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Analysis of Deformation Fixation of Thermally Compressed Scots Pine (*Pinus sylvestris* L.)

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Abstract: Heat treatment effectively inhibits the water absorption recovery of compressed wood. To elucidate this phenomenon, we prepared compressed pine and thermally compressed pine (heartwood and sapwood) using the hot pressing method at 160 °C, 180 °C, 200 °C, and 220 °C. The effects of chemical components, swelling stresses, and monosaccharides on modified wood recovery were investigated using regression analyses. Notably, the recovery of both compressed heartwood and sapwood during water absorption declined from 18.89% to 2.66% and from 58.40% to 1.60%, respectively, after heat treatment. Similarly, the swelling stresses of the compressed heartwood and sapwood at 220 °C, respectively, ranged from 0.693 MPa to 0.275 MPa and from 0.783 MPa to 0.330 MPa. These were close to the values of untreated heartwood (0.175 MPa) and sapwood (0.225 MPa). Regression functions indicated that the recovery of compressed wood is chemically dependent on hemicellulose and mechanically related to swelling stress. For monosaccharides, regression functions indicated that modified heartwood recovery primarily relied on mannose, whereas modified sapwood recovery was remarkably affected by mannose and xylose. This confirmed that the pyrolytic monosaccharides in hemicellulose promoted stress relaxation, which induced the deformation fixation of thermally compressed wood.

Keywords: thermally compressed wood; swelling stress; recovery; hemicellulose; monosaccharides; water absorption

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1. Introduction

Transverse compression is used to enhance the density and reduce the porosity of lightweight wood [1,2]. This is a straightforward and environment-friendly process that improves the mechanical properties of wood [3,4]. Compressed wood modification techniques include shaping and layered compression. Shaping compression is typically used for the overall compression of large wooden boards, such as logs and lumber [5,6], while layered compression involves the densification treatment of specific layers of boards based on application requirements [7–9].

However, a common issue with compressed wood is its tendency to undergo significant deformation recovery in moist environments, which limits its practical utility [10]. Considerably, pretreatment and posttreatment procedures are required to prevent the deformation of compressed wood. Common pretreatment methods include resin impregnation and cross-linking treatment. For example, Yan et al. (2011) investigated the effect of glycerin pretreatment on compressed wood [11]. They found glycerin could accelerate stress

relaxation in compressed wood, thereby playing an important role in deformation fixation. Regarding posttreatment, researchers have discovered that subjecting compressed wood to prolonged high-temperature treatment can effectively remedy water absorption recovery [12–14]. For instance, Lee et al. (2018) demonstrated that after 24 h of heat treatment at temperatures of 120, 140, and 160 °C, the water absorption recovery of compressed wood was <1% [15]. This was attributed to the reversible nature of elastic deformation compared to the irreversible plastic deformation of wood [16]. Therefore, plastic deformation is the primary cause of the permanent deformation of wood [17].

Heat treatment, as a green posttreatment method, has been widely studied. To elucidate the deformation fixation phenomenon, research has been conducted on the recovery and modification mechanisms of compressed wood post-heat treatment. Some studies have suggested that during hot pressing, elastic deformation occurs primarily in the microfiber crystalline regions [18], while plastic deformation takes place under high pressure [8]. The release of elastic strain energy stored in cellulose macromolecules contributes to recovery [19,20]. In addition, amorphous hemicellulose, lignin, and semicrystalline cellulose in wood create internal stresses—known as residual stresses—after compression [21]. Compression forces the wood structure to transition into a new form without breaking the covalent bonds between hemicellulose and lignin or the hydrogen bonds between hemicellulose and cellulose [20]; this induces the temporary and reversible deformation of wood under heat and moisture conditions. Because the primary components of the compressed wood do not undergo significant chemical changes, the recovery is predominantly influenced by hydrophilicity and stress accumulated in the macromolecular chains of the wood. However, this stress is typically measured using a stress–strain gauge [22,23], which can analyze stress relaxation and stress–strain relationships in dried wood. During water absorption, it cannot accurately capture the stress-release regularity of modified wood.

