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Studies on pre-treatment by compression for wood impregnation III: effects of the solid content of low-molecular-weight phenol formaldehyde resin on the impregnation

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Abstract

There is increasing interests in the significant improvement of impregnation amount and efficiency in wood by the pre-treatment of compression. Thus, attention is paid to pre-treatment by compression for harder impregnation with resin because of the large viscosity and relatively large molecular weight of the resin. Low-molecular-weight phenol formaldehyde (PF) resin of solid content of 10–48% were impregnated by pre-treatment of compression at a compression ratio of 60% and 40% for poplar and Chinese fir, respectively, to systematically study the effects of solid content on the impregnation amount, weight gain percentage (WGP). In addition, the distribution of resin in wood was analyzed by profile density and was observed by confocal laser scanning microscope (CLSM), and the change of crystallinity index (C_{I}) of resin-impregnated wood was investigated by X-ray diffractometer. The results showed: (1) compared with immersion impregnation without compression, the impregnation by pre-treatment of compression, was much larger in amount and higher in efficiency for resins in all solid contents. In a solid content range of 10–48% and at a molecular weight of about 517, there was no significant difference of impregnation amount by compression among the resins at different solid content. (2) The WGP of poplar and Chinese fir increased with the increase of resin solid content. When the resin solid content increased from 10 to 48%, the WGP increased from 8.9 to 44.2% and from 5.2 to 24.9% for poplar and Chinese fir, respectively. (3) Resin is mainly distributed in vessels and tracheids despite a few being distributed in the fiber near the longitudinal end of the poplar specimens. With the increase of solid content, the resin distribution gradually changed from diffusing on the wall to depositing in the vessel or tracheid, while the resin distribution evenness along the longitudinal direction decreased. There was more resin deposited near the longitudinal end of Chinese fir at deeper depth than that of poplar. (4) The resin can be impregnated into the amorphous area of wood without causing any change in the crystallization area. The C_{I} decreases with the increase of resin solid content. After all, it is concluded that the pre-treatment by compression for wood impregnation with low-molecular-weight PF resin is effective for all the solid contents for both poplar and Chinese fir. The WGP increased with the increase of resin solid content, despite the decrease of evenness of resin distribution at high solid content.

Keywords: Pre-treatment by compression, Wood impregnation, Phenol formaldehyde resin, Solid contents, Weight gain percentage, Crystallinity

Introduction

Poplar and Chinese fir are two of the most important plantations due to their fast-growing rates despite their relatively low strengths. Resin impregnation into wood is one of the most effective treatments to improve the

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strength, dimensional stability, and durability if the amount of impregnation is enough [1]. However, the impregnation is usually very limited in amount and very low in processing efficiency due to the relatively low permeability of wood and the occupation of the air in wood. Moreover, compared with water or other very small molecular aqueous solution, high viscosity and relatively large molecular weight of resin make the impregnation harder. In this connection, more attention is paid on compression impregnation because it can open more pores, move out air or water and cause temporarily low pressure in wood, and result in an impregnation with large amount and high efficiency. At present, studies on compression impregnation have been done with water as the impregnation media [2–8], while only limited studies [9] on compression impregnation with the resin as impregnation chemicals. Luo et al. [10] studied the effect of compression ratio on the impregnation of phenol formaldehyde resin, but only on a solid content of 20%. Impregnation with solid contents of 15–30% with 5% interval has been studied on oven-dried Masson pine [11, 12] by vacuum and pressure method. The main driving force of this method and compression impregnation is both by pressure gradient, while the pressure inside wood is probably not the same because the permeability after vacuum treatment is hardly to change, while it increased remarkably after large-scale compression. Not changed permeability means pressure in some voids inside of wood might be hardly to change because of shortage of flow paths, while large-scale compression is believed to move out of air or water by volume shrinkage through increased flow paths. The effect of the solid content of resin on compression impregnation is not clear yet.

The solid content is a must-considering factor for resin impregnation. On the one hand, at the same impregnation amount, higher solid content means more effective content (resin) impregnated into the wood; on the other hand, higher solid content means the higher viscosity of the resin solution. How the solid content affects the impregnation and the weight gain percentage (WGP) of the wood in compression impregnation has not been systematically studied yet. In addition, the effect of the solid content on the resin distribution evenness, as well as on the crystallinity needs to be further studied.

