

Study of Chemical Modification by Impregnation of Fresh Poplar Log and by Hot-Press Drying Process

Heyu Chen, Qian Lang, Hao Zhang, Guofeng Wu, Xue Zheng, and Junwen Pu *

In this work, fresh poplar logs were chemically impregnated with a pulse-dipping machine. The impregnated timbers were compressed and dried by use of a multilayer hot-press drying kiln. With a compression rate of 28.67%, such an approach not only enhanced the density and mechanical properties of the treated wood, but also influenced the set recovery from the compressive deformation. Extra urea was added into the modifier to optimize the effect of the set recovery; however the result was a slight decrease in the mechanical properties. The positions of the X-ray diffraction (XRD) peaks did not change, which indicated that the structure of the cellulose was not noticeably affected by this treatment. The Fourier transform infrared spectroscopy (FT-IR) analysis showed that the intensity amide (N–C=O) absorption peak increased significantly due to the chemical impregnation. Scanning electron microscopy (SEM) showed that the high strain in the hot-press process drastically reduced the volume of void spaces in the specimens and deformed the cell lumens.

Keywords: Poplar wood; Methylolurea; Urea; Chemical impregnation; Hot-press drying

Contact information: College of Material Science and Technology, Beijing Forestry University, Beijing, 100083, PR China; *Corresponding author: jwpu@bjfu.edu.cn

INTRODUCTION

Considering that natural forest resources are significantly decreasing all over the world, fast-growing wood should be effectively utilized in terms of its recyclability and low environmental pollution. Among the various techniques for improving the quality of timber, chemical modification and mechanical compression are thought to be promising methods (Buyuksari 2012; Chen *et al.* 2012; Hill 2006).

Fast-growing poplar wood is a porous material that has been known for its ability to be compressed to improve its mechanical properties by increasing its density. The main problem with this process is the fixation of compressive deformation. Three basic mechanisms of fixing the compressive deformation were identified by Morsing (2000): preventing the wood from being re-softened by changing the hygroscopicity of the cell wall and thus making the cell wall inaccessible to water; forming covalent crosslinks between the wood components in the deformed state; or releasing the stresses stored in the microfibrils and matrix polymers during compression.

In an effort to achieve high mechanical performance and improve dimensional stability, densification combined with oil-heat treatment was studied (Fang *et al.* 2011, 2012a, b). With suitable treatment, oil palm trunk could be an appropriate choice for an alternative source of compressed wood (Sulaiman *et al.* 2012). Also, many more attempts have been made to develop a suitable process for the densification of wood including compression treatment combined with steam and heat. Usually, density enhancement is

achieved through a dynamic process that combines temperature ranging from 130 to 220 °C, saturated steam, and mechanical compression perpendicular to the grain (Boonstra and Blomberg 2007; Dogu *et al.* 2010; Fang *et al.* 2012a, 2012b; Higashihara *et al.* 2000; Inoue *et al.* 2008; Kutnar *et al.* 2009; Kutnar and Kamke 2012; Wu *et al.* 2010). Water-soluble, low molecular weight resin systems have been studied extensively and are supposed to have non-bonding cell wall swelling during impregnation processes (Inoue *et al.* 1991a, 1991b; Ohmae *et al.* 2002). Adding low-cost urea into the modifier system could lead to such effects as protecting the wood against biodeterioration, wood surface plasticizing, or presenting as a formaldehyde scavenger for compreg wood (Ejechi 2003; Wang and Cooper 2005; Adawiah *et al.* 2012).

However, most of these methods deal with small wood samples. The objective of this study was to treat the wood sample on a larger scale. First, we impregnated the fresh poplar log with the pulse-dipping machine, and then the impregnated timbers were compressed and dried in a multilayer hot-press drying kiln. Furthermore, extra urea was added into the prepolymer (methylolurea) for impregnation to investigate the potential effect on the compressive formation and the physical–mechanical performance of the poplar wood.

EXPERIMENTAL

Materials

The fresh poplar (*Populus euramevicana* cv. ‘I-214’) wood samples used in this study were obtained from Beijing, China. The initial moisture content of the fresh poplar ranged from 70% to 80% before the impregnation.

