

Characterization of *Ailanthus altissima* Veneer Modified by Urea-formaldehyde Pre-polymer with Compression Drying

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Recently, *Ailanthus altissima* wood has received increased attention in China. Although *Ailanthus altissima* has a number of advantages, such as fast-growing character and high density, the low dimensional stability is a restraining factor. Chemical modification could be a feasible way to overcome such drawbacks. The aim of this research was to study the physical and chemical properties of *Ailanthus altissima* veneers treated by urea-formaldehyde pre-polymer impregnation and compression drying. The results showed that this approach not only significantly increased the dimensional stability of wood, but also enhanced the bending strength and compressive strength parallel to the grain. The FT-IR analysis showed that the intensity of hydroxyl (-OH) and carbonyl (C=O) absorption peaks decreased, which indicated that the NH-CH₂-OH of the pre-polymer reacted with the wood's carboxyl and hydroxyl groups. The positions of the XRD peaks did not change, which indicated that the structure of cellulose crystallinity was not noticeably affected by the chemical modification. The TGA showed that the thermal stability of modified wood was improved, while FESEM showed that the wood cell wall and vessels were impregnated with chemicals.

Keywords: Urea-formaldehyde pre-polymer; Compression drying; *Ailanthus altissima* wood; Impregnation; Chemical modification

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INTRODUCTION

Wood is a natural renewable biomass material with many useful properties, such as a high strength to weight ratio, the ability to adjust humidity with the environment, and heat and sound insulation capabilities (Liu *et al.* 2010). However, it has some troublesome inherent properties, such as changes in dimension with time due to environmental modulation and degradation due to insect attack, which results in loss of strength properties (Stamm 1964; Rowell and Ellis 1978; Rowell 1983; Schneider and Brebner 1985; Yun *et al.* 1991; Norimoto *et al.* 1992; Narahara and Fujii 1993).

China is a major consumer of wood and wood-product material in the world, but China is also a relatively poor nation when it comes to forest resources. To decrease the shortage of forest resources, the growth of fast-growing wood species is an important strategy. *Ailanthus altissima*, a fast-growing tree species, is widely used by the local people in northern China for architecture, transportation, furniture, and the like, due to its availability.

Despite the fact that fast-growing wood species such as *Ailanthus altissima* have many advantages, they have some disadvantages, such as high swelling and low dimensional stability. Hence, improving the performance of *Ailanthus altissima* to meet the requirements for more applications is necessary.

Wood modification is a promising method for the utilization of fast-growing wood. It can enhance the properties of fast-growing wood, such as dimensional stability, hardness, and biological stability. Methods for wood modification can be roughly classified into chemical and physical routes. Most methods, however, are primarily aimed at filling the cell wall micropores, because most wood properties can only be efficiently changed with the material located in the cell wall, rather than in the lumens (Klüppel and Mai 2013). Chemical modification is designed to substitute hydrophobic chemical groups for the OH⁻ groups by etherification, esterification, oxidation, silylation, or other chemical reactions (Militz *et al.* 1997). Chemicals used for this purpose include phenol-formaldehyde resin, difunctional aldehydes, anhydrides, acid chlorides, isocyanates, formaldehyde, acetaldehyde, chloral, alkyl chlorides, 3-propiolactone, acrylonitrile, and epoxides (Kumar 1994). Some chemical reactions with the hydroxyl groups of wood cell walls are required in the chemical modification. Therefore, the chemical components of the cell wall (lignin, cellulose, and hemicellulose) are altered and covalent bonds are formed (Rozman *et al.* 1997).

The chemical modification of wood has been the subject of research for many decades (Stamm and Tarkow 1947; Rowell 1983; Banks and Lawther 1994; Kumar 1994). Baysal *et al.* (2004) pointed out that treating Japanese cedar and Scots pine with furfuryl alcohol catalyzed by borates can improve the dimensional stabilization. The results confirmed that chemical reactions occurred in the cell walls, and the antismelling efficiency for both Japanese cedar and Scots pine wood were improved by 85%. Shi *et al.* (2007) showed that urea-formaldehyde resin with nano-SiO₂ can improve the general properties of poplar wood. All of the modifiers used in their study reduced the water absorption of poplar wood and enhanced flame resistance and hardness. Nano-SiO₂ also improved the hardness of the wood. Khalil *et al.* (2011) noted that propionic- and succinic anhydride-modified *Acacia mangium* wood demonstrates improved thermal stability. The better thermal stability of *Acacia* wood modified with anhydride was attributed to a decrease in the hydrophilic nature of the wood.