Phenol formaldehyde (PF) resin is one of the most common resins used in the wood industry because of its high bonding strength, water resistance, flame resistance and excellent durability [13, 14]. Impregnation of phenol formaldehyde resin into wood will significantly increase the strength, dimensional stability and biological durability of wood [15–17]. The impregnation with low-molecular-weight resin will be easier than medium/high-molecular-weight resin. Therefore, the purpose of

this study is to systematically study the effect of the solid content of low-molecular-weight phenol formaldehyde resin on compression impregnation of poplar and Chinese fir plantation wood, including the impregnation amount, WGP, resin distribution evenness and the crystallinity, thus, to increase the understanding of wood chemical impregnation by compression method.

Materials and methods

Materials

Small clear specimens with the size of 30 mm (R) \times 50 mm (T) \times 100 mm (L) were prepared on the poplar (*Populus cathayana*) and heartwood of Chinese fir (*Cunninghamia lanceolata*) plantation with the air-dried density of 0.50 g/cm³ and 0.37 g/cm³ collected from Henan Province and Pingjiang Country of Hunan Province, respectively. The specimens were longitudinally connected for the same group to minimize the individual difference. Phenol formaldehyde (PF) resin (Dynea Co., Ltd., Zhaoqing, China) with a pH value of 8–9, a viscosity of 38.8 mPa s at 25 °C, solid content of 48%, and an average molecular weight of 517 (gel permeation chromatography) was used as impregnation chemicals. The solid content of the resin solution was adjusted to 10%, 20%, 30% and 40% by distilled water, and the viscosity of the solution was 5.8 mPa s, 12.3 mPa s, 19.4 mPa s and 26.2 mPa s at 25 °C, respectively.

Pre-treatment of compression

To minimize the effects of sample variation on the test results, all the samples were oven-dried (103 °C) first so that the growth stress was released to some extent, and then vacuum pressure treated (–0.1 MPa, 30 min; 1 MPa, 24 h) so that all the samples were fully water-saturated having a similar moisture content. The specimen was put between two stainless plates of the adapter and was compressed in the radial direction by the plate driven by the compression head of fully computer-controlled Instron 5582 Universal Test Machine. The compression was fixed or released by means of tightening or loosening the nuts on four bolts located at the corner of the adapter [5]. Six repeats for each solid content level, as well as for control specimens, have been tested. According to previous research results [6], the compression rates were set as 3 mm/min and 5 mm/min and compression ratios were set as 60% and 40% for poplar and Chinese fir, respectively.

Impregnation

The specimens, together with the adapter with tightened nuts, were totally immersed in PF resin solution (10%, 20%, 30%, 40%, 48% solid content). The release of compression was conducted by loosening the nuts in the solution and

was weighed immediately after the release of compression. Then specimens were continuously immersed in the solution and were weighed in some interval until the difference of weight in two consecutive measures was less than 0.2 g in 1 h. The air-dried wood (control) was immersed directly into the PF resin solution. The mass impregnation (I_M , g/cm³) was calculated by the weight of PF resin solution gained per unit volume of oven-dried wood, as shown in Eq. (1). After the impregnation, the specimens were cured by gradient temperature rise (45 °C, 10 h; 60 °C, 10 h; 80 °C, 10 h; 100 °C, 0.5 h) [18]. Then the specimens were weighed, and its WGP (%) was calculated according to Eq. (2):

$$I_M = \frac{m_2 - m_1}{\nu}, \quad (1)$$

$$\text{WGP} = \frac{W_t - W_c}{W_c} \times 100, \quad (2)$$

where, m_1 and m_2 are the weight of the specimen before and after impregnation (g), respectively; ν is the oven-dried volume of the specimen (cm³); W_c and W_t are the oven-dried weight of specimen before and after impregnation (g), respectively.

Profile density determination

To study the longitudinal distribution of PF resin along with the specimen, the profile (radial \times tangential) density of modified wood was measured by DENSE-LAB X profile density analysis apparatus (Electronic Wood Systems GmbH, Hameln, Germany) to evaluate the resin distribution evenness. Half-length (longitudinal, 50 mm) specimen were prepared and measured because of the limitation of apparatus on specimen size.

The specimen was put in between the X-ray source with a stable intensity and the X-ray sensor so that the average X-ray intensity and their difference after penetration of the specimen of each individual cross-section along longitudinal direction were measured by the sensor. The actual density by mass/volume method is input to the instrument before measuring the profile density. The profile density curve was determined on the X-ray intensity and the actual density.

