Assessment of the Performance of Furfurylated Wood and Acetylated Wood: Comparison among Four Fast-Growing Wood Species

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Four fast-growing wood species were treated, including two hardwood species and two softwood species, with either furfurylation or acetylation for comparison and analysis. The properties of the resultant woods, including weight percent gain, bulking effect, leach rate, anti-swelling efficiency (ASE), and color changes, were compared comprehensively. The effects of wood species on modification efficiency were also evaluated by morphological analysis. The results indicated that the species of wood had little effect on successful acetylation, but that wood species with more open pits and loose and ordered structures were best suited for furfurylation. Both types of modification resulted in wood samples with more uniform colors than untreated samples. Furfurylation caused considerable color changes in all of the wood samples; acetylation resulted in wood samples slightly lighter in color (lower Δ*E** values). The differences in Δ*E** values among the four wood species were primarily due to the natural differences in the color of the woods.

Keywords: Wood modification; Furfurylation; Acetylation; Color; Fast-growing wood

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INTRODUCTION

Modification is an efficient approach for improving wood properties such as dimensional stability, water resistance, and durability (Hill 2006). Several different wood modification technologies have been successfully demonstrated over the past few decades, which can be roughly divided into three categories: chemical modification, impregnation, and thermal modification (Rowell 2012). Although many new modification methods have emerged recently, their commercial availability remains limited. Among these, acetylation and furfurylation are particularly promising (Lande *et al*. 2008; Cetin *et al.* 2011).

Wood acetylation, first proposed in 1947, uses acetic anhydride as a modifier to result in substitution of the accessible hydroxyl groups in the cell walls by acetyl function (Fig. 1a). Many different acetylation processes have been developed involving liquid or vapor systems (Fadl and Basta 2005), catalysts (Hill *et al.* 2000; Cetin *et al*. 2011), and solvents (Li *et al*. 2000). Acetylation has been shown to efficiently improve the dimensional stability and water resistance of wood (Li *et al*. 2009). Wood furfurylation also has a lengthy research history after it was first reported by Stamm in the early 1950s (Lande *et al*. 2008). The process was developed rapidly in the 1990s when a new effective catalyst system was established. Furfurylation is based on wood impregnation with furfuryl

alcohol and other agents, followed by *in-situ* polymerization at an elevated temperature (Fig. 1b). The resultant furfurylated wood products exhibit favorable qualities such as high dimensional stability water resistance, and surface hardness (Esteves *et al*. 2011; Dong *et al.* 2015). The effects of successful modification can be mostly attributed to cell wall bulking.

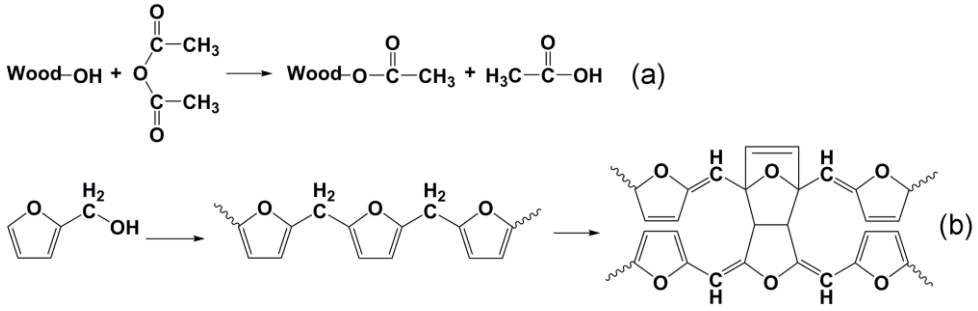

Fig. 1. The main chemical reactions of (a) acetylation and (b) furfurylation

Fast-growing wood species pose an alternative approach for the wood product industry as far as extending the global wood supply and protecting natural resources from over-exploitation. Unfortunately, fast-growing wood species possess many disadvantages compared with other types of wood, including low density, dimensional instability, and poor durability, which severely restricts the application of these resources (Yan *et al*. 2014).