During heat treatment, the hydrophilic hemicellulose in compressed wood undergoes intense degradation, leading to a relative increase in hydrophobic lignin. Moreover, the post-heat treatment increase in cellulose crystallinity decreases the hydrophilic hydroxyl groups, further reducing the equilibrium moisture content and water absorption of the compressed wood. The degraded hemicellulose further facilitates the breaking of covalent and hydrogen bonds in the wood, leading to new cross-linked network structures. Thus, the stresses in compressed wood are relaxed, aiding in deformation fixation [13]. We previously examined the chemical composition, water absorption, and hygroscopicity of thermally compressed wood and highlighted the substantial role of hemicellulose degradation in reducing water absorption [20]. Song et al. (2018) confirmed that removing hemicellulose from wood effectively inhibited moisture-induced deformation recovery in compressed wood and emphasized the significant influence of hydrophilic hemicellulose on water absorption recovery in compressed wood [24]. Hemicellulose is the second most abundant plant polysaccharide, comprising 20%–35% of wood mass. It has a lower molecular weight and a degree of polymerization typically between 80 and 200, and is composed of β-xylose, β-mannose, α-arabinose, D-glucose, D-galactose, and D-glucuronic acid units [25]. In softwood, mannans and a small amount of xylans are the main components of hemicellulose, while xylans are predominant in hardwood hemicellulose. These oligosaccharides contain abundant hydroxyl and acetyl groups, which have a strong water absorption capacity. It can be seen that the oligosaccharides in hemicellulose will have a significant effect on the water absorption of compressed wood [26]. Although some studies have investigated the recovery properties of thermally compressed wood [27,28], the underlying impact of oligosaccharides on the recovery of compressed wood has not been thoroughly studied yet. Therefore, this study focuses on elucidating the precise relationship between the oligosaccharides of hemicellulose and the recovery properties of compressed wood.

Based on the above analysis, to explain the fundamental reasons why heat treatment promotes fixing deformation of compressed wood (CW), this article discussed the effects of chemical components, swelling stresses, and monosaccharides on the recovery of com-

pressed wood using regression analysis. It could potentially improve processing techniques and enhance the performance of wood-based materials.

2. Materials and Methods

2.1. Wood Materials

Heartwood and sapwood specimens (30 [radial] × 150 [tangential] × 450 [longitudinal] mm) were obtained from *Pinus sylvestris* L. pine. Their oven-dry densities, respectively, were 0.4188 ± 0.0077 and 0.4669 ± 0.0091 g/cm^3. To ensure that plastic deformation occurred during hot pressing, all samples had an initial moisture content ranging from approximately 30%–40%. In the meantime, the moisture contents of specimens were controlled in an air conditioning chamber with a constant temperature of 23 ± 2 °C and a humidity of 95 ± 3% for a month-long exposure. Subsequently, wood was utilized for subsequent modification procedures. A subset of both heartwood and sapwood samples was reserved for the control tests.

2.2. Preparation of Thermally Compressed Wood

The heartwood and sapwood samples underwent radial compression using a hot pressing technique at 160 °C for a duration of 1 h to achieve a controlled thickness target value of 18 mm [29].

To produce thermally compressed wood, compressed heartwood and sapwood samples were subjected to continuous treatment in the press for 3 h at varying temperatures (see Table 1), followed by cooling and subsequent unloading via shutting down the heating and pressure system.

Table 1. Hot-pressing conditions of heartwood and sapwood samples.

Specimen Groups		Duration (h)	Temperature (°C)
Heartwood	1 (Control)		
	2 (CW)	1	160
	3	3	180
	4	3	200
	5	3	220
Sapwood	6 (Control)		
	7 (CW)	1	160
	8	3	180
	9	3	200
	10	3	220

2.3. Recovery Experiment of Treated Wood

Fifteen wood block samples (20 [longitudinal] × 20 [tangential] × 18 [radial] mm) were cut from treated wood. The initial dry thickness of wood blocks was determined using a vernier caliper. Subsequently, the specimens were immersed in distilled water at ambient temperature for a period of 48 h, and the water absorption thickness was measured every 24 h. Following this, the samples were dried in an oven at 100 °C for 24 h, and the dry thickness was recorded. Thus, the water absorption experiment repeated this soaking and drying cycle five times. Finally, the recovery rate of the samples was computed using the following equation:

$$R = \frac{L2 - L0}{L0 - L1} \times 100\%$$

where R is the recovery rate along the compressed thickness direction (%), $L0$ is the dried thickness of sample before compression (mm), $L1$ is the dried thickness of sample after compression (mm), and $L2$ is the thickness of sample after water absorption for 24 h (mm).

2.4. Swelling Stress of Samples during Water Absorption

Two groups of samples (20 [longitudinal] × 20 [tangential] × 18 [radial] mm and 20 [longitudinal] × 20 [tangential] × 30 [radial] mm) were cut from the treated and untreated wood samples. During water absorption, the fluctuation in swelling stress of samples in each group was monitored using a pressure sensor (Figure 1). A data acquisition system was integrated into a computer mainframe and configured with a sampling frequency of 100 ms. The pressure sensor was preconnected to the computer, and the samples were securely affixed to the clamp apparatus, which was in contact with the pressure sensor (Figure 1). The initial displayed value on the instrument panel of the sensor was adjusted to 0 N. Subsequently, the sample and pressure sensor assembly on the clamp apparatus were submerged in distilled water. The data collected by the sensor were transmitted to the relevant data acquisition system on the computer through a signal converter. Variations in swelling force over time were displayed on the data acquisition interface. The data collection concluded when the swelling force curve approached a plateau, where the experiment was terminated and the data were exported. Then, swelling stress was calculated according to the formula: stress = force/area. Each group of experiments was repeated at least six times, and the final result was determined as the average value.

