crushed to under 60 mesh with a mixer. The crushed molding (200 mg) was ball-milled with a Planetary Mono Mill PULVERISETTE 6 classic line (Fritsch GmbH, Idar-Oberstein, Germany), with vibration set at 600 rpm in zirconium dioxide (ZrO_2) vessels (12 ml) containing ZrO_2, 50 balls (5 mm × 5 mm). Grinding time was 120 min, in 10-min grinding/5-min intervals for 8 cycles to obtain ball-milled powder. A heteronuclear single quantum coherence (HSQC) experiment was applied to the ^1H-^{13}C correlation (Bruker standard pulse sequence "hsqcetgpsp.3"). The solvent was premixed with dimethyl sulfoxide (DMSO)-d_6/pyridine-d_5 (v/v,4/1), and the powder was swelled in the solvent. A Bruker Avance 800 MHz spectrometer (Ettlingen, Germany) was used at 50 °C. The central DMSO solvent peak was used as the internal standard (δH 2.49, δH 39.5 ppm). The spectral widths were 9615 and 40,243 Hz for the ^1H and ^{13}C dimensions, respectively. The number of collected complex points was 1922 for the ^1H dimension, with a recycle delay of 1.5 or 2.5 s. The number of transients was 16 or 32 and 256 time-increments were always recorded in the ^{13}C dimension. Gaussian multiplication ($LB = -2.00$) and sine-bell squared ($SSB = 2$) window functions were applied in the ^1H and ^{13}C dimensions, respectively. Prior to the FT, the data matrices were zero-filled up to 2048 points in both the ^1H and ^{13}C dimensions.

Results and discussion

Color change of the moldings

When wood powder was molded with glycerol, khaki-colored molding was obtained. Figure 1 shows the molding containing 15 wt% glycerol manufactured at 220 °C, 4 MPa, for 10 min. As glycerol is a colorless and transparent liquid triol compound with a high boiling point of 290 °C, the obtained moldings did not show hyperchromic color. Figure 2 reveals the effects of glycerol content (a) and hot-press temperature (b) on the color difference. In Fig. 2a, the value of ΔE^*_{ab} increased linearly with increasing glycerol content. The average value of 5 wt% content was 3.87, and that of 30 wt% was 18.67. The value of ΔE^*_{ab} also increased with increasing hot-press temperature, as shown in Fig. 2b. In this research, the reference value was based on the wood powder-only molding manufactured at 200 °C, 4 MPa, and 10 min. By adding 15 wt% glycerol content, color differences were observed even at hot-press temperatures below 200 °C. The increase in color difference with increasing glycerol content and hot-press temperature suggested that the wood undergoes some chemical changes during hot pressing in the presence of glycerol.

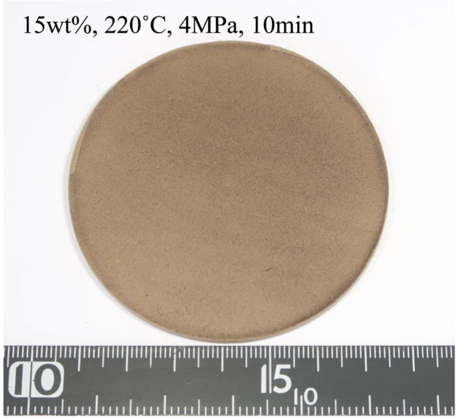

Fig. 1 Molding with glycerol

Bending properties of the moldings

Figure 3 shows the effect of glycerol content on specific bending properties. The density of the moldings with glycerol ranged from about 0.80–1.06 g/cm^3, as shown in Table 2. Density tended to increase as glycerol content increased. It is known that glycerol accelerates stress relaxation of wood at high temperature [27]. Therefore, it may be that the addition of glycerol promoted densification. The molding with wood powder only showed the worst bending properties, with average sMOR and sMOE values of 3.2 N·m/g and 0.8 MN·m/kg, respectively. The specific bending properties increased with increasing glycerol content. Maximum average values of sMOR and sMOE were obtained in molding with glycerol content of 15 wt%, and were 17.2 N·m/g and 2.4 MN·m/kg, respectively. This means that the addition of glycerol increased the sMOR and sMOE of the molding by up to approximately 5 and 3 times, respectively. The bending properties decreased with 20 and 30 wt% glycerol content. This was likely due to the decrease in the proportion of wood powder. The molding with glycerol content of 15 wt%