Preparation of Modifier Aqueous Solution

A reaction vessel was charged with urea, formaldehyde, and ammonia at a certain molar ratio (Pu *et al.* 2009a). The reaction mixture was stirred and kept at 30 °C for 3 h. Then, ammonium chloride was used to adjust the pH of the methylolurea within the range of 5 to 7. Finally, the reacted mixture was transferred into a container and sealed.

Fresh Poplar Log Pulse-dipping and Hot-press Kiln Drying

The dimensions of the fresh poplar logs for impregnation were 200 mm in diameter and 1000 mm in length. The logs were divided into two groups: The first group was impregnated by 30% methylolurea (w/w) aqueous solution with a solid content of 19.2%; the second group was impregnated by 20% methylolurea (w/w) and 10% urea (w/w) aqueous solution with a solid content of 22.8%. Next, the fresh poplar logs were impregnated by a pulse-dipping machine at a pressure of 0.7 to 0.8 MPa for 30 min (Pu *et al.* 2009b). After soaking, the impregnated logs were sawn into boards about 50 mm thick (tangential) × 120 mm wide (radial) × 1000 mm length (longitudinal) for kiln drying. It took approximately 150 h for drying. The highest drying temperature was 130 °C, and the pressure on the boards was 0.5 MPa, which was maintained all the time during the drying period (Pu *et al.* 2009c).

Physical and Mechanical Properties

Ten specimens of untreated and treated wood were analyzed in a universal mechanical testing machine (AG-100KNIMO, Japan). Tests were carried out according

to the Chinese GB/T 1936.1-91 method for testing the bending strength of wood, the Chinese GB/T 1935-91 method for testing the compressive strength parallel to the grain of the wood. The density of untreated and treated wood was analyzed according to the Chinese GB/T 1933-91 method for determining the density of wood.

Set Recovery

To measure set recovery as affected by the impregnated chemicals, the specimens (50 mm longitudinal, 15 mm tangential) were cut from compressed wood that had been conditioned in an environment-controlled room (20 °C, 65% relative humidity, 2 wk). Specimens were first dried in a convection oven overnight at 103 °C to establish an initial oven-dry weight and dimensions. The specimens were then soaked in water for 24 h and again oven-dried. The procedure was repeated for a total of five wet/dry cycles. After each cycle, the percentage of set recovery was determined by the following expression (Equation 1),

$$\text{Set recovery} = [(t_s - t_c) / (t_i - t_c)] \times 100(\%) \quad (1)$$

where t_s is the oven-dry thickness after soaking, t_c is the oven-dry compressed thickness, and t_i is the initial uncompressed thickness.

X-ray Diffraction (XRD) Analysis

The crystallinity of the samples from untreated and treated wood was evaluated by XRD, using a Shimadzu diffractometer (model XRD 6000, Kyoto, Japan). The measurement conditions were: CuK α radiation with graphite monochromator, 30 kV voltage, and 40 mA electric current. The patterns were obtained within a 5° to 30° 2 θ angular interval with a 0.05° step and a scan speed of 2°·min⁻¹. The degree of crystallinity was calculated as the ratio of the intensity differences in the peak positions.

Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

The untreated and treated wood specimens of 120 mesh size were directly used in the FTIR spectroscopy measurement. The dried specimens were embedded in potassium bromide (KBr) pellets and analyzed using a Tensor 27 (Bruker, Germany) spectrometer. They were recorded in the absorption mode in the range of 4000 to 400 cm⁻¹ with an accumulation of 64 scans at a resolution of 4 cm⁻¹.

Scanning Electron Microscopy (SEM) Analysis

A JSM 5900 SEM (Tokyo, Japan) was used to observe both elemental analysis and morphology of the untreated and treated wood. Samples to be observed under the SEM were mounted on conductive adhesive tape. Then the specimens were gold-coated with gold-palladium in a JSM sputter coater (Tokyo, Japan) with the thickness of the gold coating about 15 to 20 nm and observed in the SEM using a voltage of 15 kV.

RESULTS AND DISCUSSION

Density and Mechanical Properties

The density and mechanical properties of the wood samples are shown in Table 1.