Confocal laser scanning microscopy observation

The microscopic distribution of PF resin within the wood at different longitudinal positions was examined using the LSM 980 confocal laser scanning microscope (CLSM) (Carl Zeiss, Oberkochen, Germany). According to the characteristics of profile density, the cross-sectional slices with the dimensions of 5 mm (T) \times 5 mm (R) \times 25 μ m (L) were prepared at 0.2 mm, 10 mm and 50 mm away from the specimen's longitudinal end at the central position of

the cross-section of the specimen. The slices were dyed in 5% toluidine blue solution and rinsed, and then dehydrated with ethanol solution of different concentrations (50%, 75%, 95%, 100%). After dehydration, placed slices on glass slides covered with glass coverslips. Phenolic substances in the wood cell wall can spontaneously fluoresce under exciting light at a specific wavelength [19], which can be inhibited after toluidine blue solution dyeing. Meanwhile, PF resin cannot absorb toluidine blue, autofluorescence is not inhibited, so the distribution of resin in wood cells can be observed.

X-ray diffraction measurement

In order to further explore the influence of PF resin on the crystallinity index (C_{rI}) of wood, D8 Advance X-ray diffractometer (Bruker, Billerica, Germany) was used to obtain the diffraction pattern according to the X-ray diffraction (XRD) phenomenon of crystal structure in the wood cell wall [20] and calculated the C_{rI} of cellulose. The control and modified wood flour (80 mesh) for XRD analysis were prepared from the longitudinal end of the specimen where the resin was fully impregnated even at a high solid content. The voltage and current were set to 40 kV and 40 mA, respectively, and the scanning range to 5°–40° (2θ). The relative C_{rI} (%) was estimated based on the method proposed by Segal [21]:

$$C_{rI} = \frac{I_{002} - I_{am}}{I_{002}}, \quad (3)$$

where I_{002} and I_{am} represent the intensity of the 002 crystalline peak and diffraction of the amorphous part, respectively.

Results and discussion

Effect of the solid content of PF resin on compression impregnation

The results of impregnations with resins at different solid contents (Fig. 1) showed the impregnations at the time of the completion of compression release accounted for 60–70% of the total impregnation, although the release was completed in a very short time. The impregnation reached about 93% of the total impregnation in about 290 min for both species and then increased very slowly afterward. For the control specimen (immersion in resin without pre-treatment by compression), the impregnation increased much slowly and had much lower impregnations in all solid content conditions than that of compression impregnation. The impregnation of resins at different solid contents showed no clear pattern, although the impregnation at 48% solid content showed the lowest impregnation in both compression and control species for two species. The total impregnations at all the solid content conditions in compression specimens showed

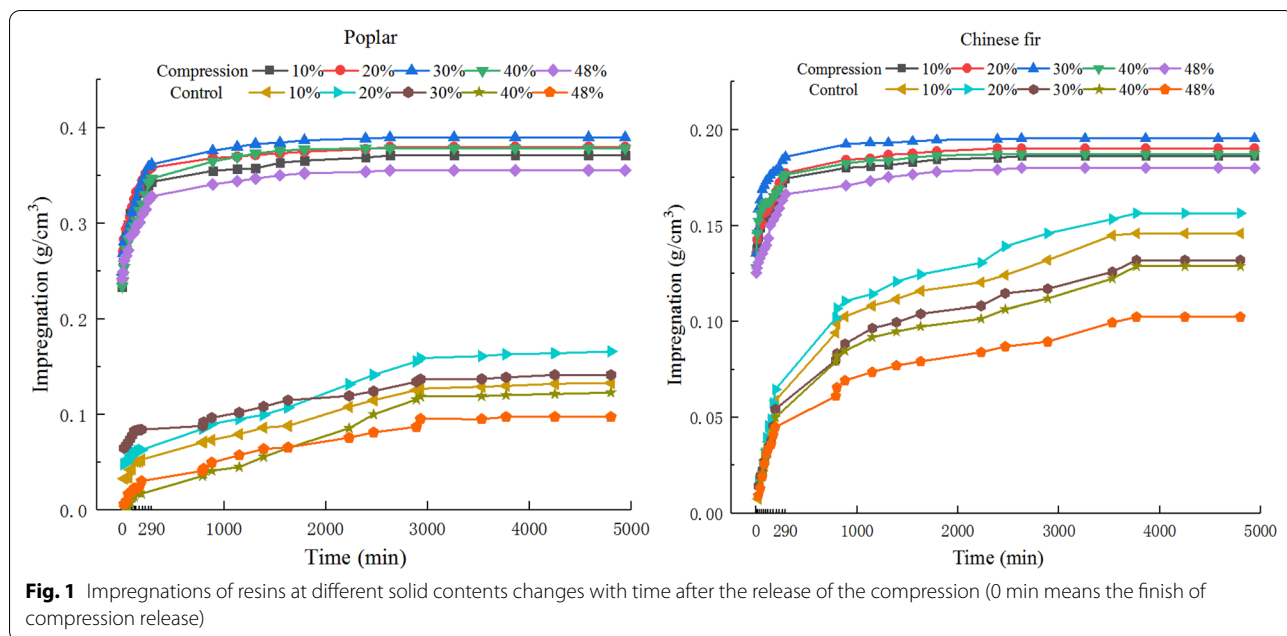


Fig. 1 Impregnations of resins at different solid contents changes with time after the release of the compression (0 min means the finish of compression release)