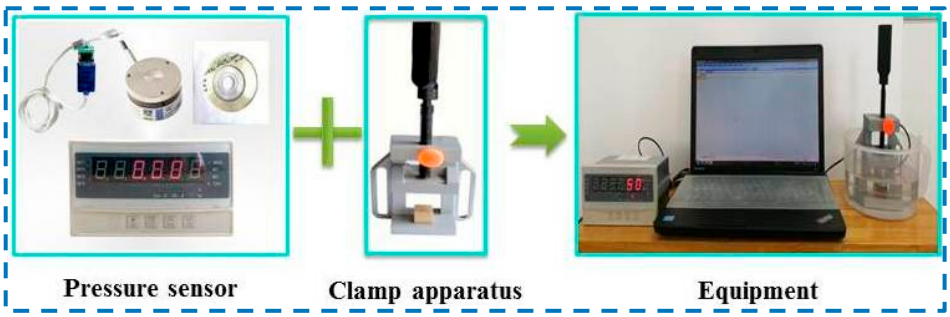

Figure 1. Equipment used to determine the swelling stress of samples during water absorption testing.

2.5. Analyses of Chemical Components in Treated Wood

Samples of the untreated wood, compressed wood, and thermally compressed wood were processed into small 3 cm segments and ground into 40–60 mesh powders using a small crusher. Next, the cellulose, hemicellulose, lignin, and extractive contents of each sample group were analyzed according to our previous study [29].

2.6. Measurement of Monosaccharide Content in Wood

After benzene alcohol extraction, the monosaccharide contents of wood powders were assessed with reference to the experimental analytical method provided by the US National Renewable Energy Laboratory [30]. Approximately 0.3 g of the wood powder was placed in a hydrolysis bottle, to which 3 mL of 72% H_2SO_4 was added. The mixture was hydrolyzed in a water bath at 30 °C for 1 h and shaken every 10 min. Subsequently, 84 mL of deionized water was introduced into the hydrolysis bottle to dilute the concentrated sulfuric acid to 4%. The bottle was securely capped and thoroughly shaken. The hydrolysate was transferred into an autoclave and processed at 121 °C for 1 h before being cooled. A 1.5 mL portion of the resulting hydrolysate was filtered through a 0.22 μm aqueous filter head and diluted 300 times. Through the above steps, lignin can be well precipitated and removed, thus avoiding interference with the test of monosaccharide contents. The monosaccharide contents of each sample were determined using a high-performance anion-exchange chromatograph (Dionex ICS-5000+; Thermo Fisher Scientific Inc., Waltham, MA, USA) equipped with a Carbopac^{TM} PA20 column, a pulsed amperometric detector, and an AS50 injector. D-glucose, d-xylose, and d-mannose were used as monosaccharide standards. All samples were tested five times, and the results were averaged.

3. Results and Discussion

3.1. Effect of Chemical Components on the Recovery of Compressed Wood

The recovery rates for heartwood and sapwood after soaking in water are illustrated in Figure 2a,b. From the first to the fifth water absorption cycle, the recovery rates of compressed heartwood and sapwood were between 12.79% and 18.89% and 42.59% and 58.40%, respectively. The recovery patterns shown in Figure 2a,b indicate that the swelling deformation of compressed heartwood and sapwood primarily increased during the first three water absorption cycles. Notably, compressed wood in the first water absorption cycle exhibited the most significant change.