Compared with the untreated control, the basic density of the methylolurea treated and urea–methylolurea treated wood both increased by 13.2%; the oven-dried density increased by 61.9% and 64.3%; the air-dried density increased by 57.8% and 62.2%; the bending strength increased by 83.9% and 58.8%; the compressive strength parallel to the grain increased by 51.9% and 43.2%.

Both impregnation and compression made a contribution to the density increase. On the one hand, with a compression rate of 28.67%, the wood interior porosity was altered to a lesser extent; on the other hand, it was the impregnated chemicals that could fill into the wood cell wall and cell cavity and also cause the density increase. What is more, with the same compression ratio, the urea–methylolurea treated wood acquired a slightly higher density compared with the wood treated with methylolurea alone, which was consistent with the modifier solid content (methylolurea was 19.2% and urea–methylolurea was 22.8%) in the impregnation process. The treated wood exhibited significantly higher mechanical properties due to the impregnated modifier that occupied the space within the cell wall. This led to the polymerization between the functional groups of wood and the chemicals. In addition, there was more wood composite per unit volume because the compression process also played an important role.

In normal conditions for the wood samples, the higher the density, the stronger the mechanical properties would be. However, comparing the two kinds of treated wood, the higher density (urea–methylolurea treated wood) led to lower mechanical properties. There is no doubt that the mechanical properties should be closely related with the strength of the wood cell wall. This result indicated that it is the cross-linking reaction caused by the methylolurea that enhanced the mechanical properties; although the extra urea did increase the density of the wood, it has less to do with any improvements in mechanical properties.

Table 1. Physical and Mechanical Properties of the Wood Samples

	Untreated Control	Methylolurea Treated	Urea–Methylolurea Treated
Basic density ($\text{g}\cdot\text{cm}^{-3}$)	0.38	0.43	0.43
(SD)	(0.06)	(0.04)	(0.04)
Oven-dried density ($\text{g}\cdot\text{cm}^{-3}$)	0.42	0.68	0.69
(SD)	(0.08)	(0.04)	(0.04)
Air-dried density ($\text{g}\cdot\text{cm}^{-3}$)	0.45	0.71	0.73
(SD)	(0.08)	(0.06)	(0.04)
Bending strength (MPa)	85.60	157.40	135.90
(SD)	(10.07)	(12.82)	(11.24)
Compressive strength parallel to the grain (MPa)	51.20	77.80	73.30
(SD)	(2.03)	(1.58)	(1.56)

Set Recovery

To make a comparison of set recovery, the fresh poplar without impregnation was dried at the same temperature, steam condition, and compression ratio as the impregnated wood (this compressed wood without impregnation will still be called an untreated control). As shown in Fig. 1, with increasing the time of the water soaking/drying cycle, all wood samples' set recovery was increased. The most obvious increase appeared in the second cycle, and after the fourth cycle set recovery reached the maximum. Comparing the untreated control and the impregnation-treated wood samples, the impregnated chemicals significantly affected the set recovery, such that the impregnation-treated wood

acquired a much smaller set recovery during all the five soaking/drying cycles. Furthermore, details in Fig. 1 (upper right) show that the extra urea-impregnated wood samples acquired even lower set recovery within the first two cycles. However, for the last three cycles, there was no evident difference between the two treated wood samples, which may be a result of the urea dissolving in the water soaking.

