Modification with Melamine Formaldehyde and Melamine-Urea Formaldehyde Resin to Improve the Physical and Mechanical Properties of Wood

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Scots pine and white poplar were modified with melamine formaldehyde (MF) and melamine-urea formaldehyde (MUF) resins to improve their physical and mechanical properties. Impregnation was conducted at 4 bar pressure for 30 or 60 min, and the samples were cured at a temperature of 150 °C for 40 min in an oven. The density, equilibrium moisture content, weight percent gain, bulking effect, water uptake, volumetric swelling, anti-swelling efficiency, modulus of rupture, modulus of elasticity, compression strength parallel to the grain, and Brinell hardness of the modified wood were determined. The anti-swelling efficiencies were 57% and 74% in Scots pine and white poplar, respectively, using the melamine formaldehyde resin. Modification of white poplar with melamine-urea formaldehyde increased the modulus of elasticity, compression strength, and Brinell hardness considerably. Both resins were successful at improving the physical and mechanical properties of Scots pine and white poplar woods.

Keywords: Melamine-urea formaldehyde; Wood modification; Anti-swelling efficiency; Mechanical properties; White poplar; Scots pine

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INTRODUCTION

Wood has been widely used for many applications, such as construction, furniture, and tools, because of its many desirable characteristics, such as a favorable mass/strength ratio, natural durability, and biodegradability. Wood also possesses some properties that can be regarded as disadvantages. The most significant problem is its lack of dimensional stability. Dimensional instability of wood, because of moisture changes, limits its use in certain applications, principally for outdoor use (Gindl *et al*. 2003b; Epmeier *et al*. 2004). A shortage in high quality hardwoods has driven researchers and manufacturers to search for alternative resources, such as softwoods and some low-density hardwoods, for value-added applications. To achieve this goal, suitable technologies are needed to improve woods' properties, *i.e.*, the dimensional stability, durability, mechanical strength, and hardness, to meet specific end-use requirements (Cai 2007; Hochmanska *et al*. 2014).

The treatment of wood, with different types of chemicals or resins, has been widely studied with the end goal of property enhancement. Melamine formaldehyde (MF) and melamine-urea formaldehyde (MUF) are two common resins for wood-related applications. Melamine formaldehyde (MF) resin, commonly used in thermosetting, has applications in coatings, adhesives, and both paper and textile treatments (Pittman *et al.* 1994). Melamine-urea formaldehyde resin has been applied in the manufacturing of plywood and particleboard for dry conditions. The synthesis process of MUF is based on a urea formaldehyde resin with a small amount of melamine added to improve the water repellency and dimensional stability. In comparison with phenolic resins, MUF is less expensive and has low viscosity. In addition, MUF is almost transparent, which is favorable for impregnation (Cai 2007). Various types and grades of MF resin are available for the impregnation of wood. Impregnation of solid wood with water-soluble MF resin has led to a significant improvement in the surface hardness and the modulus of elasticity (MOE) (Miroy *et al*. 1995; Deka and Saikia 2000; Epmeier *et al*. 2003; Deka *et al*. 2007). Gindl *et al*. (2003a) investigated the influence of melamine-formaldehyde impregnation on the transverse compression strength and stiffness of spruce wood. Gindl *et al*. (2003b) also examined factors influencing the uptake of MF resin into the cell wall of softwood. Gindl *et al*. (2004) used water-soluble MF resin to improve the hardness of Norway spruce and indicated that a minimum impregnation depth of 2 mm was required to achieve an optimal increase in hardness. Epmeier *et al*. (2003) reported that the anti-swelling efficiency (ASE) of Scots pine, beech, and birch woods increased by 30% to 75% when modified with eight different chemicals. Also, a 20% decrease in the equilibrium moisture content (EMC) was reported in wood treated with methylated melamine formaldehyde (Epmeier *et al*. 2003). Deka *et al*. (2007) indicated that the modification of Norway spruce with MF resulted in a 17.5% increase in ASE. Epmeier *et al*. (2004) compared the physical and mechanical properties of nine different modified woods and reported that the acetylation and furfurylation were the most effective modification methods for achieving a high dimensional and stiffness stability and a low EMC. The treatment of wood with phenol-formaldehyde resin is considered an important way to improve the properties of wood (Fruno *et al*. 2004; Liu and Wang 2004; Wan and Kim 2008; Huang *et al*. 2013).