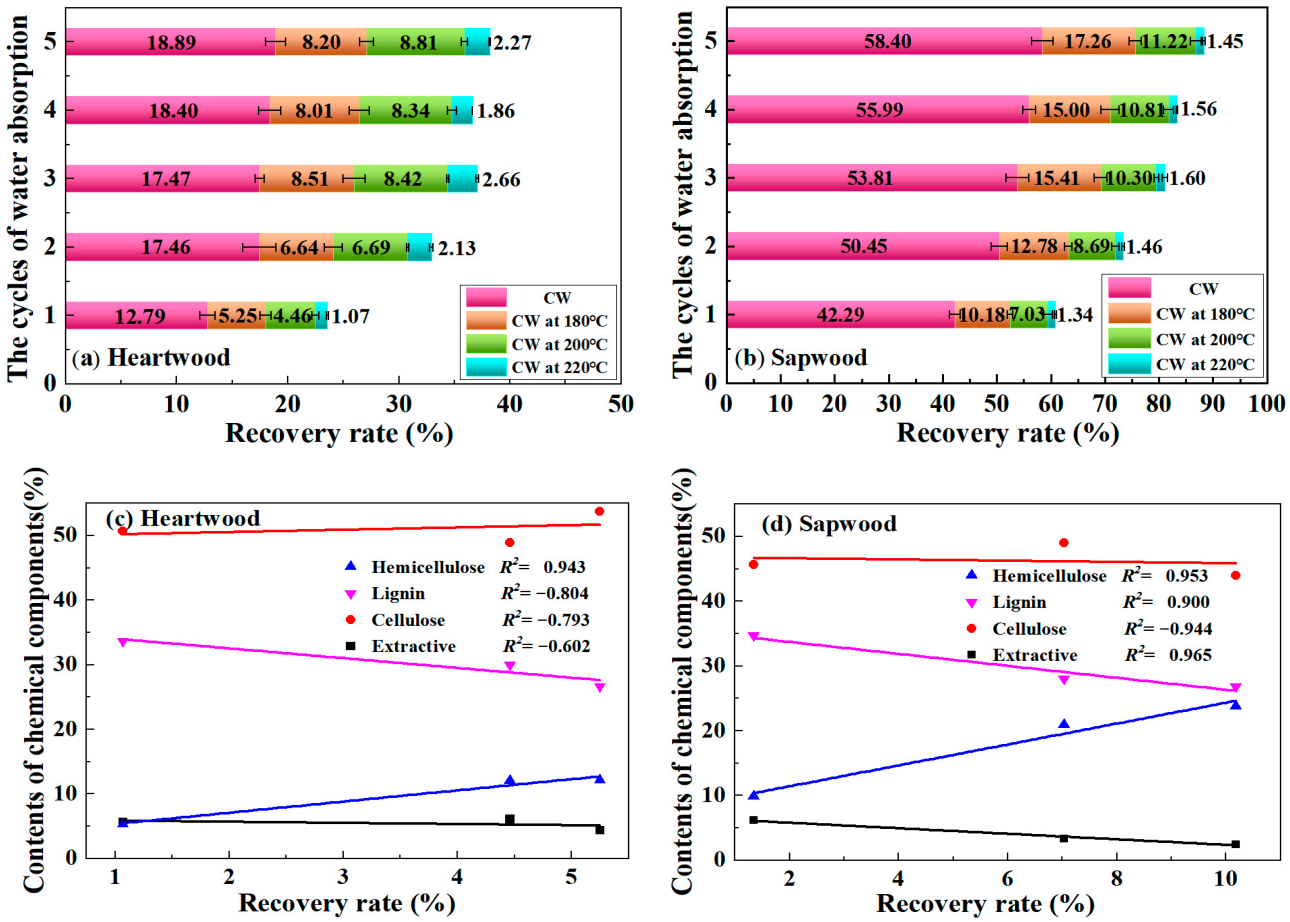

Figure 2. Recovery Analysis of modified heartwood and sapwood: (**a**,**b**) recovery rates, (**c**,**d**) relationships between recovery rates and chemical components.

After heat treatment, the swelling recovery of compressed heartwood and sapwood noticeably decreased. As the number of cycles increased, the deformation of each group increased. The recovery rates of compressed heartwood subjected to 180 °C, 200 °C, and 220 °C ranged from 5.25% to 8.51%, 4.46% to 8.81%, and 1.07% to 2.66%, respectively, over the five water absorption cycles. Similarly, the corresponding values of compressed sapwood at 180 °C, 200 °C, and 220 °C varied from 10.18% to 17.26%, 7.03% to 11.22%, and 1.34% to 1.60%, respectively, implying that the higher the temperature of heat treatment, the smaller the deformation of compressed wood. The recovery rates of the thermally compressed heartwood sample group were lower than those of the corresponding sapwood group, except for the 220 °C heat-treatment condition. This difference in recovery rate could be related to the variable chemical composition and viscoelasticity of wood cell walls. Moreover, heat treatment had a more significant impact on the recovery of sapwood than that of heartwood.

To analyze this difference in detail, Origin 8.0 software was used to establish the regression function for each chemical composition and recovery rate. The results indicate that there are good linear relationships between the recovery rates and compositions of modified heartwood and sapwood (Figure 2c,d). Positive correlations were observed between recovery and hemicellulose for heartwood and sapwood, with R^2 coefficients reaching approximately 0.950. Conversely, negative correlations were between the recovery rates and lignin, with R^2 values of 0.804 and 0.900 for heartwood and sapwood, respectively. Notably, no correlation was identified between the recovery and cellulose in modified wood.

Shao (2022) studied the effect of different lignin/hemicellulose removal times on the water absorption recovery of compressed wood and found cellulose enhanced the shape fixation of compressed wood [31]. This conclusion indicates that the increased cellulose content can reduce the shape recovery of wood [32]. Furthermore, the regression analysis in the current study highlighted the significant impact of extractive content on recovery in sapwood compared to heartwood. However, because the change in cellulose content was not obvious, it was concluded that the degraded hemicellulose and relatively increased lignin content enhanced the deformation fixation of compressed wood. Therefore, the lower recovery of thermally compressed heartwood compared to sapwood is attributed to the lower content of hemicellulose in heartwood.