Furfurylation and acetylation modification processes have been studied extensively in an effort to secure high-performing, sustainable wood material (Mohebby *et al*. 2007; Li *et al*. 2009; Herold *et al*. 2013). Because their modification mechanisms differ, these processes have somewhat discrepant effects. For example, furfurylation causes wood to darken in color (Dong *et al*. 2014). Although the mechanism responsible for the progressive coloration has been suggested (Choura *et al*. 1996), there have been few studies to focus specifically on evaluating color changes in furfurylated wood. The water sorption and mechanical properties of furfurylated or acetylated wood are also quite different (Thygesen *et al*. 2010; Xie *et al*. 2013). The effects of modification also vary depending on which species of wood undergoes the process; the factors involved include chemical composition, anatomical structure, and density (Lande *et al*. 2010). Different wood species showed significant differences in the volume occupied by the acetyl group (Kwon *et al*. 2007). Scots pine wood enters the reaction with succinic anhydride more easily than beech wood (Doczekalska *et al*. 2007). However, the studies that provided this information did not reveal the relevant mechanisms.

In this study, four fast-growing wood species were selected and treated with furfurylation and acetylation. The objectives of the experiment were to investigate the efficiency of these two modification methods by comparing the physical properties (*e.g.*, weight percent gain, bulking effect, leach rate, and anti-swelling efficiency) and color changes of different fast-growing wood species. Wood sample morphologies were also evaluated by light microscopy and field emission scanning electron microscopy to analyze the effects of differing species on the properties of modified wood.

EXPERIMENTAL

Materials

Four fast-growing wood species, *i.e.*, poplar (*Populus tomentosa* Carr.), Chinese fir (*Cunninghamia lanceolata*), eucalyptus (*Eucalyptus robusta* Smith), and Masson pine (*Pinus massoniana*), were cut into samples with dimensions of 20 (R) × 50 (T) × 50 (L) mm. The samples were first immersed in ethanol-benzene (1:2, v/v %) for seven days and then immersed in boiling water for another 12 h. The extracted samples were then dried at 105 °C until reaching constant weight; then, the oven-dried weights and sizes of samples were measured. Thirty samples of each wood species were chosen and each group (control, acetylation, and 3 groups of furfurylation) has 6 replicates.

Acetic anhydride (chemical grade 98.5%, Beijing Chemical Works, China), furfuryl alcohol (chemical grade 98.0%; Sinopharm Chemical Reagent Co., Ltd., China), maleic anhydride and disodium tetraborate (analytical grade, Beijing Chemical Works, China) were used as supplied without further purification.

Methods

Wood modification methods

Acetylation was performed according to Li's method (Li *et al*. 2000). The wood samples were immersed in acetic anhydride under vacuum conditions (*ca*. 0.095 MPa) for 30 min and then soaked under atmospheric pressure for 12 h. The impregnated samples then were wrapped with aluminum foil and oven-cured at 120 °C for 4 h. The foil was removed, and the samples were oven-dried at 105 °C until reaching a constant weight.

Furfurylation was performed according to Dong's method (Dong *et al*. 2014). First, 30, 50, and 70 wt.% furfuryl alcohol water solutions were prepared containing 2.0 wt.% maleic anhydride and 4.0 wt.% disodium tetraborate. The impregnation process was the same as that for acetylation, apart from polymerization performed at 100 °C for 12 h.

After treatment, the weights and dimensions of all samples were measured, and the weight percent gain (WPG) and bulking effect (BE) were calculated accordingly,

$$\text{WPG (\%)} = (W_1\text{-}W_0)/W_0 \times 100 \quad (1)$$

$$\text{BE (\%)} = (V_1\text{-}V_0)/V_0) \times 100 \quad (2)$$

where W_0 and W_1 are the oven-dried weights of a sample before and after treatment, respectively. V_0 and V_1 are the oven-dried volumes of a sample before and after treatment, respectively.

Physical properties

The samples were immersed in distilled water for 72 h at room temperature, and then the dimensions of the water-swollen samples were measured. The samples were then oven-dried at 105 °C until weight consistency, and the dimensions were measured again. The swelling coefficient (S) and anti-swelling efficiency (ASE) of each sample was calculated as follows,

$$S\ (\%) = (V_2\text{-}V_1)/V_1 \times 100 \quad (3)$$

$$\text{ASE (\%)} = (S_u\text{-}S_t)/\ S_u \times 100 \quad (4)$$

where V_2 is the volume of sample after 72 h immersion. S_u is the swelling coefficient of an untreated sample and S_t is that of a treated sample.