Although many studies have been focused on MF modification, it has not been widely applied in an industrial setting because of its higher cost than other polymerizable monomers and pre-polymers (Deka *et al*. 2007). Alternatively, MUF resin has a greater potential of infiltrating the industry because it is relatively low cost and has a high availability. Research on the modification of wood with MUF is limited, and there is limited research on the modification of white poplar wood, which has low density, low-cost, and is widely available. The modification of white poplar with MF or MUF could potentially improve its physical and mechanical properties and may contribute value-added products to the wood industry. In this study, the effect of MF and MUF resin modification on the physical and mechanical properties of Scots pine and white poplar woods was investigated.

EXPERIMENTAL

Materials

Scots pine (*Pinus sylvestris* L.) (SP) and white poplar (*Populus alba* L.) (WP) were used in this experiment. For the modification process, commercially available melamine formaldehyde resin (Almin-65) and melamine-urea formaldehyde resin (Genmuf-20) was provided by the Gentaş Kimya Inc., Tuzla, Turkey. The properties of these resins are given in Table 1, based on the manufacturer's information.

Table 1. Properties of Melamine Formaldehyde and Melamine-Urea Formaldehyde Resins

Properties	Melamine formaldehyde	Melamine-urea formaldehyde
Appearance	Clear, colorless liquid	Opaque, white liquid
Melamine content (%)	36 ± 1	18 ± 1
Solid content (% w/w)	51 ± 1	65 ± 1
Viscosity	15-20 (s F.C.4 20 °C)	450 to 800 (cps, 20 °C)
pH (20 °C)	9.6 to 10.5	9.0 to 9.30
Density (g/cm^3 at 20 °C)	1.220 to 1.240	1.278 to 1.282
Water tolerance (20 °C)	1/1 to 1/2	---
Gel time	35.00 to 40.00 (min, 130 °C)	36 to 38 (s,100 °C)

Methods

Preparation of samples and modification process

Samples were cut into dimensions of 20 × 20 × 360 mm (R × T × L) and acclimatized at a temperature of 20 ± 3 °C and a relative humidity (RH) of 65% for 4 weeks. The weight and the volume of the samples were determined, and then the samples were oven-dried at a temperature of 103 ± 2 °C. The oven-dried density and moisture content of the samples were calculated before chemical modification. The MF resin was diluted by 50% with distilled water, while the MUF resin was applied without further modification. The impregnation was performed separately for each treatment level and a new resin solution was used for each of the different variations. Impregnation was conducted in a stainless steel chamber at 4 bar pressure for 30 or 60 min, depending on the treatment level. The test specimens were stacked without coming into contact with one another in a glass container, and then the modification solution was poured into the container. The container was placed in the chamber, and 4 bar pressure was maintained for 30 or 60 min durations. After the impregnation process, the samples were cured at a temperature of 150 °C for 40 min in an oven, after removing excess resin on the surface. After curing, the weight and dimension of the samples were measured and recorded, representing the modified values. Before physical and mechanical analysis testing, the samples were cut into various dimensions, according to the specifications of the testing standard. Twenty replicates were prepared for each experiment, and the samples were acclimatized at a temperature of 20 ± 3 °C and a RH of 65%.

Determination of physical properties

Cross-sectional surface views of the modified Scots pine and white poplar woods were captured by using a digital microscope (Celestron 44308 Digital Pro) to illustrate the placement of the resin particles. To obtain a better image, a thin layer was cut from the surface of the samples by using a microtome (Leica SM200R) to flatten the surface.

The oven-dry densities (TS 2471 (1976)) and the equilibrium moisture content (TS 2472 (1976)) of the samples, before and after the modification treatment, were determined. The weight percent gain (WPG) of the samples was calculated according to the following equation,

$$WPG = \frac{m_i - m_0}{m_0} \times 100 \qquad (1)$$

where m_o is the oven-dry weight of the samples before the modification in grams and m_j is the oven-dry weight of the samples after the modification in grams.

