COMPARATIVE STUDY ON WEATHERING DURABILITY PROPERTY OF PHENOL FORMALDEHYDE RESIN MODIFIED SWEETGUM AND SOUTHERN PINE SPECIMENS

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ABSTRACT

The effects of low molecular weight phenol formaldehyde resin on weathering durability property of sweetgum (Liquidambar styraciflua) and southern pine (Pinus taeda) specimens were studied using six wet-dry cycles with ultraviolet light accelerated weathering test following ASTM via evaluating the water repellent efficiency, dimensional stability, and crack formation of wood. The results showed that 1) the water repellent efficiency of treated quarter-sawn sweetgum specimens was higher than those of treated quarter-sawn and flatsawn southern pine specimens; 2) the dimensional stabilities of sweetgum and southern pine specimens were all improved by impregnating low molecular weight phenol formaldehyde resin, especially for sweetgum; 3) there were clearly more cracks on exposed ends and surfaces of all treated sweetgum and southern pine specimens than those on control ones, indicating that the low molecular weight phenol formaldehyde resin modification used in this study were not able to improve the anti-cracking properties of sweetgum and southern pine specimens. Generally, the sweetgum was more suitable to be impregnated with low molecular weight phenol formaldehyde resin than southern pine with the procedure described according to dimensional stability and water repellent efficiency, in order to improve the weathering durability.

Keywords: Dimensional stability, phenol formaldehyde resin, water repellent efficiency, weathering property.

INTRODUCTION

Southern pine (Pinus taeda) and sweetgum (Liquidambar styraciflua) are two of the most common wood species in the Southern of the United State of America (USA). The southern pine has the specific gravity of 0.53 with straight grain, has been widely used in engineered wood timber and composites, while sweetgum has specific gravity of 0.55 with interlocked grain which makes the wood deform and crack easily if not dried properly. Thus, sweetgum is used principally for veneer, plywood, slack cooperage, fuel, pulpwood, boxes and crates (Forest Products Laboratory 2010).

Some studies have been conducted using chemical modified methods and thermal heated methods to improve the wood weathering durability properties, such as water-repellent efficiency (WRE), dimensional stability, crack formation, as well as mechanical properties. In case of chemical modification of wood, the following chemical solutions were commonly used, e.g. phenol formaldehyde (PF) resin (Wan and Kim 2008, Gabrielli and Kamke 2010, Ghani et al. 2017, Yan et al. 2021), melamine formaldehyde (MF) resins (Hansmann et al. 2006), urea-formaldehyde resin (Lu et al. 2021), N-methylol resin/sucrose (Huang et al. 2019), N-methylol melamine /PF (Kielmann and Mai 2016a, Kielmann and Mai 2016b), and

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graphene/polyvinyl alcohol (Cai et al. 2021, Wu et al. 2021). The results of these studies show that the dimensional stability, including anti-swelling-efficiency (ASE), WRE, and selected mechanical properties of evaluated wood species were all improved as compared with control specimens. Heat treatment is one of the wood modification methods for improving wood properties such as dimensional stability, water resistance, and biological durability without using harmful chemicals (Nasir et al. 2019, Silva et al. 2020). However, heat treatments resulted in some shortcomings, such as loss of wood toughness, reduced wood tensile and bending strengths, unstable color in exterior exposure, and surface cracks (Zivkovic et al. 2008, Yan and Chang 2019, Wang et al. 2020). It was also reported that the resistance of heat-treated wood against weathering (UV light and moisture changes) was not improved much as compared to control specimen (Jirous-Rajkovic and Mikletic 2019, Li and Ma 2021).

Furuno et al. (2004) investigated the impregnation of PF resin into wood cell walls to improve the dimensional stability and decay resistance of wood and found out that low (290 g/mol) molecular weight (MW) resin penetrated more easily into cell walls and formed thicker polymer layered-wall inside wood cell walls than medium (470 g/mol) and high (820 g/mol) MW resins did. Biziks et al. (2019) used light microscopy to examine the distribution and penetration depth of PF resin impregnated into beech (Fagus sylvatica) wood and observed that the PF resin mainly penetrated into fiber lumens and specimens with a concentration of 27 % resulting in more uniformly resin distribution in fiber lumens than other two lower concentrations (9 % and 18 %) evaluated. These observations indicated that a low MW PF resin with certain concentration was more proper for treating wood.