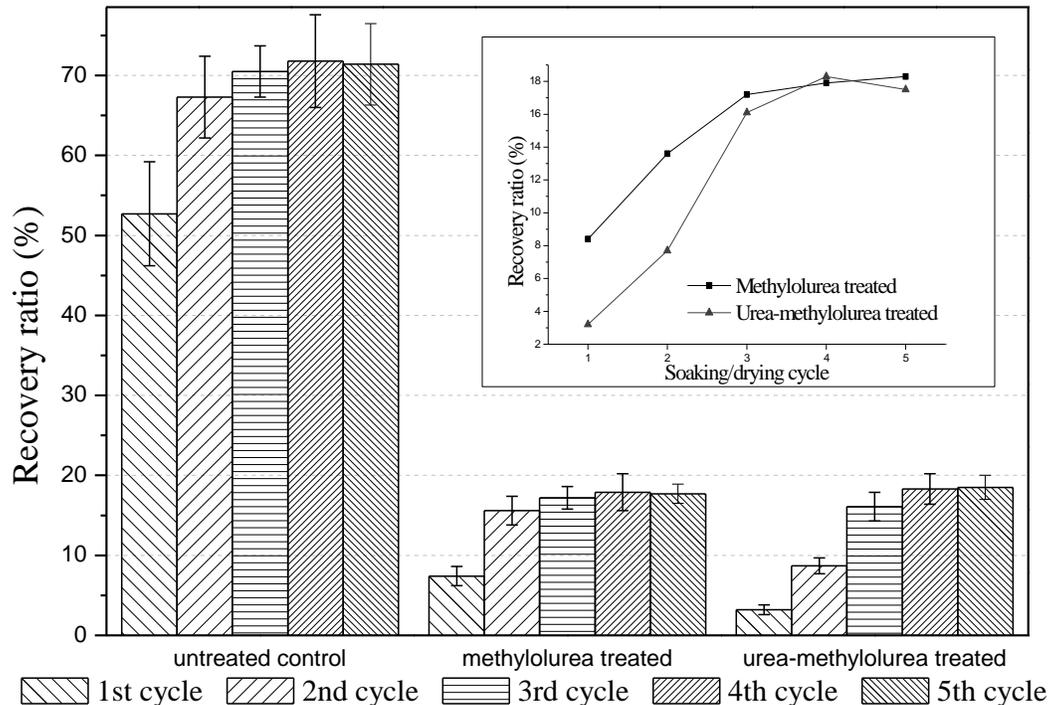


Fig. 1. Set recovery of wood compressed under untreated, methylolurea treated, and urea-methylolurea treated conditions after the hot-press drying process at 1, 2, 3, 4, and 5 soaking/drying cycles

There is no doubt that saturated steam exposure can influence the physical and mechanical properties of wood (Lenth and Kamke 2001). A few explanations for the effect of the steam and thermal treatments on the wood in transverse compression have been proposed. According to a study on the influence of pre-steaming at 120 to 220 °C for 5 to 20 min on the fixation of compressed *Pinus densiflora* specimens (Inoue *et al.* 2008), the drying process in the current study's last procedure which was saturated steam treated at 130 °C for 30 min could hardly have enhanced the compressibility of the wood. In this way, the low amount of springback for compressed wood should be the result of the chemical impregnation. In the absence of high temperature (at least 180 °C), methylolurea can induce a permanent fixation of compressive deformation due to the formation of covalent bonds in the deformed position. The impregnated extra urea could optimize the effect of the set recovery probably because the urea softened the wood microfibril by changing the polar side groups on the molecular structures of cellulose, hemicelluloses, lignin, and extractives (Davidson and Baumgardt 1970). This chemical softening effect, like steam or heat, can plasticize the wood cell wall. In this way, the compression applied in the presence of extra urea allows not only for the deformation of the cell walls without fracturing, but also absorbs the released formaldehyde from the methylolurea curing while the wood is in its compressed and densified form. Overall, the added extra urea

significantly reduced the buildup of internal stresses during the hot pressing and residual stresses after drying.

XRD Analysis

The XRD patterns of untreated and treated wood samples are shown in Fig. 2. For the untreated wood, the peak of the cellulose crystal diffraction (101) occurred near the 16° diffraction angle, and an obvious trough was observed near 19.1° , which is a characteristic of the amorphous wood region. The most significant diffraction peak (002) of the cellulose crystal surface was near 22.5° (Cave 1997; Mulinari *et al.* 2010). However, the diffraction patterns changed little for the treated wood. The trough at 19.1° was smoothed, which was interpreted as the formation of acetic acid (from hemicelluloses due to the high temperature) by heating of the wood in the presence of water or steam leading to less amorphous cellulose (Mitchell 1988). According to the results of the experiment, the crystallinity of the untreated wood was 37.88%, while the results for methylolurea treated wood and urea–methylolurea treated wood were 38.79% and 39.03%, respectively. Comparing the three curves, the position of the peaks did not change, which indicated that the structure of the cellulose was steady. The crystallinity of the treated wood increase may be caused by the hot-press drying process, because usually the high temperature degrades the hemicelluloses to a greater extent than the other macromolecular components (Bourgois *et al.* 1989). Considering the temperature used was 130°C , far below the temperature in the thermal treatment, it probably had little influence on the hemicelluloses, where the crystallinity increased slightly.