The bulking effect (BE) of the modification as a percent was calculated according to Eq. 2,

$$BE = \frac{V_i - V_0}{V_0} \times 100 \tag{2}$$

where V_o is the oven-dry volume of the samples before the modification in cm^3 and V_i is the oven-dry volume of the samples after the modification in cm^3.

To determine the effect of the modification on the dimensional stability, the water uptake (WU), swelling (S), and anti-swelling efficiency (ASE) values of the samples were determined. According to the testing standard, TS 4086 (1983), 20 × 20 × 30 mm (T×R×L) samples were immersed in distilled water for 4 wk. After immersion, the dimension and the weight of the samples were recorded, and the dimensional stability values were calculated according to Eqs. 3 through 5,

$$WU = \frac{m_w - m_i}{m_i} X100 \tag{3}$$

where m_w is the weight of the samples after water immersion in grams and m_i is the oven-dry weight of the samples after the modification in grams,

$$S = \frac{V_W - V_i}{V_i} X100 \tag{4}$$

where V_w is the volume of the samples after water immersion in cm^3 and V_i is the oven-dry volume of the samples in cm^3; and

$$ASE = \frac{S_m - S_u}{S_u} X100 \tag{5}$$

where S_m is the swelling ratio of the modified samples as a percentage and S_u is the swelling ratio of the unmodified samples as a percentage.

Determination of mechanical properties

Certain mechanical properties of the modified samples, including the modulus of rupture (MOR), modulus of elasticity (MOE), compression strength parallel to grain (CS), and Brinell hardness (BH) were determined according to the TS 2474 (1976), TS 2478 (1976), TS 2595 (1977), and TS 2479 (1976) testing standards, respectively.

Statistical analysis

The data were analyzed statistically using an SPSS software system (SPSS17, IBM Corp., Armonk, NY). An analysis of variance (ANOVA) model was used to determine the effect of the MF and MUF resins on the mechanical and physical properties of wood. Duncan's multiple range tests were used for pairwise comparisons when the overall ANOVA model was significant. Data for Scots pine and white poplar woods were analyzed using separate models.

RESULTS AND DISCUSSION

Physical Properties

Modification with MF or MUF increased the oven-dry density of the samples. The density of the wood increased when the resins occupied the cell lumens and cell walls. Cross-sectional surface views of the modified Scots pine and white poplar woods are shown in Fig. 1. The oven-dry density and the EMC of the modified Scots pine and white poplar samples are tabulated in Table 2.

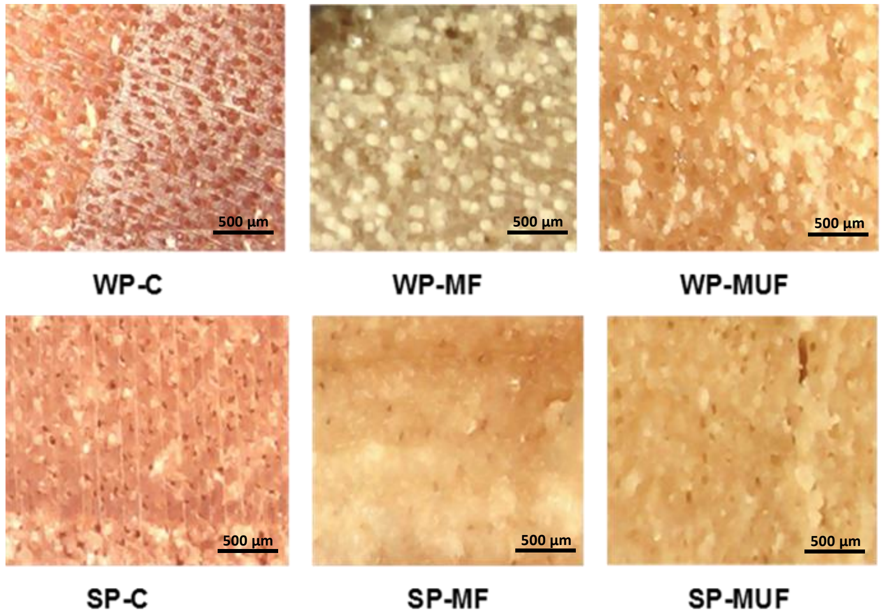

Fig. 1. Cross-sectional view of the modified wood
SP: Scots pine, WP: White poplar, C: Control, MF: Melamine formaldehyde MUF: Melamine-Urea formaldehyde