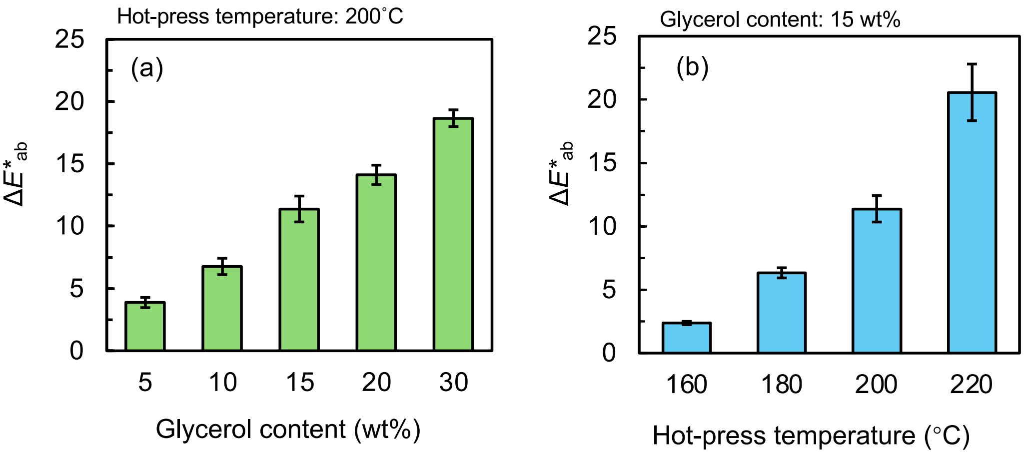

Fig. 2 Effects of glycerol content (**a**) and press temperature (**b**) on color difference of moldings. The diameter of molding: 7 cm

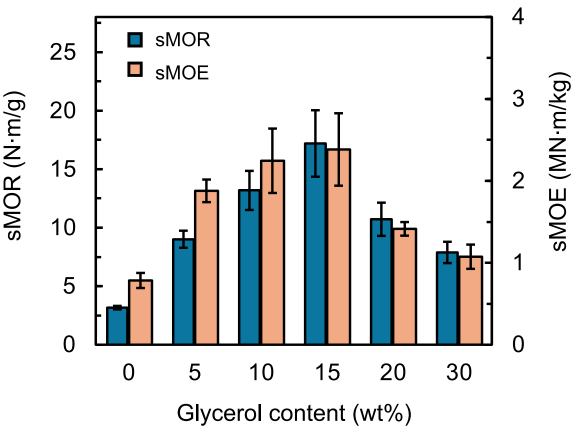

Fig. 3 Effect of glycerol content on the specific MOR and MOE of moldings

showed a significant improvement in bending properties compared to that with wood powder only (0 wt%), indicating that the glycerol may act as an adhesive.

Figure 4 presents the effect of hot-press temperature on specific bending properties. Based on the result in Fig. 3, the glycerol content was kept constant at 15 wt%. Specific bending properties increased with increasing hot-press temperature. Both sMOR and sMOE increased significantly between 180 and 200 °C, approximately tripling. This suggested that some reaction occurred at over 200 °C. The maximum values of sMOR and sMOE were obtained with the molding made at 220 °C, which were 20.8 N·m/g and 2.7 MN·m/kg, respectively. From these results, it was concluded that hot-press temperature has a significant effect on the bending properties of moldings. The values of MOR and MOE of Figs.3 and 4 are shown in supplementary information.

According to the results of Figs. 3 and 4, the best bending properties were obtained with manufacture conditions of 15 wt% glycerol content and hot-press temperature of 220 °C. In our previous research [28], molding with wood powder (Japanese cedar) and citric acid of 20 wt% was manufactured at 200 °C, 4 MPa, for 10 min. The values of sMOR and sMOE of the molding with 20 wt% citric acid were 38.1 N·m/g and 4.9 MN·m/kg, respectively. Compared to these results, the bending properties of the molding with glycerol were inferior to those with citric acid.