Samples were then immersed in distilled water for 20 d, after which the excess water on the surface was removed by a soft cloth and the weights of the samples were measured to calculate their water uptake (WU). Then the samples were weighed after oven-drying at 105 °C,

$$WU\ (\%) = (W_2\text{-}W_1)/W_1 \times 100 \quad (5)$$

$$L\ (\%) = (W_1\text{-}W_3)/(W_1\text{-}W_0) \times 100 \quad (6)$$

where W_2 and W_3 are the weight and oven-dried weights of a wood sample after 20 d water immersion, respectively.

Color change measurement

The color of the tangential sections of samples before and after treatment was examined with a color measuring instrument (Dataflash 110 Datacolor, USA) according to the CIE Lab color system, where L^* is a measure of lightness, a^* is the chroma from green to red, and b^* is the chroma from blue to yellow (Aydemir *et al*. 2012). Three samples were used for each treatment, and three repetition areas were examined for each sample. To avoid uneven distribution on the surface, all samples were planed prior to measurement. The color difference of each sample was calculated according to the following equation,

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (7)$$

where ΔE^* is the color difference, ΔL^* is the lightness difference, and Δa^* and Δb^* are the chroma differences.

FE-SEM and light microscopy

The morphologies of untreated and treated wood samples were characterized by field emission scanning electron microscopy (FE-SEM). The interior portions of the tangential and cross planes were exposed by cutting with a surgical blade, then mounted on conductive adhesives, gold-sputter-coated, and observed with a Hitachi SU8010 instrument (Japan) at 500× and 1000× magnification and 5 kV accelerating voltage. In addition, sections cut transversely through wood samples at a thickness of 30 μm (Leica RM2255, Germany) were stained using safranin and photographed with a digital microscope (Olympus BX53, USA).

RESULTS AND DISCUSSION

Assessment of Physical Properties

Detailed data regarding the physical properties (*e.g.*, WPG, LR, ASE, BE, *S*, and WU) of the four wood species before and after modification are shown in Table 1. The WPG values of each furfurylated wood species increased linearly as the concentration of furfuryl alcohol increased. Poplar and Chinese fir exhibited higher WPG than eucalyptus or pine, and Chinese fir exhibited the lowest leach rate of all the species. In addition, a gentle increase of WPG for pine was indicated. For acetylation, the WPG of the four treated species was close, which agreed with the study by Li *et al.* (2000). Most of the leach rates for all modification were under 8.0%.

For furfurylation, eucalyptus showed a linear increase in BE as furfuryl alcohol concentration increased, possibly because of the increased WPG. The BE of Chinese fir

and pine did not increase noticeably as WPG increased, however. This phenomenon was likely related to BE maximization at the initial 30% to 50% concentration, as confirmed according to the swelling coefficient (*S*) of the control. Acetylated wood showed relatively higher BE values than furfurylated wood, especially pine. The wet curing process of furfurylation reduces BE because the poly(furfuryl alcohol) precipitates before drying (Klüppel and Mai 2013). This difference confirmed that the modification mechanisms between the furfurylation and acetylation are distinct.