Table 2. Oven-Dry Densities and Equilibrium Moisture Content of Modified Wood

Wood species	Chemical	Impregnation time (min)	Sample code	Oven-dry density (g/cm^3)			EMC (%)
				Unmodified	Modified	Change (%)	Modified
Scots pine	--	--	SP-C	0.42	-	-	10.18
	MF	30	SP-MF-30	0.41	0.52	26.8	8.36
		60	SP-MF-60	0.42	0.59	40.5	7.67
	MUF	30	SP-MUF-30	0.40	0.44	10.0	7.49
		60	SP-MUF-60	0.41	0.46	12.2	7.61
White poplar	--	--	WP-C	0.34	-	-	9.52
	MF	30	WP-MF-30	0.31	0.46	48.4	8.66
		60	WP-MF-60	0.32	0.51	59.4	7.03
	MUF	30	WP-MUF-30	0.37	0.48	29.7	6.76
		60	WP-MUF-60	0.33	0.47	42.4	7.99
SP: Scots pine, WP: White poplar, C: Control, MF: Melamine formaldehyde MUF: Melamine-Urea formaldehyde, EMC: Equilibrium moisture of content							

The highest increase (59.4%) in density was observed in the WP-MF-60 samples, while the lowest (10%) density was observed in the SP-MUF-30 samples. The MF-modified samples exhibited a greater increase in density than the samples modified with MUF for both wood species. Although the densities of the pure resins were similar, dilution of the MF with water in the impregnation process may have enhanced its penetration into the wood. Also, the increase in density of modified white poplar was higher than that of Scots pine, regardless of the resin. Although swelling effect of the treatment was similar in both wood species, much higher weight percent gain values were determined in white poplar compared to Scots pine. This indicated that the amount of resin placed in the voids in white poplar was higher than that of Scots pine, because of poplar' higher porosity. Therefore, the density increase was higher in the white poplar.

All of the modifications reduced the EMC by 1.5% to 2.5% compared with the unmodified (control) samples. The MF- or MUF-filled cell lumens of the samples prevented water penetration into the wood; however, there was no statistical difference between the EMC of the MF- or MUF-modified samples. In agreement with the results of this study, Epmeier *et al*. (2003) reported a slight decrease in EMC at 90% RH while using methylated melamine formaldehyde resin. The dimensional stability of the modified wood was determined based on the weight percent gain, bulking effect, water uptake, and anti-swelling efficiency values given in Table 3.

Table 3. Weight Percent Gain, Bulking Effect, Water Uptake, Volumetric Swelling, and Anti-Swelling Efficiency Values of Modified Wood

Sample Codes	WPG (%)	BE (%)	WU (%)	S (%)	ASE (%)
SP-C	-	-	115.5^{a}	14.0^{c}	-
SP-MF-30	36.3^{b}	6.7^{ab}	63.0^{b}	7.1^{ab}	49.3^{ab}
SP-MF-60	53.8^{c}	9.2^{a}	62.0^{b}	6.0^{a}	57.0^{a}
SP-MUF-30	14.4^{a}	4.1^{b}	65.4^{b}	7.5^{ab}	46.4^{ab}
SP-MUF-60	17.3^{a}	4.6^{b}	64.0^{b}	9.5^{b}	32.5^{b}
WP-C	-	-	205.2^{x}	11.9^{x}	-
WP-MF-30	61.4^{x}	9.9^{x}	135.7^{y}	3.5^{y}	70.5^{x}
WP-MF-60	73.1^{x}	9.5^{x}	128.4^{y}	3.1^{y}	74.1^{x}
WP-MUF-30	44.5^{y}	4.9^{y}	118.9^{y}	7.6^{z}	35.4^{y}
WP-MUF-60	48.9^{y}	5.3^{y}	124.4^{y}	6.8^{z}	42.6^{y}

Means within a column with different subscripts differ ($P < 0.05$)