3.2. Effect of Swelling Stress on the Recovery of Compressed Wood during Water Absorption

As shown in Figure 3a,b, with prolonged water absorption, the swelling stress of the heartwood and sapwood groups increased and stabilized. The maximum swelling stress of untreated heartwood and sapwood, respectively, were approximately 0.175 Mpa and 0.225 Mpa. After compression, these values increased to 0.693 Mpa and 0.783 Mpa for heartwood and sapwood, respectively, representing a substantial rise of 296.26% and 247.92% compared to the untreated samples. Blomberg and Persson (2007) observed that the increase in volume of densified wood during water absorption was attributed to the expansion of native cell walls and the shape recovery of deformed cell walls [33]. Thus, the total residual stresses include residual stress from the recovery of compressed cell walls and residual stress from the swelling of native cell walls [34]. The residual stress of the recovery of deformed cell walls was identified as a significant factor in the swelling behavior of densified wood. This thoroughly explains the significant increase in swelling stress of compressed wood observed in this study.

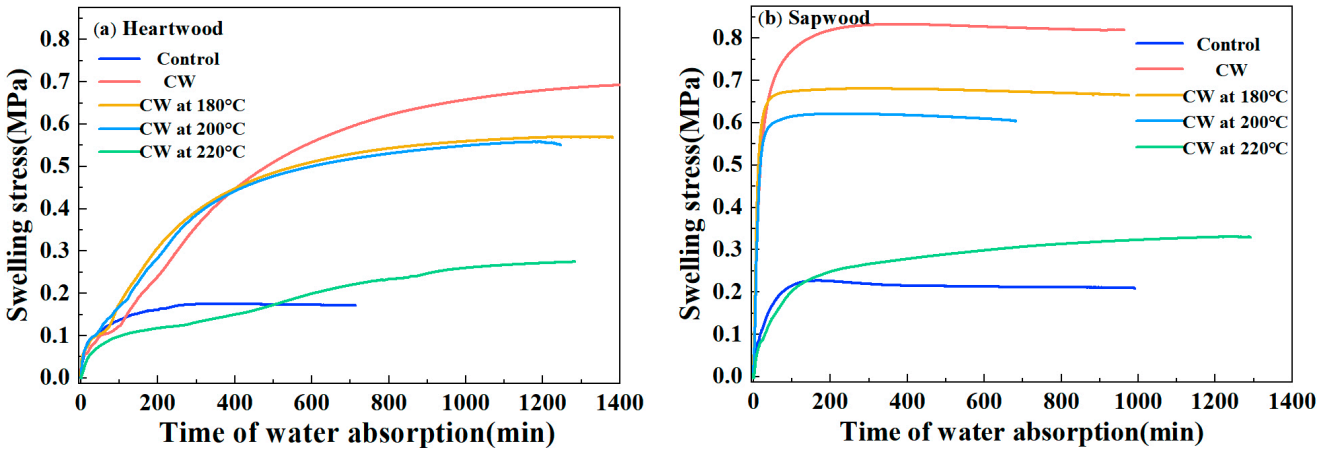

Figure 3. Changes in the swelling stress of (**a**) heartwood and (**b**) sapwood samples during water absorption.

After heat treatment, the swelling stresses of both compressed heartwood and sapwood evidently dropped. The maximum swelling stresses of compressed heartwood treated at 180 °C, 200 °C, and 220 °C, respectively, reached 0.570 Mpa, 0.555 Mpa, and 0.275 Mpa, representing decreases of 17.80%, 19.96%, and 60.34% compared with the compressed wood. Similarly, compared with compressed sapwood, the swelling stresses of thermally compressed sapwood samples exposed to 180 °C, 200 °C, and 220 °C separately became 0.680 Mpa, 0.620 Mpa, and 0.330 Mpa, representing reductions of 13.13%, 20.78%, and 57.84%. The degradation of chemical components in wood due to high-temperature treatment resulted in the release of residual stress. The reduced residual stress further promoted a significant decrease in swelling stresses of thermally compressed wood during water absorption. Thus, the deformation fixation of compressed wood was effectively improved by heat treatment. In addition, the results of the sapwood group were apparently larger than the values of the heartwood group, potentially because the sapwood had greater elasticity and was therefore more susceptible to elastic deformation than the heartwood. Consequently, a greater residue stress was formed inside sapwood.

The slopes of the curves presented in Figure 3a,b demonstrate that the changes in the rates of swelling stress during water absorption for heartwood groups were lower than those for sapwood groups. It took approximately 1000 min for the heartwood groups to reach the maximum swelling stresses, whereas the sapwood groups only required 200 min. This noticeable distinction is in close proximity to the natural structural characteristics of heartwood and sapwood. Heartwood is composed of mature dead cells, while sapwood is made up of living cells capable of metabolism and growth [35]. Compared with sapwood, heartwood has relatively few pits, and most of them are aspirated pits. In addition, extractives originating from heartwood aggravated pore blocking, resulting in reduced penetration. This implied that heartwood naturally has a lower water absorption rate than sapwood [36,37]. Furthermore, sapwood has a greater hemicellulose content than heartwood, which causes the hydrophilicity and water absorption rate of sapwood to be greater than those of heartwood. Therefore, these differences between heartwood and sapwood were responsible for their differing swelling stress rates.