Table 1. Physical Properties of Wood Samples

Sample[a]	WPG[b] (%)	BE (%)	S (%)	ASE (%)	LR (%)	WU (%)
Poplar						
control	—	—	9.02(0.31)	—	—	146.78(0.32)
Acetylation	18.66(0.31)[C]	6.79(0.52)	3.97(0.18)	55.97(3.50)	4.77(0.53)	90.59(5.75)
30%	80.93(1.78)	3.62(1.20)	5.76(0.92)	36.17(1.29)	7.91(0.22)	54.92(2.89)
50%	108.55(10.04)	7.07(1.49)	4.43(1.03)	50.87(6.88)	6.47(0.61)	35.45(2.94)
70%	135.05(3.28)	8.21(0.71)	3.99(0.57)	55.81(3.49)	6.01(0.32)	26.84(1.89)
Eucalyptus						
control	—	—	9.57(0.13)	—	—	91.58(3.81)
Acetylation	21.60(1.22)	8.26(0.38)	3.10(0.04)	67.56(3.72)	10.75(2.5)	62.33(16.59)
30%	38.89(2.68)	3.02(0.17)	6.50(1.22)	32.11(5.35)	5.86(1.04)	50.32(1.88)
50%	62.68(12.04)	3.56(0.61)	5.81(0.14)	39.27(5.44)	5.96(0.34)	35.94(2.49)
70%	81.99(15.31)	5.29(1.42)	4.59(0.12)	52.07(4.40)	6.44(0.94)	30.38(4.19)
Chinese fir						
Control	—	—	6.69(0.49)	—	—	100.05(17.66)
Acetylation	24.83(1.78)	7.45(0.66)	2.24(0.11)	70.90(4.24)	7.50(2.73)	56.15(0.14)
30%	76.32(4.32)	7.61(0.21)	3.99(0.29)	48.11(3.20)	4.35(0.53)	56.52(1.37)
50%	107.75(17.53)	7.38(1.29)	3.44(0.37)	55.25(1.26)	3.36(0.09)	43.42(2.69)
70%	137.49(23.90)	8.43(1.38)	3.26(0.04)	57.59(2.86)	2.63(0.30)	34.32(1.95)
Pine						
Control	—	—	12.90(0.42)	—	—	101.42(2.00)
Acetylation	23.01(0.27)	11.81(0.52)	4.50(0.11)	65.13(1.00)	6.28(0.57)	64.06(0.99)
30%	35.92(1.54)	8.19(0.51)	8.11(1.37)	37.09(2.75)	9.34(0.37)	78.47(6.37)
50%	49.44(2.54)	10.68(0.83)	6.87(1.40)	46.69(2.75)	7.64(0.35)	71.62(10.14)
70%	55.63(4.12)	10.02(1.08)	5.75(1.24)	55.41(3.61)	7.57(0.22)	67.75(2.58)

[a] "30%", "50%", and "70%" refer to furfuryl alcohol concentrations.
[b] WPG = weight percent gain; BE = bulking effect; S = swelling coefficient; ASE = anti-swelling efficiency; LR = leach rate; WU = water uptake.
[c] The values in brackets refer to standard deviation.

The ASE results are also shown in Table 1. For all four species, acetylation with relatively lower WPG had a favorable effect on dimensional stability, suggesting that the species does not considerably impact acetylation. Furfurylated samples, however, showed distinct ASE values either inter-group or intra-group. All samples showed increased ASE values as furfuryl alcohol concentration increased, which was related to the increased polymer filling. A higher WPG value indicates higher cell wall bulking and increased cell lumens, which form a barrier and reduce water absorption, resulting in enhanced dimensional stability. The sample with the lowest leach rate was Chinese fir, which had the highest ASE value as a result of efficient cell wall bulking, even after drying (Lande *et al*. 2004a); therefore, changes in volumetric dimensions were relatively small. The ASE values tended to be fairly constant regardless of furfuryl alcohol concentration, although poplar and eucalyptus (hardwood) exhibited different ASE values at the same furfuryl

alcohol concentration, as did Chinese fir and pine (softwood), which was possibly related to natural differences in structure and density.

Differences in water uptake during 20 days of water immersion among the four wood species are also shown in Table 1. Compared with the control samples, acetylated and furfurylated wood showed lower water uptake capabilities. Although the ASE of acetylated wood was higher than that of furfurylated wood, acetylation gave rise to higher water uptake capability overall. This phenomenon also elucidates the difference in mechanism between acetylation and furfurylation. The water uptake of furfurylated wood decreased as WPG increased because of the bulking effect of the polymer in the wood.