Water resistance of the moldings

To investigate the water resistance of the molding with glycerol, boiling treatment was performed. Figure 5 shows the effects of different glycerol content on changes in weight (a) and thickness (b) of the moldings manufactured at 200 °C. The moldings manufactured with 0 and 5 wt% glycerol contents collapsed in boiling

Table 2 Density of the molding with glycerol

Glycerol content (wt%)	0	5	10	15	20	30
Density (SD*) (g/cm3) (Press temperature: 200˚C)	0.80 (0.01)	0.89 (0.02)	0.93 (0.01)	1.09 (0.03)	1.04 (0.04)	1.06 (0.04)
Press temperature (˚C)	160	180	200	220		
Density (SD*) (g/cm3) (Glycerol content: 15wt%)	0.77 (0.04)	0.89 (0.03)	1.09 (0.03)	1.13 (0.06)		

SD Standard deviation

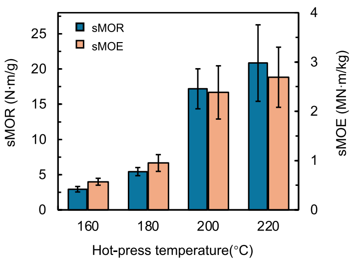

Fig. 4 Effect of hot-press temperature on the specific MOR and MOE of moldings

water, while the moldings manufactured with 10 to 30 wt% glycerol content maintained their shape. In terms of weight change (a), weight gain just after boiling treatment decreased with increasing glycerol content. This means that water absorption was inhibited by increasing glycerol content. The molding manufactured with 30 wt% glycerol had the minimum weight increase of 24.6%. Weight decrease was observed in drying treatment, indicating that some elution occurred during boiling treatment. In particular, weight loss increased with increasing glycerol content, suggesting that glycerol may have eluted. In terms of thickness change (b), the thickness swelling of the moldings just after boiling treatment also showed lower values with increasing glycerol content. One of the reasons seemed to be that the swelling of wood powder was inhibited by increasing glycerol content. On the other hand, shrinkage of the molding in drying treatment was significant with increasing glycerol content. Although each behavior of weight and thickness changes must be considered the proportion of wood powder and glycerol, at least the molding with glycerol had good water resistance against boiling water.

Figure 6 shows the effects of hot-press temperature on changes in weight (a) and thickness (b) of moldings with 15 wt% glycerol content. The moldings manufactured at 160 and 180 °C collapsed in boiling water and showed no water resistance; meanwhile, moldings manufactured at 200 and 220 °C maintained their shape. Regarding weight change (a), the weight gain of moldings at 220 °C just after boiling treatment was inhibited compared with the molding at 200 °C. The value of molding at 220 °C was 33.0%. Weight decrease in drying treatment showed similar value. For thickness change (b), the thickness of the molding at 220 °C just after boiling treatment was also lower than that of 200 °C. The value of molding at 220 °C was 20.7%. Based on the results obtained, molding having water resistance was obtained by the hot-press temperature of 200 °C and more. In addition, as glycerol content was same in here, it was clarified that the molding at 220 °C had high water resistance compared to that of 200 °C. In the previous molding with citric acid of 20 wt% manufactured at 200 °C, 4 MPa, for 10 min, the weight gain of the molding after boiling treatment for 4 h was

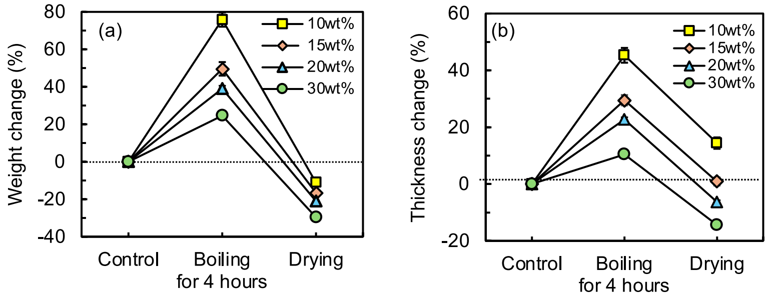

Fig. 5 Effect of glycerol content on boiling treatment of the moldings manufactured at 200 °C