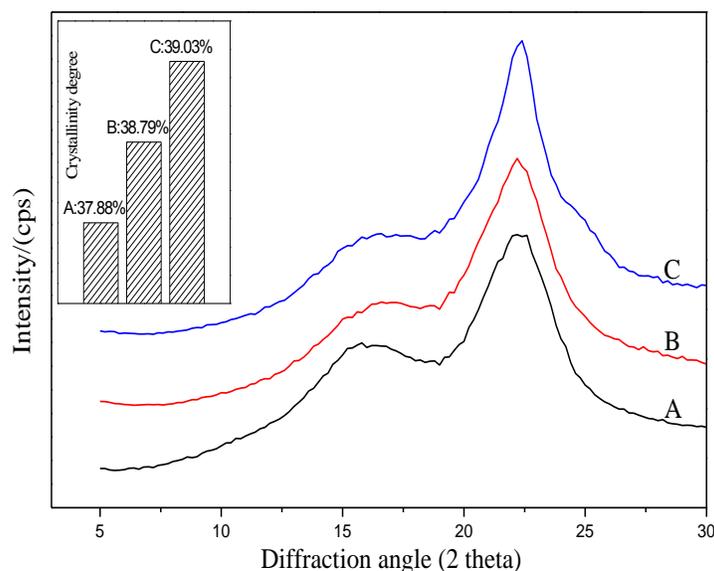


Fig. 2. XRD curves of: (A) treated wood without heat, (B) methylolurea treated wood, (C) urea–methylolurea treated wood

FT-IR Analysis

Figure 3 shows the FT-IR spectrum of the untreated and the treated wood. The absorption bands assignment is presented. The FT-IR spectrum of the untreated control (Fig. 3A) presents the same basic structure as all the wood samples, which is the presence of bands at 3330 cm^{-1} for $-\text{OH}$ stretching, 2908 cm^{-1} for $-\text{C}-\text{H}$ stretching, 1619 cm^{-1} for $-\text{OH}$ bending and 1738 cm^{-1} for $\text{C}=\text{O}$ stretching, aromatic $\text{C}-\text{H}$ in plane deformation, and symmetrical $\text{C}-\text{O}$ stretching at 1028 cm^{-1} (Devi and Maji 2011; Horikawa *et al.* 2006;

Rodrigues *et al.* 2004). In Fig. 3B and Fig. 3C, the modifier impregnation was evidenced by strong absorptions at 1650 cm^{-1} corresponding to amide ($\text{N}-\text{C}=\text{O}$), which was from the methylolurea and urea. As shown in Fig. 3C, two small absorption peaks appeared at 3330 cm^{-1} and 3473 cm^{-1} , characteristic of the $-\text{NH}_2$, which marked the defining feature of the impregnated extra urea. The chemical bonding was confirmed by the clear decrease at 2908 cm^{-1} for $-\text{C}-\text{H}$ stretching and at 1028 cm^{-1} for aromatic $\text{C}-\text{H}$ in the plane deformation after the chemical treatment, which could be a result of the reaction between the group ($-\text{NHCH}_2\text{OH}$) in methylolurea and the group (lignin at least) in wood. In addition, the methylolurea treated wood (Fig. 3B) showed even lower absorption at 2908 cm^{-1} and 1028 cm^{-1} than the extra urea treated wood (Fig. 3C), indicating that adding methylolurea acts as an active agent and adding urea acts as a softening agent. Also, with the temperature increase in the drying process, the chemical bonding accelerated between the wood and the methylolurea. At the same time, with the moisture content of the timber decreasing, polymerization also occurred in the modifier itself.