a variation of 3.43% and 3.00%, while that in controlled specimen showed a variation of 19.01% and 15.33% for poplar and Chinese fir, respectively, suggesting that the compression weakened the effect of solid conditions on the impregnations.

The compressed wood almost finished its recovery as soon as the completion of compression release, causing the temporarily low pressure within the wood compared with the pressure outside the wood. The resin was quickly driven into the wood under this pressure gradient. This explains the portion of impregnation is very high (60–70%) during the release period. Due to the limited permeability of the wood, it might still exist some pressure difference and available space after the completion of compression release, the impregnation before 290 min increased obviously, and it increased slowly afterward.

The impregnation in the control specimen was mainly driven by the capillary pressure [22], which was much smaller than the pressure difference caused by wood

recovery. In addition, due to more fluid flow paths [23] and the move out of the air by compression, it showed a much larger amount and higher efficiency in pre-treatment by compression than that in control, as illustrated in Fig. 1.

No clear pattern and small variation (3.43% and 3.00% for poplar and Chinese fir, respectively) among different solid contents (Fig. 1) suggested that the effect of the solid content on impregnation is not obvious. The results of analysis of variance (ANOVA) (Table 1) confirmed this conclusion. The porosity ratio of wood in the same species is same, and the available impregnation void after the wood recovery at the same compression ratio is also same. Suppose the effects from the viscosity of resin in different solid contents are neglectable and the molecule is small enough to pass the perforate or pit, the impregnation amount of resin in different solid contents under same impregnation method should be same for the same species of wood. The average molecular weight of the

Table 1 ANOVA of the influence of resin solid contents on the compression impregnation amount

Species	Source of difference	Sum of squares	Degree of freedom	Mean square	F value	P value
Poplar	Among groups	0.011	4	2.86E-3	1.75	0.19
	Within groups	0.024	15	1.63E-3		
	Total	0.036	19			
Chinese fir	Among groups	0.005	4	1.36E-3	1.69	0.19
	Within groups	0.016	20	8.05E-4		
	Total	0.022	24			

P > 0.05 means the influence is not significant

resin in this test was 517. In this condition, the impregnation of resin in different solid contents (10–48%) had a small variation. This explains the difference of impregnation amount between resins with different solid contents is not significant (Table 1).

Although the difference between resin with different solid contents was not significant, there still existed some differences. The impregnation amount of resin solution (I_M) in Fig. 1 is calculated based on the mass change of resin solution (Formula 1). The density of the resin increased with the increase of solid content. The impregnations of resin volume per volume of wood (I_V) (Table 2) for both species were almost same for those with a solid content from 10 to 30%, while they slightly decreased when the solid contents were beyond 30%. This was probably attributed to the viscosity change. The viscosity of resin increased from 5.8 to 19.4 mPa·s with the increase of solid content from 10 to 30% in a slope, while that increased sharply from 26.2 to 38.8 mPa·s with a steep slope. This explains the volume impregnation (I_V) of the resin remains almost the same when the solid content is equal or less than 30%, and decreases slightly when the solid content is greater than 30% (Table 2), although the influence is not statistically significant.

The fact that the impregnation in poplar is higher than that in Chinese fir is attributed to, in one hand, the penetration through perforate in poplar is much easier than that through the pit in Chinese fir [24]; in the other hand, the compression ratio of poplar (60%) is bigger than that of Chinese fir (40%).

Effect of the solid content of PF resin on WGP of wood

Figure 2 shows the effect of the solid content of PF resin on WGP of poplar and Chinese fir. When the solid content of PF resin increased from 10 to 48%, the WGP increased from 8.9–44.2% and 5.2–24.9% for poplar and Chinese fir, respectively.