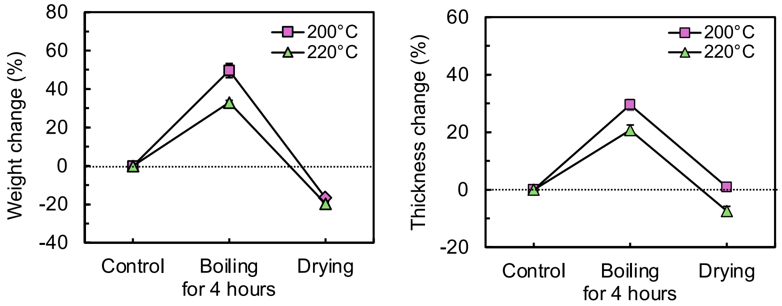

Fig.6 Effect of hot-press temperature on boiling treatment of the moldings with glycerol content of 15 wt%

more than 60% [28]. Therefore, the molding with glycerol may be slightly more water resistant.

Combined with the results of the bending properties and water resistance, it was suggested that glycerol acts as a water-resistant adhesive by heating at more than 200 °C, and the optimal manufacturing condition for glycerol–wood molding was 15 wt% and 220 °C in this research.

Bonding mechanism of glycerol

The bonding mechanism of glycerol for wood was investigated by FT-IR and 2D-NMR measurements. Figure 7 shows the infrared spectra of the moldings before and after the boiling treatment, and the wood powder as a reference. Considering the results of the bending test and boiling treatment, the moldings manufactured at 220 °C and glycerol content of 15 wt% were used. Overall, no significant peak changes were observed. One reason for this would be that the glycerol content in the molding was not substantial. However, a slightly peak increase at around 1100 and 1160 cm^{-1} was observed in the moldings before and after boiling treatment compared to that of wood powder. The absorption peaks of wood have been studied extensively, and the peaks at around 1100 and 1160 cm^{-1} are attributed to C–O stretching and C–O–C, respectively [29, 30]. The peak of 1100 cm^{-1} is also attributed to C–O of the secondary alcohol group in glycerol [31]. Since glycerol itself is water soluble, the remaining glycerol in the molding would be eluted during boiling treatment. Judging from the increase in the absorption peaks in the molding after boiling treatment, glycerol might be reacting with wood. According to the previous studies, it was known that polyol including glycerol reacts with cellulose and lignin by ether linkages under a process of biomass liquefaction [32]. In addition, it was reported that some interaction between glycerol and wood components occurs in glycerol-impregnated wood at high temperatures, such as 120 and 160 °C [27, 33]. However, the analysis using FT-IR has not been performed.

To clarify the detailed chemical changes of wood components during the adhesion process, the molding with 15 wt% glycerol content manufactured at 220˚C for 10 min was analyzed with 2D-NMR. Figure 8 shows 2D-NMR spectra of the molding and wood powder. In the spectrum of the wood powder, the signals for each wood components were assigned and colored. For example, glucose (Glc) residues, mannose (Man) residues, xylose (Xyl) residues, β-O-4 bonds, β-β bonds, β-5 bonds of lignin and G-type aromatic ring in lignin were colored in red, orange, dark-blue, cyan, purple, green, blue, respectively. When the glycerol backbone in the molding (GLY)'s spectrum are focused on, the etherified glycerol's signals (Gly1,3ether, green) were observed at 3.77/69.4 and 3.71/70.6 ppm, although the signals of the unreacted glycerol (Gly1,3 and Gly2, light-green) still remained. The result means the etherification of some primary hydroxyl (OH) groups in