Visible cracks of wood influence the appearance of wood products severely, which arise in the wood surface during weathering because of the growth of micro-cracks, photochemical reactions, and moisture-induced stress. One way to minimize these types of cracks is that wood for outdoor applications should be radially sawn (Coupe and Watson 1967). Cracks in the radial section are not only fewer but also smaller than those in tangential sections (Sandberg and Söderström 2006, Wan et al. 2017, Tu et al. 2020). Meanwhile, some studies were also focused on treating methods to reduce the cracks of wood when used in outdoor applications. Hansmann et al. (2006) studied the weathering resistance regarding the wood color and hardness of spruce (Picea abies) and poplar (Populus nigra) treated with different MF resins when subjected to artificial weathering experiment. Their results showed that the treated specimens also had advantages compared to untreated reference specimens regarding discoloration and crack formation. Altgen et al. (2012) reported that the crack formation was higher for the thermally modified wood than for the control one.

From material science point of view, the durability of sweetgum and southern pine subjected to the same process has never been studied and compared with. The main objective of this study was to investigate the effect of low MW PF resin modification on the dimensional stability and anti-cracking properties of sweetgum and southern pine subjected to accelerated weathering tests.

MATERIALS AND METHODS

Materials

The wood materials used in this study were southern pine (Pinus taeda) and sweetgum (Liquidambbar styraciflua), which were from Mississippi State, USA. The moisture content (MC) of southern pine and sweetgum were 12.01 % and 10.41 % respectively. The specific gravities (SG) of them were 0.53 and 0.55 respectively after being conditioned in a condition chamber at 20 °C ± 2 °C and 50 % ± 5 % relative humidity (RH) for two weeks.

All specimens were treated with a commercially available low MW PF resin with concentration of 48.42 % by impregnation (Starkville, Mississippi, MS, USA). The information of the resin is listed in Table 1.
Table 1: Information of low molecular weight PF resin.

| Parameters          | Values     |
|---------------------|------------|
| Non-volatile content (%) | 48.42%     |
| Specific gravity    | 1.15       |
| Viscosity (cps)     | 125        |
| pH                  | 8.5        |
| Free Phenol (%)     | 8.0        |
| Free Formaldehyde (%) | 0.15 max  |
| Molecular weight (Mn) | 310        |

**Specimen preparation**

The dimensions of all tested specimens were 560 × 100 × 24 mm (length × width × thickness) machined by clear wood. For southern pine, quarter-sawn and flat-sawn specimens were prepared. For sweetgum, the specimens were all quarter-sawn. Figure 1 shows the dimensions and grain orientations of all tested specimens.

![Figure 1: Dimensions of (a) quarter-sawn southern pine, (b) flat-sawn southern pine, and (c) quarter-sawn sweetgum specimens.](image)

**Modification procedure**

Southern pine and sweetgum specimens were impregnated with the low MW PF resin of 20 % concentration of stock solution based on the previous study (Wan and Kim 2008). The main steps were that 1) weigh the specimens and place the specimens into a pan of cylinder with sticks between adjacent specimens; 2) vacuum the cylinder to -85 kPa and keep the vacuum for 15 min and then add low MW PF resin to cover the specimens; 3) evacuate the cylinder and apply air pressure of 690 kPa for 30 min; 4) release the pressure and pour the PF resin solution out, take out specimens and weigh specimens after impregnation; 5) put all specimens into an oven at 80 °C and 103 °C for 24 hours respectively. Finally, the treated specimens were obtained. Six specimens were prepared for each type of specimen.