Compression drying or modification is a method in which wood boards are mechanically compressed between heated metal plates during the drying process (Mikkola and Korhonen 2013). Because of the pressure, the change in wood thickness is greater than it is with the normal drying method. Therefore, compression drying can improve surface hardness, basic density, and other performance of the wood. Heräjärvi (2009) reported the effects of treatment with compression drying on European and hybrid aspen wood. The results showed that the hygroscopicity of compression-dried specimens was significantly decreased. Compression drying has been shown to decrease drying time and warp remarkably.

The aim of this study was to investigate the influence of modification by urea-formaldehyde pre-polymer with compression-drying on *Ailanthus altissima* veneers. The physical and chemical performance of natural and modified wood was evaluated, including the thermal stability, dimensional stability, group reactivity, and microstructure.

EXPERIMENTAL

Materials

The fresh *Ailanthus altissima* wood used in this study was harvested from Pingquan, China. Logs were sawn into boards about 1.5 m in length and 0.2 m in width and air-dried to about 20% moisture content.

Methods

Preparation of urea-formaldehyde pre-polymer

Urea, formaldehyde, and ammonia were added to a three-necked flask at a set molar ratio. The reaction mixture was stirred and warmed at 35 °C for 3 h. Then, sodium hydroxide or hydrochloric acid was used to adjust the pH of the pre-polymer to 5-7.

Vacuum impregnation and compression drying

A vacuum impregnating tank was used to treat the wood veneer. *Ailanthus altissima* wood was impregnated with the urea-formaldehyde pre-polymer (15% w/w), carbamide (25% w/w), and water (60% w/w) at 0.9 to 1.1 MPa for 3 h. After impregnation, it took about one day to be air-dried. After that, the veneer was compression-dried using a hot-press machine (BY214×8/12(5) H₁R, Qiulin, China). The pressure was about 3 MPa, and the highest temperature was 130 °C. The procedure took about 5 h.

Mechanical properties and dimensional stability

The raw and modified wood timber was cut to appropriate dimensions. Ten specimens of raw and modified were analyzed in each test. And a universal mechanical testing machine (MWW-50, Ruipu, China) was used in tests of mechanical properties. Tests were carried out on the basis of GB/T 1933 (2009), GB/T 1935 (2009), GB/T 1936.1 (2009), GB/T 1938 (2009), GB/T 1934.2 (2009), and GB/T 1932 (2009). Furthermore, the specimens for GB/T 1938 (2009) and GB/T 1936.1 (2009) with a dimension of 300 mm × 20 mm × 20 mm, for GB/T 1935 (2009) with a dimension of 30 mm × 20 mm × 20 mm. The other tests were carried out with specimens of 20 mm × 20 mm × 20 mm. The average data of ten specimens for each kind of measurements were recorded.

Hygroscopicity

Raw and modified wood veneers were cut into specimen cubes with sizes of 20 mm × 20 mm × 20 mm according to GB/T 1934.1 (2009). The specimens were dried in an oven at 103 °C until the average quantity change was less than 0.02 g every 2 h (Chen *et al.* 2014). Then, the specimens were soaked in water for 15 days. The water uptake improved with specimen immersion time. After immersion, the excess water on the surface of the specimens was removed by a soft cloth and the weights of the specimens were immediately recorded. The amount of absorbed water was calculated according to the following formula,

$$A(\%) = \left(\frac{m_1 - m_2}{m_2} \right) \times 100 \quad (1)$$

where m_1 is the measured weight and m_2 is the original weight (g).