At the solid content range from 10 to 48%, the difference of the mass impregnation amount of resin solution

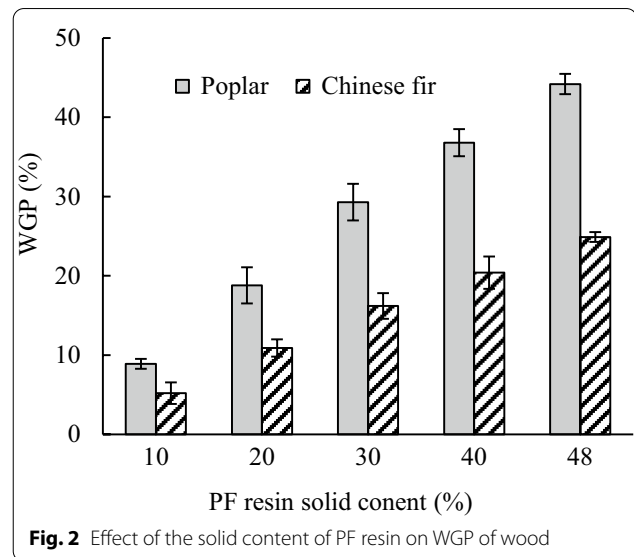


Fig. 2 Effect of the solid content of PF resin on WGP of wood

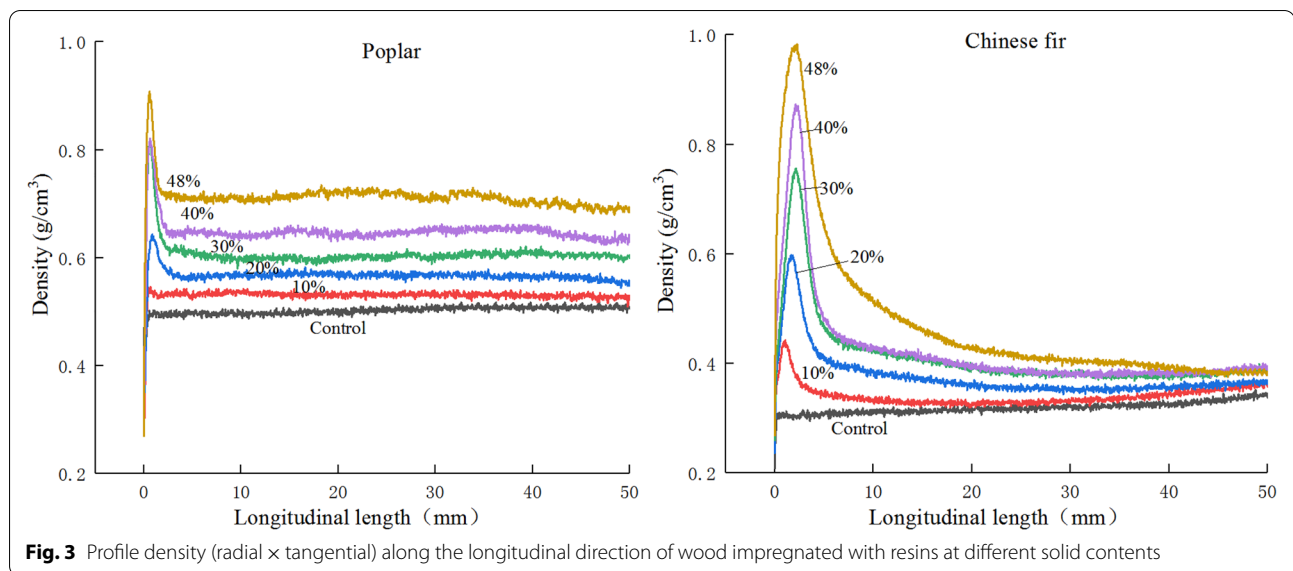
was not significant (Table 1). With the increase of the solid content of the resin solution, the water content in the solution decreased linearly. Therefore, after drying, the WGP increased with the increase of the solid content of resin. The fact that the WGP in poplar was higher than that in Chinese fir was attributed to a much higher impregnation amount in the former than in the latter.

Effect of the solid content on the distribution of resin in wood

The density increased from 0.53 g/cm³ to 0.71 g/cm³ and 0.34–0.47 g/cm³ with the increase of solid content from 10 to 48% for poplar and Chinese fir, respectively. The profile density (radial × tangential) (Fig. 3) showed the density of wood impregnated with high solid content resin is greater than that impregnated with low solid content resin in all longitudinal directions for both species. Compared with the control specimen (without impregnation), the density of impregnated specimen fluctuated in some extent. The higher the solid content was, the

Table 2 The impregnation in mass and in volume of resins at different solid contents

Solid content (%)	Resin density (g/cm ³)	Poplar		Chinese fir	
		Mass impregnation (I_M) (g/cm ³)	Volume impregnation (I_V) (cm ³ /cm ³)	Mass impregnation (I_M) (g/cm ³)	Volume impregnation (I_V) (cm ³ /cm ³)
10	1.02	0.371	0.364	0.186	0.183
20	1.04	0.380	0.365	0.190	0.183
30	1.06	0.390	0.368	0.195	0.184
40	1.08	0.378	0.350	0.187	0.173
48	1.10	0.355	0.323	0.180	0.164



more the fluctuation was, suggesting that the resin distribution evenness decreased with the increase of solid contents of resin. The difference of density profile peak between two species suggested that there was more resin deposited near the end in a deeper depth of Chinese fir than that of poplar.