**Accelerated weathering test**

Figure 2 shows the set-up of the accelerated weathering test. The specimens were stored in condition chamber at 20 °C ± 2 °C and 50 % ± 5 % RH for two weeks before tests and then initial dimensions and weights of all specimens were measured. The procedure of accelerated weathering test mainly refers to ASTM D2898-10 (2017). The specific steps are that 1) 24-h wet with water spray to make sure that the specimens are...
saturated; 2) measure the weights and dimensions (width and thickness) at ends of water spray or wet step according to Figure 3; 3) 24-h dry at 80 ºC with the combination of ultra violet (UV) light (UVA-340 nm); 4) measure the weights and dimensions of specimens again after drying; 5) repeat the wet-dry-UV light procedure for 6 times, allowing 15 minutes interval when going from heat and light step to water spray step; 6) dry the specimens in an oven of 103 ºC and measure the dimensions of them again. And, the cracks generated on exposed surfaces and at exposed ends were recorded at the end of each drying step with the numbers and lengths of cracks of these specimens being measured.

Figure 2: Setup for accelerated weathering test (a) outside view and (b) inside view.

Figure 3: Method of measuring the dimensions (W is width and T is thickness) of exposed surface and the end of wood specimens.

Weight gains of PF resin

The weight percentage gains (WPG) low MW PF resin penetrated into the samples were calculated using following Equation 1.

\[ WPG = \frac{W_0 - W_d}{W_0} \times 100 \]  \hspace{1cm} (1)
Where \( w_0 \) and \( w_d \) are oven-dried weights of control and treated samples.

**Water repellent efficiency**

The water absorptions and WREs of all specimens were calculated using Equation 2 and Equation 3 respectively.

\[
\text{Water absorption (\%)} = \frac{W_f - W_i}{W_i} \times 100\% \tag{2}
\]

\[
WRE (\%) = \frac{W_u - W_t}{W_u} \times 100\% \tag{3}
\]

Where \( WRE \) is the water-repellent efficiency; \( W_f \) is the weight of a specimen after water soaking for 24 hours (g); \( W_i \) is the initial weight of a specimen (g); \( W_u \) refers to the water absorption of a control specimen; \( W_t \) represents the water absorption of a treated specimen.

**Dimensional stability**

Volumetric swelling coefficient (SC) and the anti-swelling efficient (ASE) were calculated using Equation 4 (Islam et al. 2012) and Equation 5 (Ghani et al. 2017).

\[
SC (\%) = \frac{V_f - V_i}{V_i} \times 100 \tag{4}
\]

\[
ASE (\%) = \frac{S_c - S_t}{S_t} \times 100 \tag{5}
\]

Where \( SC \) is the volumetric swelling coefficient; \( V_f \) refers to the final volume of a specimen after water absorption (mm\(^3\)); \( V_i \) refers to the initial volume of a specimen before water absorption (mm\(^3\)); \( ASE \) means the anti-swelling efficiency; \( S_c \) refers to the volumetric swelling coefficient of a control specimen; \( S_t \) represents the volumetric swelling coefficient of a treated specimen.

**Crack formation**

The number and length of cracks on exposed surfaces and at exposed ends were used to evaluate the anti-cracking property of low MW PF resin modified specimens after the accelerated weathering test was completed. All specimens were oven dried after measuring the dimensions, and the lengths and the number of cracks on ends and surfaces were observed with naked eyes and measured by a caliper. During the process cracks at ends and on surfaces of specimens exposed to water spray were counted. For cracks with their lengths longer than 5 mm, the total lengths and the numbers of cracks were all recorded (Sandberg 1999).

**Statistical analysis**

All data evaluated in this study were analyzed using the analysis of variance (ANOVA) by SPSS Software (2013) using general linear model (GLM) procedure, and mean comparisons were analyzed using the protected least significant difference (LSD) at the 5 % significant level.
RESULTS AND DISCUSSION

Weight gains of PF resin

Table 2 shows the WPG of all samples, which indicating that low MW PF resins penetrated into the quarter-sawn sweetgum samples were significantly greater than other into flat-sawn and quarter-sawn southern pine samples.