Powder X-ray diffraction

Both raw and modified wood samples were ground into sawdust (60 to 80 mesh). The changes in the crystallinity of the raw and modified wood samples were analyzed using a Shimadzu (Kyoto, Japan) powder X-ray diffractometer (Model XRD 6000) with Cu- α radiation ($\lambda = 0.15406$ nm) at 30 kV and 40 mA. The samples were put on an aluminum holder (25 mm in diameter) and observed under the continuous scanning mode (Xing *et al.* 2010). The patterns were detected in the range of $2\theta = 10$ to 50° with a step size of 0.05° and scan rate of $2^\circ/\text{min}^{-1}$. The crystallinity was evaluated by the total diffracted area and the area under the crystalline peaks. The degree of crystallinity was evaluated as the ratio of the intensity differences in the peak positions. The ratio of the crystallinity of samples to the standard amorphous material was taken as the relative crystallinity, represented as follows,

$$C_r = F_c / (F_a + F_c) \times 100\% \quad (2)$$

where C_r is the relative crystallinity and F_a and F_c are the areas of the crystal and noncrystalline regions, respectively (Sun *et al.* 2008).

Fourier transform infrared (FT-IR) spectroscopy

The chemical composition of the raw and modified specimens was detected by Fourier transform infrared spectroscopy (FT-IR) (Tensor 27, Bruker, Germany). The specimens of 120 mesh size were used in the FT-IR measurement. For the FT-IR analysis, specimens of 120-mesh size were prepared. The spectra 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} .

Thermogravimetric analysis (TGA)

The thermal properties of the raw and modified samples were measured using a thermogravimetric analyzer (DTG-60, Shimadzu, Japan). The sample pan was placed on a Pt basket in the furnace and heated from room temperature to 600 $^\circ\text{C}$. The heating rate was 10 $^\circ\text{C min}^{-1}$; $\alpha\text{-Al}_2\text{O}_3$ was used as the reference material. During testing, the heating unit was flushed under a continuous nitrogen flow at a pressure of 8 kPa. The powder of wood samples (5 to 10 mg) was put into the platinum crucible with the diameter of 8 mm.

Field emission scanning electron microscope (FESEM) analysis

The impact of modification on the surface morphologies was examined using a FESEM (SU-8020, Hitachi, Japan). Samples containing prepared transverse and radial faces to be observed under the FESEM were mounted on aluminum stubs with conductive adhesive tape. The following are the working conditions: accelerating voltage of 1 kV; working distance of 20 mm; and illuminating current of 0.7 nA.

RESULTS AND DISCUSSION

Mechanical Properties and Dimensional Stability

The mechanical and physical properties of specimens are shown in Table 1. Density is one of the most significant parameters of fast-growing wood. Table 1 shows that the basic density of modified wood increased by 3.33% compared with the raw wood samples, and the oven-dried density of modified wood increased by 5.96% . The bending

strength, tensile strength parallel to the grain, and compressive strength parallel to the grain increased by 8.96%, 14.92%, and 32.50%, respectively, compared with the raw wood samples. The improved mechanical properties can be explained by impregnation of urea-formaldehyde pre-polymer and compression drying.

Table 1. Mechanical Properties of Modified and Raw Wood Samples

Properties	Modified	Raw	Improvement (%)
Basic density (g·cm ⁻¹) (SD)	0.651 (0.04)	0.630 (0.05)	3.33
Oven-dried density (g·cm ⁻¹) (SD)	0.676 (0.07)	0.638 (0.08)	5.96
Bending strength (MPa) (SD)	148.3 (8.17)	136.1 (7.73)	8.96
Tensile strength parallel to grain (MPa) (SD)	121.7 (3.34)	105.9 (2.97)	14.92
Compressive strength parallel to grain (MPa) (SD)	110.9 (2.59)	83.7 (2.26)	32.50

Radial, tangential, and volume swelling of 14.46%, 10.09%, and 10.55%, respectively, were obtained by treatment with impregnation, as shown in Table 2. The shrinkage also improved by 15.49%, 15.00%, and 14.91%, respectively. Compared with the raw samples, even though the impregnation of pre-polymer into wood cells did not substantially reduce the equilibrium moisture content of wood, the cell wall bulking due to pre-polymer deposition did help the cell wall become more hydrophobic.