Color Changes

Color is one of the most important properties relative to the value of wood products, as it is an important aesthetic component (Usta 2007). Wood modification can change the color of wood in different degree. Generally, furfurylated wood has a noticeably darker color than acetylated wood, but this experiment showed that color comparisons among wood species in relation to either type of modification were fairly subtle. Color change comparisons are shown in Fig. 2. According to the results obtained, acetylated wood induced indistinct modification in all color components. The *L** values of poplar and pine slightly decreased, while those of eucalyptus and Chinese fir increased after acetylation, likely as a result of the difference in natural color among these species. Compared with the control, however, acetylation made the differences less noticeable. Furfurylation resulted in more dramatic color changes in all of the samples, primarily a decrease in *L**; the *L** value after modification was dependent on the concentration of furfuryl alcohol. The *a** values of all furfurylated samples noticeably increased compared with those of the untreated samples, although they decreased as alcohol concentration increased (except for the pine sample.) The *b** values varied among species. Increased values of *a** and *b** indicated that the wood surface tended to turn red after treatment (Temiz *et al*. 2007). Although the WPG of furufrylated wood linearly increased, color darkness increased out of step, probably because the natural color of wood had been completely covered by the color of poly(furfuryl alcohol) generated by the conjugated structure (Choura *et al*. 1996). Especially, the color changes of furfurylated pine wood were slight under different polymer loading, which indicated that lower WPG can cover the natural color of wood completely.

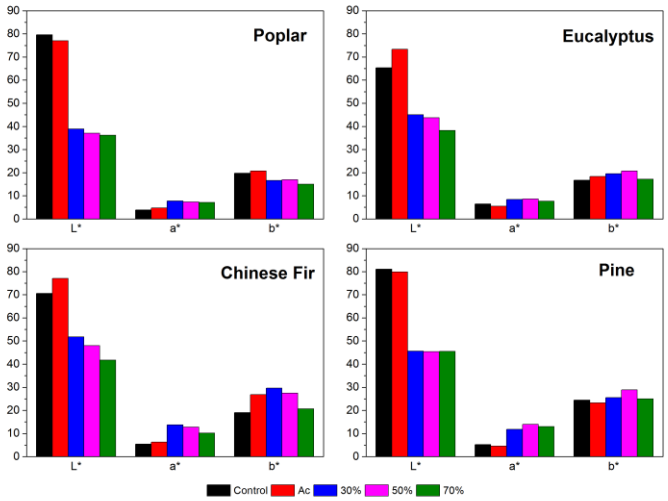

Fig. 2. Color changes (*L*, a**, and *b**) of four wood species before and after modification

The Δ*E** variations are shown in Fig. 3. The Δ*E** values of samples after acetylation changed only slightly, while those after furfurylation changed dramatically, especially for poplar and pine woods, whose natural color is lighter than the other two species (Fig. 3); acetylation and furfurylation reduced this difference by lightening the color of eucalyptus and Chinese fir and darkening the color of poplar and pine, respectively. In addition to pine wood, changes in Δ*E** increased as furfuryl alcohol concentration increased, likely because of the increasing loading of polymer.

Darker color is beneficial in that it can mask many blemishes and discolorations. Many treatments intended to darken the color of wood products (*e.g*., wood dyeing and thermal modification) have been reported (González-Peña and Hale 2009; Esteves and Pereira 2009; Zhao *et al*. 2014). However, some published papers have reported that furfurylated wood exhibited extensive greying effects on their surfaces after a long time of outdoor weathering (Temiz *et al*. 2007; Mantanis and Lykidis 2015). Therefore, the potential application of the fufurylated fast-growing wood could be for indoor use, such as furniture, wood floor, and some decorations, to replace the valuable timber.

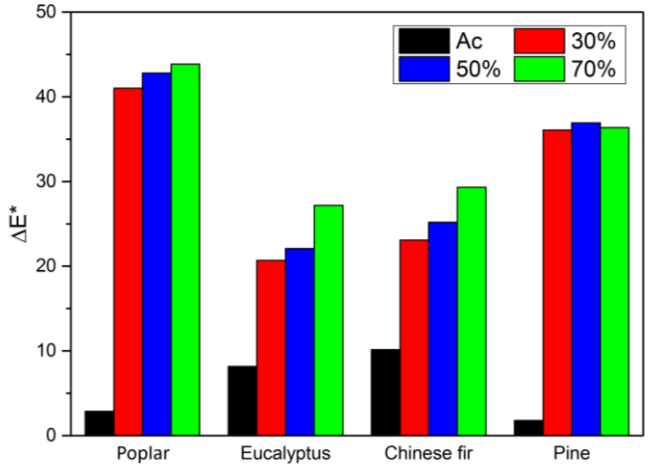

Fig. 3. Color changes (*ΔE**) of four wood species before and after modification

Morphology Characterization

Sections cut transversely through the wood samples were observed under a light microscope to investigate the differences between earlywood and latewood (Fig. 4).