The WPG values of the white poplar were higher than those of Scots pine. Also, MF caused a higher WPG than MUF. The highest WPG value was determined in MF-60 for both wood species. The BE values showed a similar trend with the WPG values. These results showed that MF or MUF impregnated both the cell lumens and the cell walls. The BE of MF-modified wood was higher than that of MUF-modified wood. It was reported that an aqueous melamine formaldehyde solution can penetrate the secondary cell wall of Scots pine or larch wood (Gindl *et al*. 2002) and the amorphous region of cellulose fibrils (Hua *et al*. 1987). Dilution of MF with water decreased its viscosity; therefore MF may have penetrated in wood better than MUF. Higher molecular weight of the melamine-formaldehyde (156.149 g/mol) compared to urea-formaldehyde (90.082 g/mol) may have contributed the higher WPG and BE in modification with MF. Although a longer impregnation time increased the WPG values, there was no statistically significant difference between the 30-min and 60-min impregnation durations, with the exception of the MF modification in Scots pine. Pressure applied in the impregnation process affects the penetration of the impregnation chemicals. Results showed that 30 min impregnation at 4 bar pressure was enough to achieve adequate penetration of MF or MUF resin. Because both wood species were classified as moderately difficult species for impregnation (Aslan 1998), and because of the relatively small dimensions of the samples, it can be said that penetration of the resins were good. Although the BE values were similar for both species, the WPG values of white poplar were much higher than that of Scots pine, most likely because of the lower density and larger cell lumen of white poplar wood. This result indicated that the majority of the resin occupied the cell lumen of the wood. This was also visualized in Fig. 1.

Modification with MF or MUF reduced the water uptake of the wood substantially. Reductions in the WU varied from 34% to 59%; however, there was no statistical difference between groups. The WU values of white poplar were approximately double that of Scots pine. Consequentially, water penetrated primarily the wood cell lumen; thus, the white poplar wood took on more water because of its larger lumen space. Modification of wood with MF or MUF reduced the swelling of the wood. Swelling in modified Scots pine was higher than that of MF-modified white poplar. Scots pine swelled more because of thick cell wall and narrow lumina of latewood (Erdin and Bozkurt 2013). Difference between the swelling values of MUF-modified Scots pine and white poplar relatively low. The lower WPG in MUF-modified samples may have caused this result. The ASE values of the MF-modified samples were higher than that of MUF-modified samples. Deka *et al*. (2007) reported a 17.5% increase in ASE for MF-modified Norway spruce. Likewise, Epmeier *et al*. (2003) reported a higher ASE (60% to 75%) for acetylation, furfurylation, and maleoylation, and a lower ASE (22% to 45%) in succinylation, methylated melamine formaldehyde-modification, and oil-heat treatment. In this study, higher ASE values of 57% and 74% for MF-modified Scots pine and white poplar, respectively, were achieved. The ASE of MUF-modified wood was comparable with the ASE of various modified wood reported in the literature (Epmeier *et al*. 2004; Deka *et al*. 2007). The ASE of MUF-modified woods were 46.4% and 42.6% for the Scots pine and white poplar, respectively. The ASE values showed a similar trend with the BE values as expected. Because of swelling of the wood is based on the swelling of the cell walls, primarily resins placed in the cell wall prevent swelling of wood more. The water penetrates into the cell wall through cell lumina, pits, and rays. White poplar has large cell lumina, simple perforation plate and extremely large simple ray-vessel pits and, Scots pine has large pits, resin canals, rays with large fenestriform pits, (Schoch *et al.* 2004; Erdin and Bozkurt 2013); therefore, resins penetrated well into the woods. Cured resin in these voids and cell lumina, blocked the ways and prevent the water penetration to the cell wall.

Mechanical Properties

The MOR, MOE, CS, and Brinell hardness values of the modified woods were determined (Table 4).

Table 4. Mechanical Properties of Modified Wood

Sample Code	MOR (N/mm^2)	MOE (N/mm^2)	CS (N/mm^2)	HBR (N/mm^2)
SP-C	80.32^{b}	6894.37^{b}	49.52^{a}	1.44^{a}
SP-MF-30	57.59^{a}	5684.94^{a}	44.96^{a}	1.86^{ab}
SP-MF-60	61.58^{a}	5678.86^{a}	49.43^{a}	2.11^{b}
SP-MUF-30	77.13^{b}	6952.99^{b}	50.39^{a}	1.56^{a}
SP-MUF-60	73.45^{b}	7441.05^{b}	52.68^{a}	1.72^{ab}
WP-C	69.25^{z}	5070.19^{y}	38.31^{x}	1.32^{x}
WP-MF-30	44.37^{x}	3798.96^{x}	35.18^{x}	1.53^{xy}
WP-MF-60	49.80^{x}	4678.33^{xy}	38.26^{x}	1.57^{xyz}
WP-MUF-30	59.58^{y}	6401.41^{z}	57.77^{y}	1.90^{yz}
WP-MUF-60	57.52^{y}	6424.00^{z}	59.03^{y}	1.95^{z}