Table 2. Dimensional Stability of Modified and Raw Wood Samples

Properties	Modified (%)	Raw (%)	Improvement (%)
Swelling of radial (SD)	7.1 (0.44)	8.3 (0.57)	14.46
Swelling of tangential (SD)	9.8 (0.61)	10.9 (0.81)	10.09
Swelling of volume (SD)	17.8 (1.21)	19.9 (1.36)	10.55
Shrinkage of radial (SD)	6.0 (0.39)	7.1 (0.45)	15.49
Shrinkage of tangential (SD)	6.8 (0.43)	8.0 (0.55)	15.00
Shrinkage of volume (SD)	13.7 (0.98)	16.2 (1.25)	14.91

Hygroscopicity

Figure 1 represents the hygroscopicity of raw and modified wood samples. In both raw and modified samples, water absorption increased with increasing immersion time. However, the *Ailanthus altissima* wood became more repellent after chemical modification. As has been stated earlier, hygroscopicity is one of the most indicative characteristics of wood and has a major influence on both dimensional stability and durability (Akyildiz *et al.* 2009). Raw samples absorbed more water than modified samples. The hygroscopicity of both raw and modified wood increased quickly within 24 h. At the end of 15 days, the value for the raw wood was 88.0%, while that for the modified wood was 110.0%. The modified wood had a lower hygroscopicity than the raw

wood because of the chemical reactions that occurred between the functional groups of wood and the modifier, which led to a decrease in hydroxyl groups in the wood. The larger polymers remaining in the lumen may form a barrier on the lumen surface and decrease the water absorption to some extent. According to research conducted by Heräjärvi (2009), the water absorption of compression-dried specimens will have a significantly slower pace than that of conventionally dried specimens.

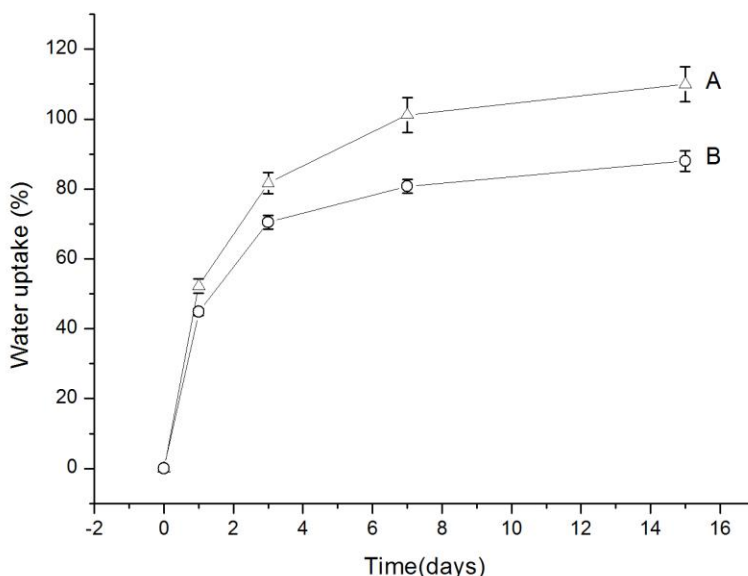


Fig. 1. Hygroscopicity curves of wood; (A) raw wood, (B) modified wood

Fourier Transform Infrared Spectroscopy Analysis

In Fig. 2, the FT-IR spectrum of raw wood is characterized by the absorption bands (shown in curve B) appearing at 3400 cm^{-1} (OH stretching), 1738 cm^{-1} (C=O stretching of acetylated xylem), and 1261 cm^{-1} (C-O stretching of acetyl group) (Yap *et al.* 1991). The vibrational band of modified wood is shown at 3430 cm^{-1} for the hydroxyl groups, while the characteristic band of raw wood is at 3394 cm^{-1} . This may be attributed to intramolecular hydrogen bonding and -OH bending (1040 cm^{-1}). After the compression-drying process, the treated veneer showed a decline of -OH absorbance at 3430 , suggesting that hygroscopicity decreased. The absorption peak at 1669 cm^{-1} was indicative of amide (N-C=O) from urea-formaldehyde. This was due to the bond between the $\text{-NHCH}_2\text{OH}$ functional groups of pre-polymer and the wood carboxyl C=O. Furthermore, the peak at 1463 cm^{-1} , which may be assigned to C-H deformation (methyl and methylene), shows an increase in the case of urea-formaldehyde pre-polymer-treated samples relative to untreated samples. The appearance of the peak around 1619 cm^{-1} is because of aromatic skeletal vibration and carbonyl stretching.

Such changes could be explained by well-known reactions in wood chemistry. As wood temperature increases in the drying process, chemical bonding is produced between wood and methylation as a result of chemical reactions between wood and pre-polymer (Wu *et al.* 2012). The acetic acid separated from the hemicelluloses, which was further catalyzed with urea-formaldehyde pre-polymer, causing the degree of polymerization to increase between the urea-formaldehyde pre-polymer and wood (Lang *et al.* 2013).