Table 2: Weight percentage gains of all samples evaluated.

| Sample type                  | WPG (%)      |
|------------------------------|--------------|
| Quarter-sawn southern pine   | 13.39 (1.38) A |
| Flat-sawn southern pine      | 11.08 (0.95) A |
| Quarter-sawn sweetgum        | 11.67 (1.37) B |

Water repellent efficiency

Figure 4 shows the weight gains of all control and treated specimens during accelerated weathering tests. It indicated that the weight gains of control specimens were significantly higher than those of treated specimens accordingly. In case of southern pine, the weight gain of control and treated flat-sawn specimen were bigger than those of quarter-sawn specimens respectively. In addition, the weight gains of quarter-sawn southern pine were bigger than those of quarter-sawn sweetgum at first five weathering cycles.

Figure 4: Weight gains of specimens during accelerated weathering test.

Table 3 shows the water absorptions of all specimens during accelerated weathering test, indicating that the water absorptions of treated specimens are smaller than those of control specimens. It proves that low MW PF resin impregnating modification improved the water proof properties of southern pine and sweetgum specimens significantly.

Table 3: Comparisons of water absorptions during accelerated weathering test.

| Cycle | Quarter-sawn southern pine | Flat-sawn southern pine | Quarter-sawn sweetgum |
|-------|-----------------------------|-------------------------|-----------------------|
|       | Control | Treated | Control | Treated | Control | Treated |
| 1     | 32.23(27.47) A | 12.33(46.84) B | 34.61(20.92) A | 17.16(16.91) B | 23.8(2.7) A | 12.28(6.41) B |
| 2     | 33.85(20.8) A | 15.95(18.15) B | 39.76(13.97) A | 20.31(14.56) B | 29.42(6.24) A | 10.43(10.72) B |
| 3     | 37.37(33.63) A | 17.73(19.76) B | 36.34(23.47) A | 23.6(17.45) B | 33.61(6.62) A | 10.78(8.49) B |
| 4     | 34.28(17.44) A | 20.22(18.91) B | 40.66(12.42) A | 23.75(25.4) B | 34.67(8.34) A | 10.98(10) B |
| 5     | 35(18.63) A | 22.44(17.56) A | 40.74(10.16) B | 26.41(19.44) B | 37.27(5.85) A | 11.2(7.31) B |
| 6     | 34.83(19.87) A | 23.68(19.72) A | 41.41(12.35) B | 27.71(18.45) B | 38.95(6.42) A | 11.95(4.39) B |

Note: The value in the parentheses is coefficient of variation (COV); the values in the same row of the same specimen type with the same character are the same.
Table 4 shows the WRE of all specimens during weathering test. The WREs of treated sweetgum were significantly higher than those of treated quarter-sawn and treated flat-sawn southern pine during accelerated weathering cycles correspondingly, and the coefficients of variance (COV) of treated sweetgum were all significantly smaller than the ones of treated quarter-sawn and treated flat-sawn southern pine, showing that sweetgum might result in a resin impregnated product with less variation. In addition, the WREs of treated quarter-sawn and treated flat-sawn southern pine decreased with the increase of testing cycles, while the WREs of sweetgum specimens did not change significantly during accelerated weathering test. This was because much more MW PF resins penetrated into sweetgum specimens than into southern pine specimens. Figure 5 shows the images taken by a confocal laser scanning microscope system (LSM 510 by Carl Zeiss Inc. MS, USA) in the cross sections of control and treated southern pine and sweetgum respectively. The PF resins were less visible in southern pine than in sweetgum. And for treated sweetgum specimens, PF resin filled and attached firmly in fibers making the WREs of sweetgum specimens much higher and more stable than treat quarter-sawn and treated flat-sawn southern pine during the accelerated weathering test. Concerning the difference between southern pine samples, at first three weathering cycles, quarter-sawn specimens behaved significantly better than flat sawn specimens.

| Cycle | Treated quarter-sawn southern pine | Treated flat-sawn southern pine | Treated quarter-sawn sweetgum |
|-------|-----------------------------------|-------------------------------|-------------------------------|
| 1     | 59.63(26.74) B                    | 50.57(27.86) C                | 68.37(7.69) A                 |
| 2     | 50.17(14.87) B                    | 47.32(30.71) C                | 64.55(4.57) A                 |
| 3     | 46.15(13.71) B                    | 30.41(71.46) C                | 67.85(4.66) A                 |
| 4     | 39.43(16.27) B                    | 41.03(48.18) B                | 67.94(9.07) A                 |
| 5     | 33.55(23.42) B                    | 33.56(51.38) B                | 69.88(3.89) A                 |
| 6     | 29.27(30.2) B                     | 31.73(58.36) B                | 69.23(3.91) A                 |

Note: The value in the parentheses is coefficient of variations (COV); the values in the same row with the same character are not significantly different.