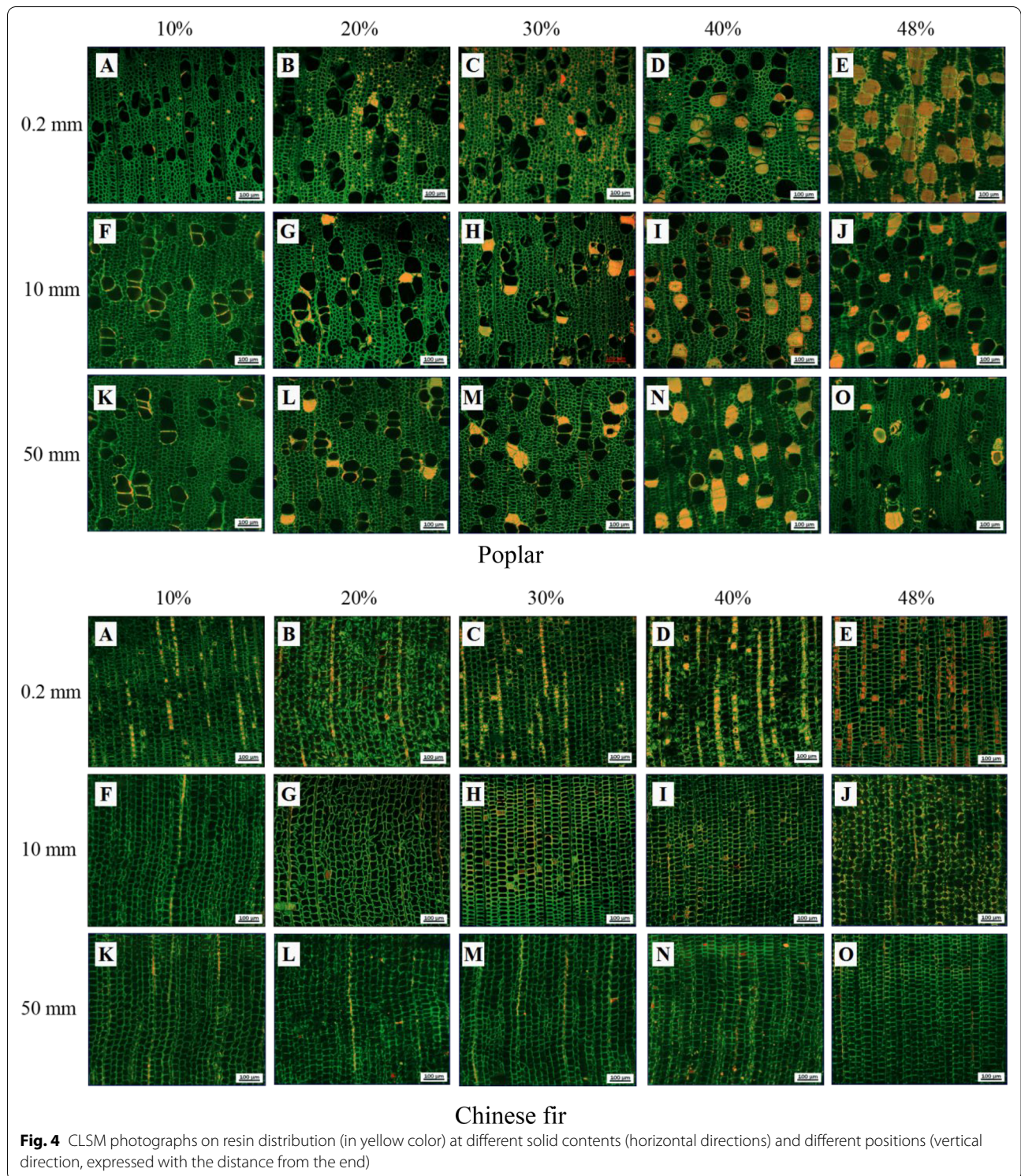
CLSM photographs (Fig. 4) showed that, at a solid content of 10% and 0.2 mm from the end (Fig. 4A), the resin deposition (in yellow color) was found on fiber, tracheid, while not on the vessel; at a solid content of 10% and 10 mm from the end (Fig. 4F), the resin deposition was found on tracheid, not in fiber and vessel, suggesting that the order the resin more likely to be trapped is fiber > tracheid > vessel. The resin distributions difference among different distance from the end, for poplar, showed no obvious difference for all solid contents except 48%; for Chinese fir, showed obvious difference at all solid contents, and this difference was becoming more obvious with the increase of solid content. With the increase of resin solid content, the resin was diffused in and then deposited in the vessel or tracheid, suggesting that resin distribution increases with the increase of solid content in both species. This is understandable because the impregnation amount of resin solution in volume was almost same for resin solutions at all solid contents (Table 2).