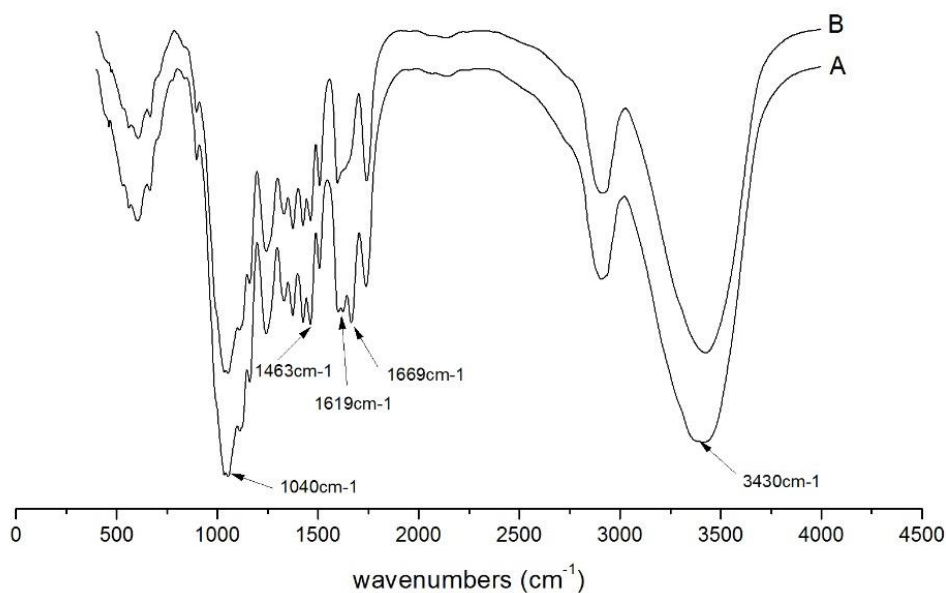


Fig. 2. FT-IR curves of wood; (A) modified wood, (B) raw wood

X-Ray Diffraction Analysis

X-ray diffraction is a method generally used to evaluate the degree of crystallinity of a wide range of materials (Poletto *et al.* 2012). Among wood components, only cellulose is crystalline, whereas hemicellulose and lignin are non-crystalline (John and Thomas 2008). The XRD spectra for untreated and treated specimens are shown in Fig. 3.

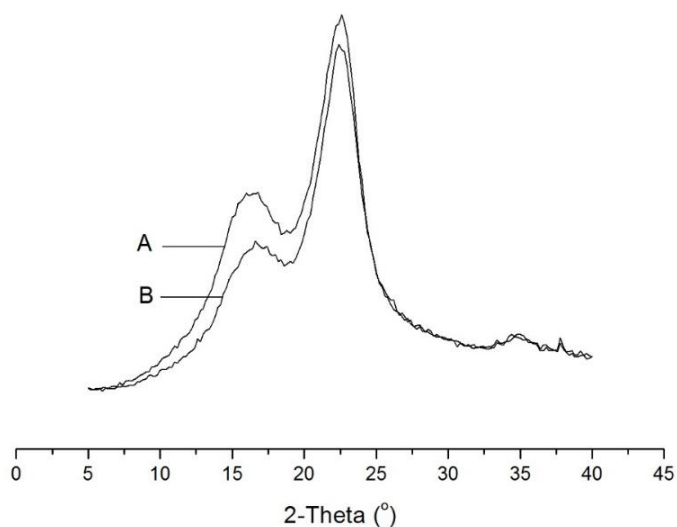


Fig. 3. X-ray diffraction curves of wood; (A) modified wood, (B) raw wood

Generally, maxima were found at 17.5°, 22.5°, and 35.5°, corresponding to the cellulose crystal plane diffraction peaks of (101), (002), and (040), respectively. The position of these peaks did not change after modification, which indicated that the main structure of cellulose was not noticeably influenced by the modification. Meanwhile, crystallinity of the modified wood had increased after the treatment compared to the raw wood, from 42.19% to 44.98%. The free hydroxyl groups present in the cellulose macromolecules are likely to be involved in a number of intramolecular and intermolecular hydrogen bonds, which may give a rise to various ordered crystalline arrangements (Poletto *et al.* 2011; Popescu *et al.* 2011). The increased crystallinity of modified samples may be caused by the urea-formaldehyde pre-polymer, which filled in the microfibrils of the cell wall and reacted with free hydroxyl. Another explanation is the decrease in hemicelluloses during the compression-drying process.