To understand the recovery mechanism of modified wood, regression analyses between swelling stress and each chemical component and recovery in heartwood and sapwood were investigated (Figure 4). The results indicate that the swelling stresses in heartwood and sapwood were linearly positively correlated with hemicellulose content, with coefficients of determination (R^2) of 0.856 and 0.968, respectively. Similarly, the swelling stresses in heartwood and sapwood were negatively linearly correlated with lignin content, with R^2 values of 0.823 and 0.956, respectively. There was an exponential positive correlation between the swelling stress and recovery rate for heartwood and sapwood, with a fitting degree of 0.998. During the densification process, elastic deformation is the primary cause of the water absorption recovery of wood [16].

Previous research has investigated the impact of chemical composition on the elasticity of wood during heat treatment and found the elasticity of wood is positively correlated with hemicellulose content [38] and negatively correlated with lignin and cellulose content [39]. It further confirmed that the swelling stress of compressed wood is directly connected to the elastic residual stress stored in hemicellulose, similar to the results of the correlation analysis between recovery and hemicellulose in compressed samples in the current study. Therefore, it can be inferred that the recovery behavior of compressed wood after water absorption is principally dependent on hemicellulose and swelling stresses caused by the residual stress. After heat treatment, the residual stress of compressed wood declined due to the changing hemicellulose content. Correspondingly, the recovery was restrained, thereby realizing the desired fixed deformation of compressed wood.

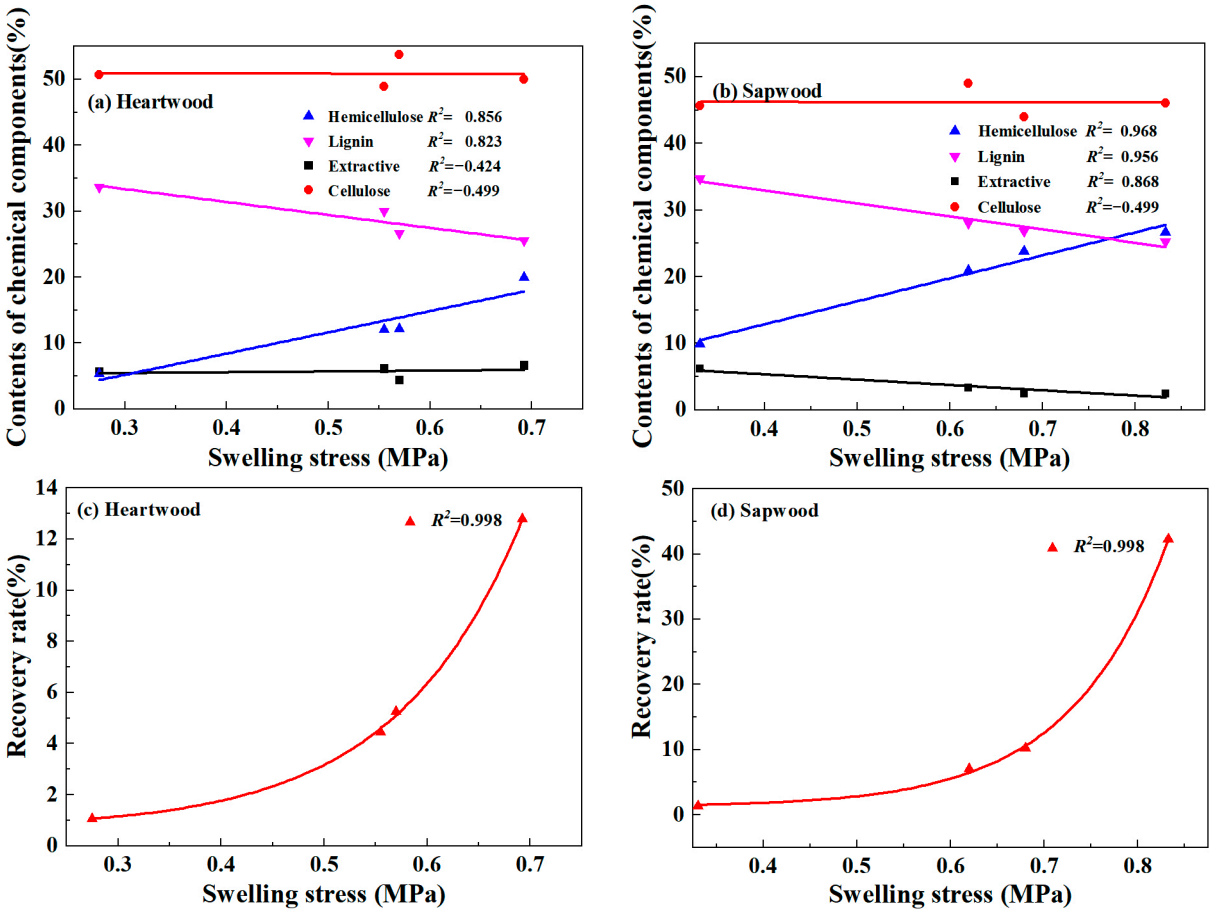

Figure 4. Relationships between swelling stress and chemical components in heartwood and sapwood as well as recovery rate.