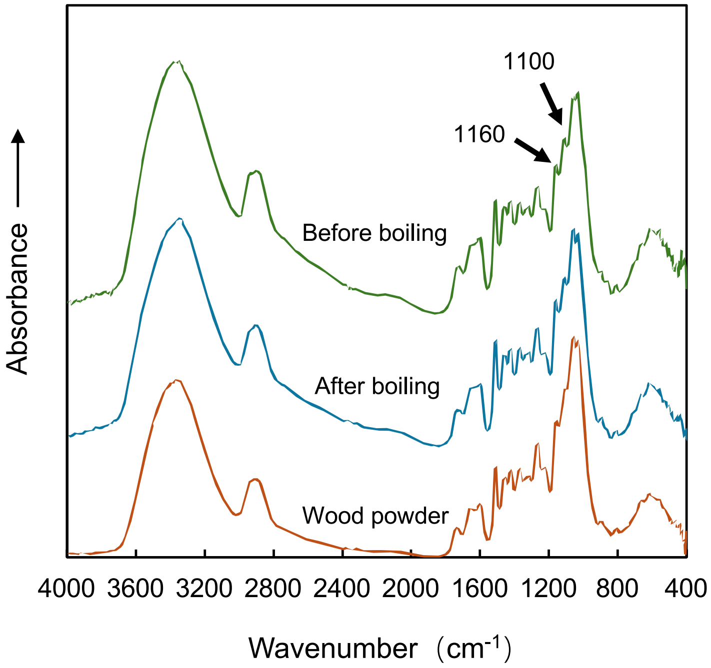

Fig.7 FT-IR spectra of moldings manufactured at 220 °C

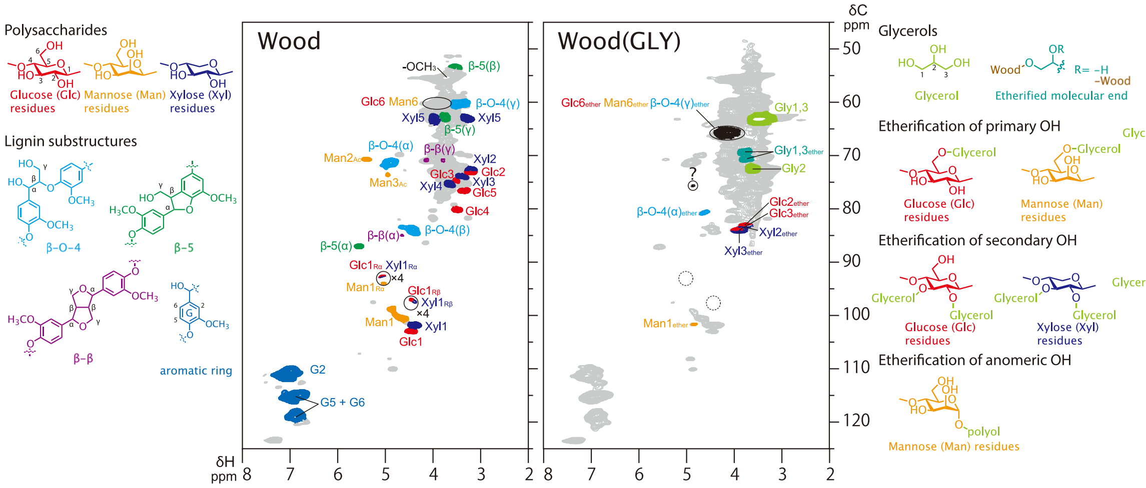

Fig. 8 2D-NMR spectra of wood and molding

glycerol. Next the substructures of wood components were focused on. As for polysaccharides, the etherified signals of H6/C6, H2/C2 and H3/C3 were observed at 4.11/65.7 ppm (black), 3.70/83.2 and 3.89/84.0 ppm (red and dark-blue). Thus, the primary OHs of cellulose and glucomannan, and the secondary OHs of cellulose and xylan were partly etherified by the glycerol. For lignin, the Hγ/Cγ [4.11/65.7 ppm (black)] and Hα/Cα [4.62/80.8 ppm (cyan)] signals of the etherified β-O-4 substructures appeared, revealing that some primary OHs at the γ position or benzylic OHs at the α position were etherified. Furthermore, the appearance of the Man's anomeric signal at 4.84/101.7 ppm (orange) also clarified the etherification of anomeric hydroxyl groups of glucomannan. On the other hand, the reducing end signals of polysaccharides disappeared in the spectrum of the molding, speculating that the reducing end may have been etherified by glycerol. Based on the results obtained in FT-IR and 2D-NMR, estimated chemical reactions between wood powder and glycerol are shown in Fig. 9. Basically, the primary OH of glycerol reacts with wood components by ether linkages. As for the reaction with detailed wood components, primary and secondary OH of polysaccharide residues and lignin react with glycerol. In addition, reactions with anomeric OH occur. It was clarified that glycerol acts as an adhesive for wood powder through chemical bonding.

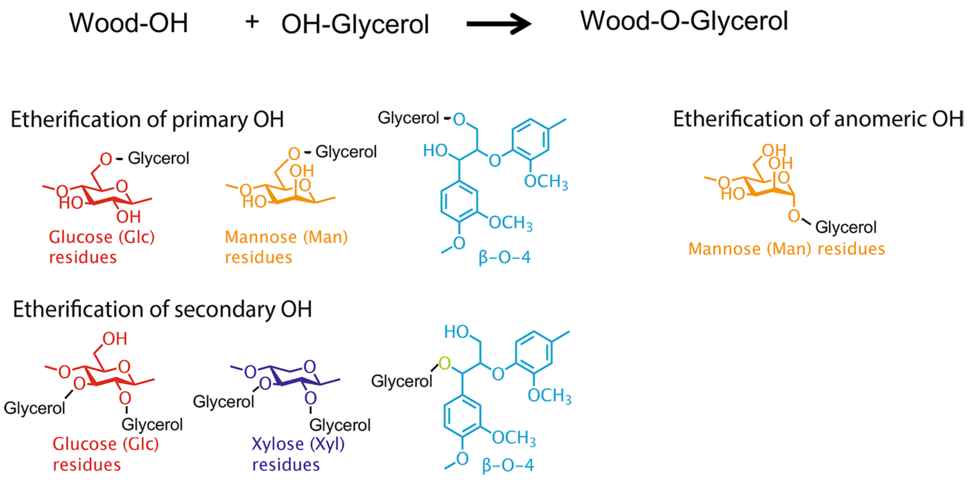

Fig. 9 Estimated reactions between wood and glycerol

Conclusions