Thermogravimetric Analysis

Figure 4 shows the TG analysis curves for the raw and modified wood samples. The mass loss at 150 °C was about 4%, which was due to the dehydration of wood powder, and further degradation occurred as a three-step process. In the first step, the mass loss of natural and treated woods was 58% in the temperature range of 150 to 320 °C, associated with the degradation of hemicellulose, lignin, and urea-formaldehyde pre-polymer during the heat-treatment process. However, the thermal degradation of modified wood was slower than that of natural wood; the crosslinking reaction with urea-formaldehyde pre-polymer enhanced the thermal stability of wood. In the second step, 73% of wood was degraded because of oxidative decomposition. For this step, the oxidative decomposition of treated and untreated wood was similar. From the thermal analysis curves of wood, it was evident that the oxidative decomposition of wood occurred at higher temperatures and the heat release was distributed between two sharp and closely spaced isotherms, which indicated a high rate of heat release. Therefore, at higher temperatures, the increase in the thermo-oxidative stability of fast-growing wood can be attributed to the wood modifier, which was impregnated into the inner structure of wood and caused a crosslinking reaction.

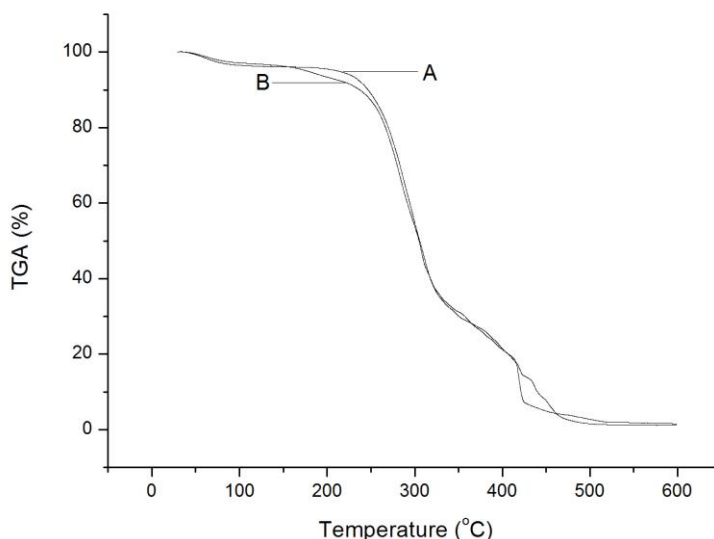


Fig. 4. Thermogravimetric analysis curves of wood; (A) modified wood, (B) raw wood

Field Emission Scanning Electron Microscopy

High-resolution scanning electron micrographs of raw and modified wood surfaces are shown in Fig. 5. Figure 5a shows that the wood is a heterogeneous and porous material (Wålinder and Johansson 2001). Figure 5b shows the obvious precipitate on the surface of the modified wood. The wood fiber and vessels were decorated with particles after the impregnation. In addition, the pores, macropores, and other multilevel pore structures were also infiltrated with the pre-polymer. Figures 5c and 5d show the radial sections of the unmodified and modified wood, respectively. Compared to the unmodified wood, it can be seen that many granular materials are distributed in the lumen. The surface of the modified wood was smoother (Wu *et al.* 2012) because the surface of raw wood had pores and voids. In the modified wood surface, the pores and voids were closed because of the impregnation with the pre-polymer. Furthermore, the urea-formaldehyde pre-polymer not only filled the lumens, but also impregnated the cell walls.

The chemical can become permeated into the wood fiber and other vertical cells under the applied pressure. The chemical particles became rod-like structures or discontinuously distributed in the wood cell. The SEM analysis indicated that the pre-polymer was impregnated into cell wall and cell cavities of the wood.