3.3. Effect of Monosaccharide Changes on the Recovery of Compressed Wood

Based on the conclusions from Section 3.2, this section mainly analyzed the impact of monosaccharides on the recovery of compressed wood. The concentration changes of glucose, xylose, and mannose in the control and modified samples from heartwood and sapwood were investigated as the contents of galactose, arabinose, and uronic acids are minor [40]. As shown in Table 2, glucose was the most abundant compound in both untreated heartwood and sapwood, with concentrations of 4.067 and 3.189 mg/L, respectively, followed by mannose (0.872 and 0.693 mg/L) and xylose (0.297 and 0.231 mg/L).

Table 2. The determined monosaccharide contents in heartwood and sapwood.

Heartwood	Glucose (mg/L)	Xylose (mg/L)	Mannose (mg/L)	Sapwood	Glucose (mg/L)	Xylose (mg/L)	Mannose (mg/L)
Control	4.067 ± 0.050	0.297 ± 0.008	0.872 ± 0.022	Control	3.189 ± 0.041	0.231 ± 0.011	0.693 ± 0.015
CW	3.030 ± 0.036	0.256 ± 0.011	0.652 ± 0.017	CW	3.213 ± 0.033	0.135 ± 0.007	0.718 ± 0.009
180 °C	3.351 ± 0.023	0.155 ± 0.007	0.742 ± 0.007	180 °C	3.601 ± 0.019	0.275 ± 0.012	0.702 ± 0.013
200 °C	3.625 ± 0.018	0.086 ± 0.004	0.641 ± 0.011	200 °C	3.066 ± 0.030	0.209 ± 0.009	0.616 ± 0.024
220 °C	3.683 ± 0.017	0.036 ± 0.005	0.440 ± 0.013	220 °C	3.693 ± 0.011	0.018 ± 0.002	0.481 ± 0.008

Similarly, the mannose content decreased by 49.50% and 30.58%, respectively.

With increasing heat-treatment temperature, the measured glucose content in heartwood and sapwood roughly increased, whereas that of mannose and xylose exhibited an inverse change. It is deduced that hemicellulose in wood is mainly composed of large amounts of mannose and small amounts of xylose, which is consistent with previous research [26]. After heat treatment at 220 °C, the amounts of xylose in compressed heartwood

and sapwood, respectively, declined by 87.86% and 92.04% compared with their respective controls. Likewise, the mannose content decreased by 49.50% and 30.58%, respectively. Notably, the reduction in xylose content surpassed that of mannose, potentially resulting from the low level of xylose. High temperatures aggravate the decomposition of mannose and xylose, suggesting that polysaccharide polymers in hemicellulose are susceptible to decomposition under severe heat treatment. The concentration of the three monosaccharides in each heartwood group appeared higher than that in the corresponding sapwood groups, demonstrating that the accumulation of these monosaccharides is closely associated with the thickening of wood cell walls—a transformation that takes place as a tree progresses from sapwood to heartwood as it matures.

Regression analysis was used to analyze the relationships between the three monosaccharides, recovery rate, and swelling stress of modified wood (Figure 5). The recovery rate in heartwood was linearly positively related to the mannose and xylose contents, with R^2 coefficients of 0.953 and 0.652, respectively (Figure 5a,b). The same correlations were observed for the sapwood, with R^2 values of 0.997 and 0.976, respectively. In the case of glucose, a good correlation (R^2 = 0.825) was obtained with the recovery rate of sapwood but not for the heartwood. Similarly, there existed strong positive correlations between mannose content and swelling stress in heartwood and sapwood with R^2 values of 0.838 and 0.941, respectively (Figure 5c,d). Under the same conditions, weak correlations (R^2 = 0.429 and R^2 = 0.625) were observed between xylose content and swelling stress. However, no correlations were found between glucose and swelling stress in either heartwood or sapwood. Hence, the recovery of heartwood is mainly dependent on mannose composition, whereas mannose and xylose significantly affect the recovery of sapwood. The swelling stresses of heartwood and sapwood were primarily influenced by mannose, followed by xylose, which is potentially attributed to the high content of mannose, which contains more hydrophilic functional groups relative to xylose.