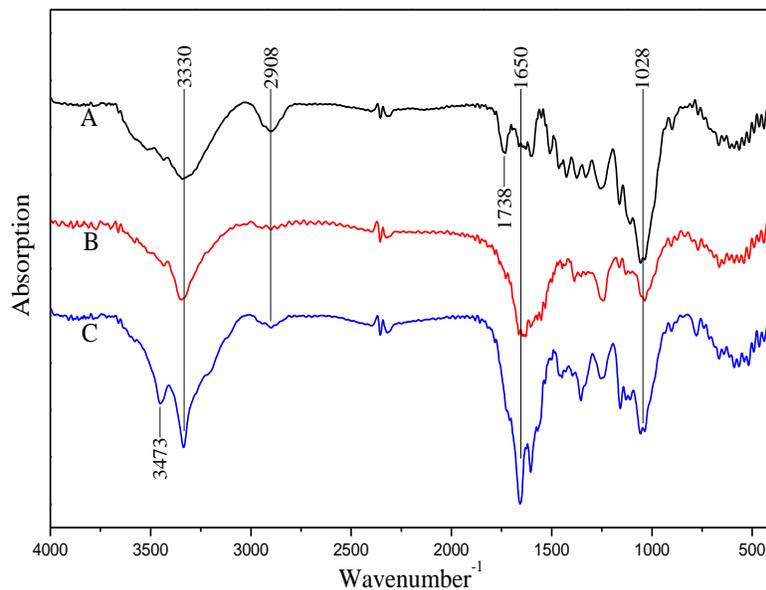


Fig. 3. The FT-IR curves of the wood: (A) untreated control, (B) methylolurea treated wood, (C) urea-methylolurea treated wood

SEM Analysis

To have an idea of the morphology after compression, the untreated and treated wood was scanned with scanning electron microscopy (SEM). The SEM images give a clear view of the anatomical characteristics of the transverse section in Fig. 4A, 4B, and 4C. With tissue types such as diffuse porous hardwood (Fig. 4A) with little difference in density between earlywood and latewood, the characteristic large vessels of poplar can easily be distinguished on the images. For the treated wood in Fig. 4B and 4C, the high strain in the hot-press process drastically reduced the volume of void spaces in the specimens and deformed the cell lumens (particularly evident in the vessels). The cells were deformed without any fracture of the cell walls, while the rays appeared to buckle. Since the degree of densification between methylolurea treated wood and urea-methylolurea treated wood were almost the same, the morphology differences between them after the hot-press process could not be distinguished.

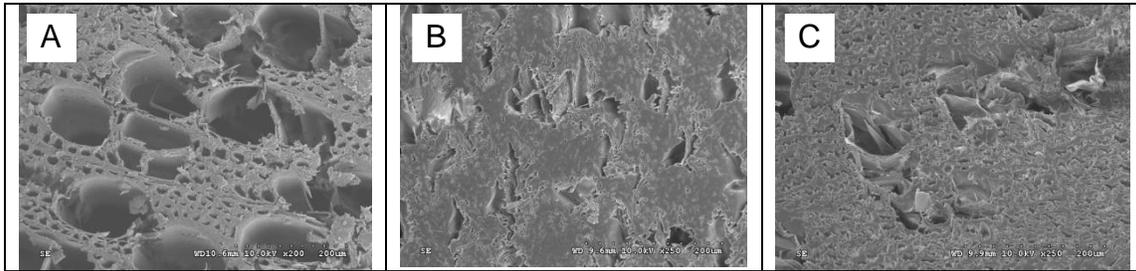


Fig. 4. SEM micrographs of untreated and treated wood: (A) untreated control, (B) methylolurea treated wood, (C) urea–methylolurea treated wood

CONCLUSIONS

1. Chemical impregnation and the hot-press drying process can enhance the density and mechanical properties of treated wood and also influence the set recovery of compressive deformation.
2. Adding extra urea can soften wood microfibril, which reduces the buildup of internal stresses during hot pressing and residual stresses after drying, but leads to a slight decrease in mechanical properties.
3. Chemical bonding was produced between methylolurea and wood functional groups during the drying process; however, the structure of cellulose was not noticeably affected.
4. High strain in the hot-press drying process drastically reduced the volume of void spaces in the specimens and deformed the cell lumens.

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