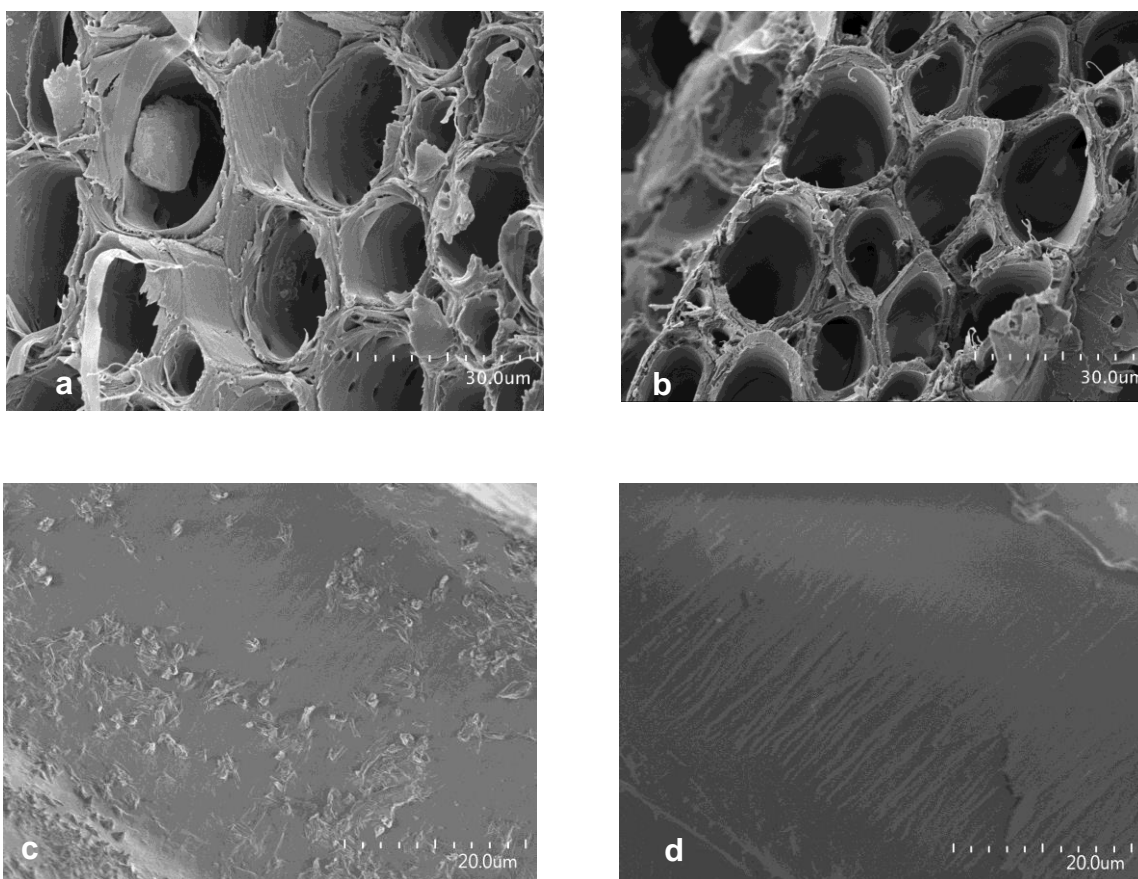


Fig. 5. FESEM micrographs of modified and raw wood; (a) transection of modified wood, (b) transection of raw wood, (c) radial section of modified wood, (d) radial section of raw wood

CONCLUSIONS

1. *Ailanthus altissima* veneers modified by urea-formaldehyde pre-polymer and compression-drying can improve its properties, such as the dimensional stability, the bending strength, and the tensile strength parallel to the grain. Meanwhile, the hygroscopicity of wood was reduced significantly.
2. The FT-IR analysis showed that the intensity of hydroxyl and carbonyl absorption peaks decreased because the NHCH_2OH of pre-polymer reacted with the wood carboxyl ($\text{C}=\text{O}$) and hydroxyl ($-\text{OH}$) groups.
3. In the XRD spectrum, the positions of the peaks did not change according to the wood crystalline phases and were revealed at 17.5° , 22.5° , and 35.5° . Hence, the structure of cellulose did not change in comparison with the untreated wood.
4. The TGA showed that the thermal stability of heat-treated wood was enhanced. FESEM analysis indicated that the pre-polymer was impregnated into the cell walls and the cell lumens of the wood.

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