The adhesiveness of glycerol was investigated using wood-based molding manufactured from wood powder and glycerol. The color of the wood-based molding increased with increasing glycerol content and hot-press temperature. However, the obtained moldings did not exhibit black color. As the result of investigating the effects of glycerol content and hot-pressing temperature on specific bending properties of the wood-based molding, 15 wt% and 220 °C were the best condition, respectively. The water resistance test by boiling treatment showed that the weight gain and thickness swelling of molding were inhibited with increasing glycerol content. In addition, moldings manufactured at hot-press temperature more than 200 °C maintained their shape, suggesting that glycerol acts as a water resistant adhesive by heating at more than 200 °C. The investigation of adhesion mechanism by FT-IR and 2D-NMR revealed that adhesion is caused by chemical bonding through ether linkages between glycerol and wood components. Consequently, there is a possibility that glycerol itself could be used as a promising bio-based wood adhesive.

Abbreviations

sMOR	Specific modulus of rupture
sMOE	Specific modulus of elasticity
FT-IR	Fourier transform infrared spectroscopy
NMR	Nuclear magnetic resonance
DEMSO	Dimethyl sulfoxide
SD	Standard deviation
Glc	Glucose
Man	Mannose
Xyl	Xylose
Gly	Glycerol
OH	Hydroxyl

Supplementary Information

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Supplementary Material 1.

Supplementary Material 2.

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Author contributions

LJ performed the experiments, analyzed data and wrote the initial draft of the manuscript. UT assisted LJ's experiments. MM and CS contributed to interpretation of data. AD and SS contributed to sample preparation and interpretation for NMR measurement. UK (corresponding author) designed this study and was a major contributor in writing the final manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and analyzed in the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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