**Dimensional stability**

The thickness and width of specimens were used to calculate the anti-swelling efficiency (ASE) to evaluate effects of low MW PF resin modification on dimensional stability of specimens. Table 5 shows the ANOVA results of width and thickness changes for specimens evaluated indicating that the specimen type, impregnation treatment, and the two-way interaction of specimen type and impregnating treatment were significant, but the three-way interaction was not significant. Therefore, the further discussions were focused on significant factors.

| Source                                | Width changes | Thickness changes |
|---------------------------------------|---------------|------------------|
|                                       | F  | p | F | p          |
| Specimen type                         | 359.9 | <0.05 | 249 | <0.05 |
| Impregnating treatment                | 320.2 | <0.05 | 262.5 | <0.05 |
| weathering test                       | 7.1 | 0.08 | 0.7 | 0.65 |
| Specimen type * Impregnating treatment| 138.7 | <0.05 | 164.5 | <0.05 |
| Specimen type * weathering test       | 0.9 | 0.54 | 0.2 | 1.0 |
| weathering test*Impregnating treatment| 0.2 | 0.97 | 0.3 | 0.92 |
| Specimen type * weathering test*Impregnating treatment | 4.6 | 0.08 | 1.1 | 0.38 |
Figure 5: Images of (a) control southern pine and (b) treated southern pine (c) control sweetgum and (d) treated sweetgum taken by confocal laser scanning microscope in cross sections (red and green parts refer to PF resin and wood respectively).

Table 6 shows the comparisons of width changes of all specimens treated with low MW PF resin impregnation during accelerated weathering test, suggesting that widths changes of treated specimens were all significantly smaller than those of control specimens accordingly, especially for sweetgum specimens. It suggests that low MW PF resin impregnation significantly improves the dimensional stability in the width direction of tested specimens. And the width changes of all control and treated specimens did not increase significantly with the increasing of accelerated weathering cycle.

Table 6: Comparisons of width changes during accelerated weathering test.

| Cycle | Quarter-sawn southern pine | Flat-sawn southern pine | Quarter-sawn sweetgum |
|-------|---------------------------|-------------------------|-----------------------|
|       | Control | Treated | Control | Treated | Control | Treated |
| 1     | 2.43 (12.8) Ac | 1.33 (11.9) Bb | 6.16 (17.1) Aa | 4.04 (30.8) Ba | 4.02 (12.1) Ab | 1.02 (27.8) Bc |
| 2     | 2.88 (10.7) Ac | 1.75 (12.0) Bb | 6.40 (16.1) Aa | 4.86 (37.0) Ba | 4.72 (15.1) Ab | 0.18 (193.7) Bc |
| 3     | 3.03 (10.8) Ac | 2.06 (25.0) Bb | 6.37 (15.5) Aa | 5.81 (28.9) Aa | 5.49 (16.5) Ab | 0.22 (159.0) Bc |
| 4     | 2.97 (9.1) Ac | 1.95 (23.8) Bb | 6.43 (11.5) Aa | 6.30 (27.0) Aa | 5.85 (17.6) Ab | 0.09 (475.1) Bc |
| 5     | 2.99 (9.3) Ac | 2.44 (27.4) Bb | 6.41 (14.6) Aa | 6.67 (26.0) Aa | 6.26 (16.5) Ab | 0.19 (157.2) Bc |
| 6     | 2.79 (12.7) Ac | 1.96 (37.7) Bb | 6.48 (14.0) Aa | 6.92 (26.3) Aa | 6.58 (16.0) Ab | 0.28 (102.2) Bc |

Note: The value in the parentheses is coefficient of variations (COV); the values in the same row of the same specimen type with the same uppercase letters are not significantly different; the values in the same row of the same treatment not followed by a common lowercase letter are significantly different one from another.