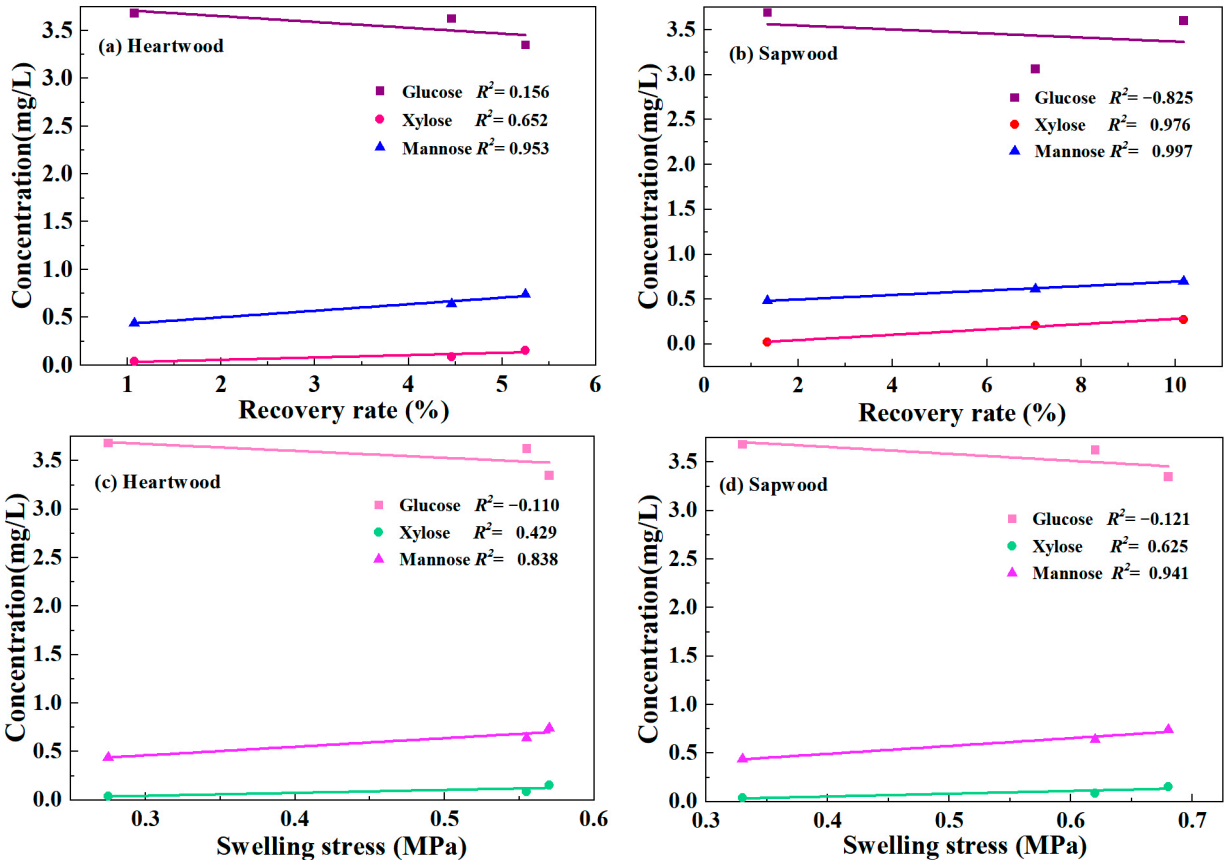

Figure 5. Relationships between monosaccharide contents and recovery rates and swelling stress of wood.

Although the recovery rate and swelling stress in heartwood and sapwood were not correlated with their glucose contents, higher concentrations of glucose reduced the recovery and swelling stress of modified wood (Figure 5). This is consistent with the previous conclusion that cellulose can enhance the shape fixation ability of compressed wood [41].

4. Conclusions

(1) With increasing numbers of soaking cycles, the deformation of compressed sapwood and heartwood increased. After high-temperature treatment, the recovery rates of compressed heartwood and sapwood decreased from 18.89% to 2.66% and from 58.40% to 1.60%, respectively. Regression analysis for each chemical composition and recovery rate indicated that the degradation of hemicellulose reduced the recovery of thermally compressed wood.

(2) The swelling stresses of compressed heartwood and sapwood were 0.693 and 0.783 MPa, which decreased by 60.34% and 57.84%, respectively, after heat treatment at 220 °C. The swelling stresses were linearly positively correlated with hemicellulose content, linearly negatively correlated with lignin content, and exponentially positively correlated with the recovery rate, indicating that the recovery of compressed wood is chemically attributable to the chemical components and mechanically attributable to the swelling stresses caused by residual stress.

(3) With increasing heat-treatment temperature, the concentration of glucose in thermally compressed wood gradually increased, while those of mannose and xylose decreased significantly. The recovery of thermally compressed heartwood was predominantly affected by mannose, while the recovery of thermally compressed sapwood was affected by mannose and xylose. Interestingly, glucose can enhance the shape fixation of compressed wood.

(4) High-temperature treatment degraded the mannose and xylose content in hemicellulose, which reduced the elasticity of wood, resulting in a reduction in elastic residual stress. This further led to reduced swelling stress and recovery, which realized the deformation fixation of compressed wood.

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