The impregnation is dependent on the permeability of wood. The tracheid has more pits, longer length, more lumen space than fibers do; the vessel has more lumen space and less penetration resistance than the tracheid does because the perforate in the vessel is much easier to penetrate than the pits in the tracheid. Therefore, the permeability order is fiber < tracheid < vessel. This

explains why the resin was deposited in the fiber only at 0.2 mm position, and why the difference among different positions was obvious in Chinese fir while not obvious in poplar with 48% solid content exception. The impregnation is mainly conducted from two ends after release of compression due to much bigger permeability in the longitudinal direction than in transverse direction [25], the resin solid content might be slightly decreased along the longitudinal direction in tracheid since the resin may be trapped and the solution may go further. Large viscosity at 48% solid content was a disadvantage factor for impregnation that may cause the resin trapped in vessel despite high permeability of vessel, while the resin was trapped in the tracheid even at viscosity as low as 10% solid content.

The conclusions that resin distribution increases with the increase of solid content in both species and decreases with the increase of distance from the end are in agreement with the conclusion on Masson pine impregnation with PF resin conducted by Wang [11, 12].

Based on the density profiles along the longitudinal direction (Fig. 3) and the CLSM photographs on resin distribution (Fig. 4), as the compromise of distribution evenness and a high WGP, it is recommended a solid content range of 10–40% and 10–30% for poplar and Chinese fir, respectively. The effect of resin diffusing or depositing in the wood cell on the strength of wood need to be further studied, to find a solution with a reasonable amount of resin, that makes the wood high in strength while light in weight. Meanwhile, the effect of different molecular weights on impregnation also needs to be studied.



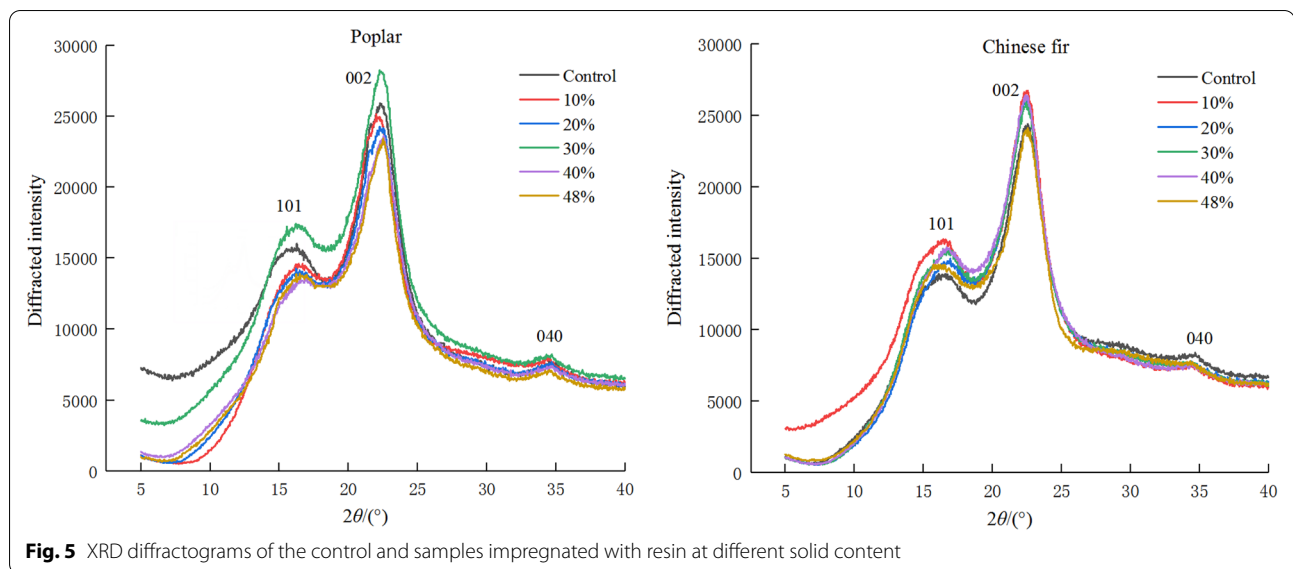


Fig. 5 XRD diffractograms of the control and samples impregnated with resin at different solid content