In addition, Table 6 also shows the comparisons of width changes of all specimens for impregnating treatment during accelerated weathering test. In case of control specimens, the width changes of flat-sawn southern pine were significantly bigger than the ones of quarter-sawn southern pine and quarter-sawn sweetgum at the first four wet cycles, but at the last two wet cycles, the difference between width changes of flat-sawn quarter-sawn and quarter-sawn sweetgum was not significant. In case of treated specimens, the width changes of
treated flat-sawn southern pine were significantly bigger than the ones of treated quarter-sawn southern pine and treated quarter-sawn sweetgum. In addition, comparing the difference of width changes between control and treated specimens, the width changes decreased of quarter-sawn sweetgum were much more than flat-sawn and quarter-sawn southern pine, indicating that the effect of low MW PF impregnation was more efficient for sweetgum than southern pine with the procedure described, in order to improve the wood dimensional stability.

Table 7 shows the comparisons of thickness changes of all specimens used in this experiment, indicating that thickness changes of flat-sawn southern pine and quarter-sawn sweetgum were significantly decreased by impregnating PF resin, especially for quarter-sawn sweetgum, while the thickness changes of quarter-sawn southern pine did not decrease significant. The reason why treated quarter-sawn sweetgum had bigger COV was unknown.

Table 7: Comparisons of thickness changes during accelerated weathering test.

| Cycle | Quarter-sawn southern pine | Flat-sawn southern pine | Quarter-sawn sweetgum |
|-------|----------------------------|-------------------------|-----------------------|
|       | Control                    | Treated                 | Control               | Treated                 | Control               | Treated                 |
| 1     | 1.18 (17.6) Ac             | 1.18 (24.0) Aa          | 1.29 (13.7)Ab         | 0.82 (32.4) Bb         | 2.09 (5.5) AA         | 0.44 (32.5) Bc          |
| 2     | 1.16 (20.5) Ac             | 1.12 (30.1) Aa          | 1.19 (17.8)Ab         | 0.86 (32.4) Bb         | 2.05 (12.5) Aa         | 0.43 (73.0) Bc          |
| 3     | 1.11 (22.0) Ac             | 1.12 (30.0) Aa          | 1.18 (19.8)Ab         | 0.89 (30.4) Bb         | 2.20 (13.1) Aa         | 0.35 (125.2) Bc         |
| 4     | 1.08 (17.8) Ac             | 1.11 (31.3) Aa          | 1.15 (16.8)Ab         | 0.90 (35.0) Bb         | 2.22 (13.3) Aa         | 0.36 (105.8) Bc         |
| 5     | 1.12 (16.6) Ac             | 1.11 (30.9) Aa          | 1.14 (21.6)Ab         | 0.87 (36.3) Bb         | 2.18 (12.8) Aa         | 0.38 (92.1) Bc          |
| 6     | 1.07 (16.6) Ac             | 1.14 (28.1) Aa          | 1.09 (16.9)Ab         | 0.90 (31.3) Bb         | 2.23 (11.8) Aa         | 0.45 (81.7) Bc          |

Note: the value in the parentheses is coefficient of variations (COV); the values in the same row of the same specimen type with the same uppercase letters are not significantly different; the values in the same row of the same treatment not followed by a common lowercase letter are significantly different one from another.

Table 7 also shows the thickness changes for impregnating treatment during accelerated weathering test. In the case of control specimens, the thickness changes of quarter-sawn sweetgum were significantly bigger than the ones of flat-sawn and quarter-sawn southern pine during accelerated weathering test. In the case of treated specimens, the thickness changes of treated quarter-sawn southern pine were significantly bigger than treated flat-sawn southern pine and treated quarter-sawn sweetgum during accelerated weathering test. In addition, comparing the difference of thickness changes between control and treated specimens, the differences of quarter-sawn sweetgum were much bigger than those of southern pine, indicating that the PF treatment was the most effective on quarter-sawn sweetgum, and then on flat-sawn southern pine and quarter-sawn southern pine, in reducing thickness changes.