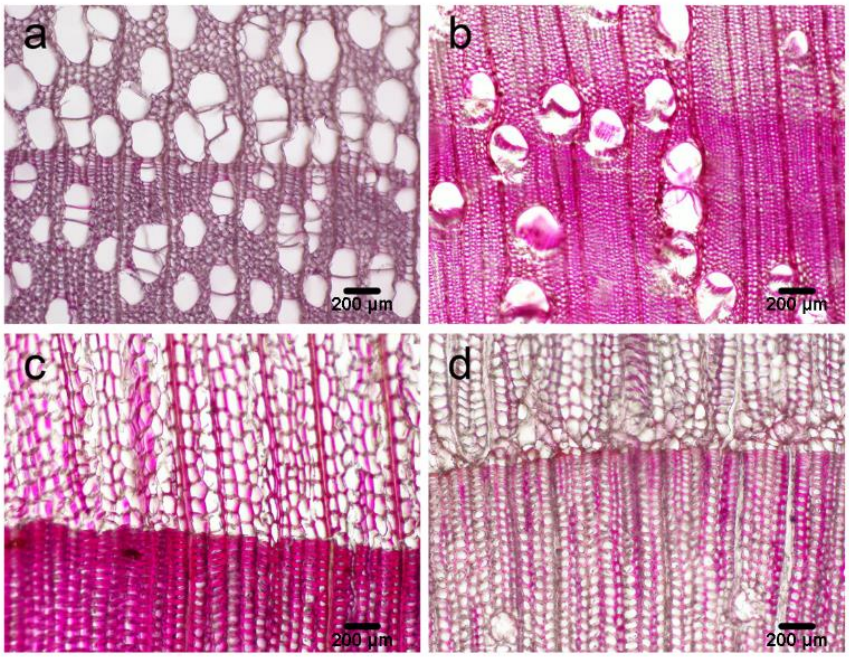

Fig. 4. Observations of four wood species by light microscope: (a) poplar, (b) eucalyptus, (c) Chinese fir, and (d) pine

Poplar and eucalyptus exhibited distinct qualities of diffuse-porous wood. The poplar showed looser structure than that of eucalyptus, which showed a lower density and increased impregnability. Besides, tyloses deposits were found in vessels of eucalyptus wood. Chinese fir and pine showed distinguishable earlywood and latewood (Cramer *et al*. 2005). Additionally, some resin canals were observed in pine wood. Due to the rapid growth of these varieties, they have different anatomical structures (early-late growth ratio, tree ring width), which also impacts the modification efficiency (Csordós *et al*. 2014).