Table 3 Diffraction intensity and C_rI of wood impregnated with resin solution at different solid contents

Species	Solid content (%)	I_{am}	I_{002}	C_rI (%)
Poplar	Control	13,287	25,942	48.8
	10	13,341	24,979	46.6
	20	13,059	24,281	46.2
	30	15,512	28,242	45.1
	40	13,012	23,617	44.9
	48	12,984	23,444	44.6
Chinese fir	Control	11,795	24,405	51.7
	10	13,245	26,756	50.5
	20	13,046	26,126	50.1
	30	13,374	25,983	48.5
	40	13,976	26,446	47.2
	48	12,876	24,138	46.7

Effect of the solid content of PF on the C_rI of impregnated wood

Figure 5 and Table 3 show the XRD diffractograms and C_rI of the control and samples impregnated with resin solutions at different solid content, respectively. As presented in the diffractograms, cellulose patterns were identified with 101, 002 and 040 peaks observed around 16.3°, 22.2° and 34.5° 2θ angles, respectively [20]. In which, the angle of 002 peak showed no change, suggesting that the crystal region has not been affected. As presented in Table 3, the C_rI decreased with the increase of the solid content of resin solution. This was attributed to both the significantly increased volume of microvoids caused by compression [10] and the swollen cell wall at high moisture state, which facilitated the

penetration of the resin into amorphous region and had a hydroxy bond at amorphous region. Studies on wood impregnation with PF resin [26, 27] showed that PF resins penetration into the cell wall was found on the resin with a molecular weight of 290–470, not on the resin with a molecular weight of 820. The average molecular weight of the resin used in this study was 517, suggesting that it can penetrate the cell wall, and therefore had a chance to bond with amorphous region of cellulose. With the increase of resin solution solid content, more resins were penetrated to amorphous region, while it is very hard to penetrate to crystal region [28]. Increased amorphous region and rather stable crystal region made the C_rI decrease. In addition, the pH value of the resin before dilution was alkaline which reportedly reduces the C_rI of wood [29]. The lower the concentration of PF resin solution, the lower the pH may be. This might attribute to the decrease of C_rI with the increase of resin solid content.

It is believed that the wood strength is proportional to the C_rI [30]. In this study, the C_rI decreased with the increase of solid content of PF resin due to the increase of amorphous region, while the crystal region had not been affected. How this change will affect the wood strength needs to be further studied.

Conclusions

- (1) Compared with immersion impregnation without compression, the impregnation by pre-treatment of compression, was much larger in amount and higher in efficiency for resins in all solid contents. In a solid content range of 10%–48% and at a molecular weight

of about 517, there was no significant difference of impregnation amount by compression among the resins at different solid content.

- (2) The WGP of poplar and Chinese fir increased with the increase of resin solid content. When the resin solid content increased from 10% to 48%, the WGP increased from 8.9% to 44.2% and from 5.2% to 24.9% for poplar and Chinese fir, respectively.
- (3) Resin is mainly distributed in vessels and tracheids despite a few distributed in the fiber near the longitudinal end of the poplar specimen. With the increase of solid content, the resin distribution gradually changed from diffusing on the wall to depositing in the vessel or tracheid, while the resin distribution evenness along the longitudinal direction decreased. There was more resin deposited near the longitudinal end of Chinese fir in deeper depth than that of poplar.
- (4) The resin can be impregnated into the amorphous area of wood without causing any change in the crystallization area. The C_rI decreases with the increase of resin solid content.

After all, it concluded that the pre-treatment by compression for wood impregnation with low-molecular-weight PF resin is effective for all the solid contents for both poplar and Chinese fir. The WGP increased with the increase of resin solid content, despite the decrease of evenness of resin distribution at high solid content.

Abbreviations

PF: Phenol formaldehyde; I_M : Mass impregnation; WGP: Weight gain percentage; CLSM: Confocal laser scanning microscope; XRD: X-ray diffraction; C_rI : Crystallinity index; I_v : Volume impregnation; ANOVA: Analysis of variance.

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Author contributions

YZ and HW wrote the manuscript. YZ conceived and designed the experiments, HW performed the experiment of the study and was responsible for data collection; HW and YZ analyzed the data. Both authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analyzed during 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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