Means within a column with different subscripts differ ($P < 0.05$)

Modification with MF reduced the MOR for Scots pine, while modification with MUF reduced the MOR slightly. The reduction in the MOR by MF was approximately 25%. Epmeier *et al*. (2003) reported an approximately 10% increase in MOR with the methylated melamine formaldehyde resin modification; however, acetylation and furfurylation were not significant. The white poplar wood, with both the resins, exhibited a reduction in the MOR. There was also a significant difference between the MOR of MF-modified samples and MUF-modified samples. The reduction in the MOR of the MF-modified samples was higher than that of the MUF-modified samples.

Modification with MF decreased the MOE, while modification with MUF increased the MOE for both wood species. The increase in MOE of MUF-modified Scots pine was insignificant compared to unmodified samples; however, the increase in MOE of MUF-modified white poplar was significant. It can be inferred that the modification with MF or MUF did not always affect the MOE. Epmeier *et al*. (2004) indicated that although the MOE was affected by a change of density, an increase in density caused by chemical modification does not affect the MOE in the same manner.

The compression strength parallel to the grain values of the Scots pine was not affected by the modification with MF or MUF; however, there was a slight decrease in the MF-modified samples and a slight increase in the MUF-modified samples. The CS of the MUF-modified white poplar was higher than that of control samples. Gindl *et al*. (2003a) reported an increase in compression strength perpendicular to the grain of MF-modified spruce and attributed this improvement to a modification of the cell wall and not the filling of the tracheid lumina.

The BH of MF- or MUF-modified wood was higher compared to the BH of unmodified wood; however, there was no obvious difference in the modified groups, based on the statistical analysis. The increase in BH of MUF-modified white poplar and MF-60-modified Scots pine were approximately 45%. In accordance with these results, Gindl *et al*.(2002), Epmeier *et al*. (2003), Lande *et al*. (2004), and Deka *et al*. (2007) reported that the compression strength of the wood notably increased by chemical modification at high weight percent gain levels.

Because of the notable improvement in the dimensional stability of MF/MUF-modified Scots pine and white poplar, they could be used for outdoor applications, such as garden furniture, doors/windows, and wooden siding fixtures. In particular, MUF-modified wood is suitable for use in humid conditions, such as in bathrooms, because of its lower EMC. White poplar’s structural elements under compression loading could be modified with MUF because the modification with MUF increased its CS substantially. The modification with MF or MUF increased the BH of Scots pine and white poplar, which is favorable for wooden elements exposed to abrasion and/or scratching, *i.e.*, wood for flooring, table tops, or cabinet doors.

CONCLUSIONS

1. The physical and mechanical properties of Scots pine and white poplar can be improved by modification with melamine formaldehyde and melamine-urea formaldehyde resins.

2. The dimensional stability of the Scots pine and white poplar was enhanced by melamine-formaldehyde (MF) or melamine-urea-formaldehyde (MUF) modification. The enhancement of white poplar was higher than that of Scots pine with the MUF modification.
3. The modification with MF or MUF increased the density and decreased the equilibrium moisture content (EMC) of the Scots pine and white poplar woods.
4. The MF and MUF penetrated not only into the cell lumens, but also into the cell walls. The MF resin penetrated the wood better than the MUF resin; thus, the WPG values of the MF-modified woods were highest.
5. Because there was no considerably difference between the 30-min and 60-min impregnation durations, a 30 min impregnation at 4 bar pressure is recommended to adequately modify the wood with MF and MUF.
6. The modification with MUF did not affect the mechanical properties of Scots pine, while modification with MF decreased the mechanical properties. The modification with MUF increased the MOE, CS, and BH of white poplar wood.

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