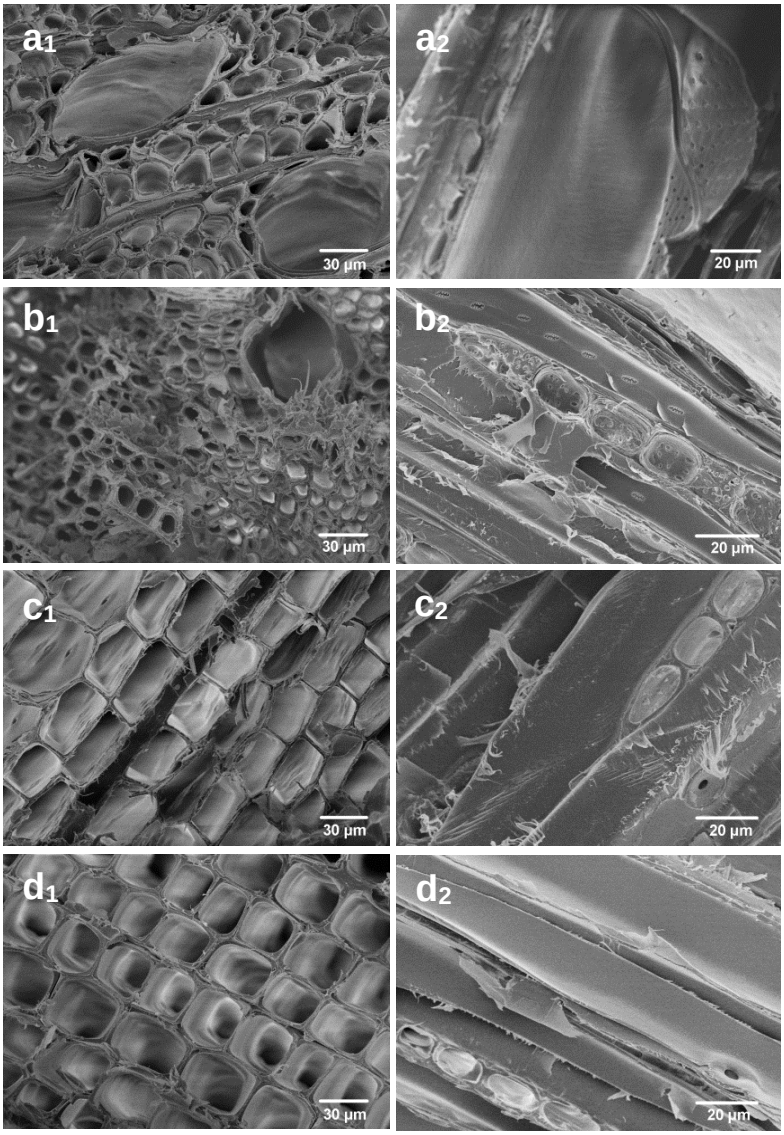

Fig. 5. FE-SEM observations of four wood species: (a) poplar, (b) eucalyptus, (c) Chinese fir, and (d) pine

The cross and tangential morphologies of the four species at magnifications of 500× and 1000× were also characterized by FE-SEM, as shown in Fig. 5. The anatomical characteristics of the different species differed remarkably, particularly the large vessels shown in the cross sections of poplar and eucalyptus and the window-like tracheid cavities in Chinese fir and pine, which exhibit softwood characteristics. The diameter of vessels in poplar was larger (a1) than that of eucalyptus. On vessel walls, there were many serried and orderly open pits (a2). Moreover, the ray cell lumens were empty in poplar wood. These phenomena contributed to the diffusion of water (Engelund *et al*. 2010). The eucalyptus exhibited vestured pits on vessel walls and obstructed ray cell lumens (b2). The tracheid walls of Chinese fir had more pits (c1) than those of pine (d1). But both had fewer pits and their pits were bordered (c2 and d2). Although the density and WPG of poplar and Chinese fir were similar, their ASE values were discrepant. This could be due to the loose

and ordered structure of Chinese fir. As a result, the modifier more easily penetrated the cell walls, became evenly distributed, and then improved the ASE, which also resulted in lower leaching rates (Lande *et al*. 2004b). Although the structure of pine and Chinese fir were similar, more pits on the cell wall of Chinese fir resulted in higher penetration of modifier. In summary, looser structure and more open pits contributed to the penetration of furfuryl alcohol; ordered structure could be helpful to the even distribution of polymer within the wood.

CONCLUSIONS

1. Four fast-growing wood species, including two hardwood species and two softwood species, were treated with furfurylation or acetylation in this study. Furfurylation improved the physical properties of wood by causing cell wall bulking, while acetylation accomplished this by substituting acetyl function for the hydroxyl groups of cell wall. Because of the differing modification mechanisms, furfurylation is likely more suitable for wood species with more open pits and loose and ordered structures; the effects of acetylation, however, were not influenced by the species of wood.
2. Furfurylation resulted in noticeable color changes in all of the samples, primarily a decrease in L^* that depended on polymer loading. Acetylation resulted in only slight color changes, however, making the Chinese fir and eucalyptus color lighter. Both treatments were shown to successfully reduce unevenness in the color of wood samples.

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