Table 8 shows the ASEs of all treated specimens during accelerated weathering tests. ASE values are interpreted as followings: ASE > 0 means that low MW PF treatment has a positive effect on anti-swelling property; ASE < 0 means that the treatment has a negative effect on anti-swelling property; ASE = 0 means the treatment has no effect on anti-swelling property, as compared with control samples. The higher the ASE values after treatment are, the greater the mentioned positive effects are.

The low MW PF resin treatment improved the ASE or the dimensional stabilities of quarter-sawn sweetgum and southern pine specimens (Table 8). ASEs of all treated samples decreased with the increase of weathering cycles, especially for flat-sawn southern pine specimens, while ASE values of treated quarter-sawn sweetgum did not change intensively. Meanwhile, the ASE values of treated quarter-sawn sweetgum were much higher than the ones of treated quarter-sawn and flat-sawn southern pine.

Table 8: Comparison of ASEs of low MW PF resin treated specimens during accelerated weathering test.

| Specimen type             | 1   | 2   | 3   | 4   | 5   | 6   |
|---------------------------|-----|-----|-----|-----|-----|-----|
| Treated quarter-sawn pine | 15.64 | 20.54 | 11.38 | 11.97 | 5.11 | 3.77 |
| Treated flat-sawn pine    | 39.54 | 18.81 | 3.85 | -3.78 | -6.45 | -9.50 |
| Treated sweetgum          | 49.30 | 46.56 | 40.81 | 36.75 | 35.18 | 34.69 |
Above comparisons and analyses confirm that low MW PF resin impregnation improved the volumetric dimensional stability of sweetgum and southern pine, especially for sweetgum, showing that sweetgum was a better wood species for described low MW PF resin impregnation, in terms of volumetric dimensional stability.

Crack formation

Table 9 shows the photos of cracks at ends and on surfaces of all control and treated specimens after accelerated weathering tests. Based on observations, all cracks at ends of both specimens were initiated from latewood and then developed along radial direction. Long cracks were generated when two or more short cracks initiated from latewood and connected in radial direction. The cracks on radial surface initiated primarily at borders of earlywood and latewood. Cracks on tangential surface occurred frequently in both earlywood and latewood, which matched with the observations of Sandberg (1999).

Table 9: Photos of cracks at ends and on surfaces of specimens.

| Specimen type                  | PF resin     | End of specimen | Surface of specimen |
|-------------------------------|--------------|-----------------|---------------------|
| Quarter-sawn southern pine    | Control      | ![Image]        | ![Image]            |
|                               | Treated      | ![Image]        | ![Image]            |
| Flat-sawn southern pine       | Control      | ![Image]        | ![Image]            |
|                               | Treated      | ![Image]        | ![Image]            |
| Sweetgum                      | Control      | ![Image]        | ![Image]            |
|                               | Treated      | ![Image]        | ![Image]            |

To further study the effect of low MW PF resin treatment on southern pine and sweetgum, all cracks on exposed surfaces and at ends of all specimens were compared. The influence of low MW PF resin treatment on crack formation, i.e., number of crack length longer than 5 mm, and total length of cracks on exposed surfaces and at ends of all tested specimens, was analyzed by ANOVA. Table 10 show the ANOVAs of cracks on exposed surfaces and at ends of specimens. The ANOVA results (Table 10) for cracks on exposed surfaces of specimens indicate that the two-way interaction was not significant, in terms of total crack length. However, the ANOVA results (Table 10) for cracks on exposed ends of specimens indicating that the two-way interaction was significant, so further discussion and analysis were focused on the significant factors.

Table 10: Summary of ANOVA results for cracks on exposed surfaces of specimens.

| Sources                        | Cracks on surfaces | Cracks at ends |
|--------------------------------|-------------------|----------------|
|                                | Number of crack > 5 mm | Total length of cracks (mm) | Number of crack > 5 mm | Total length of cracks (mm) |
| Specimen type                  | F   | p     | F   | p     | F   | p     | F   | p     |
| Impregnating treatment         | 93.3 | <0.05 | 1.27 | <0.05 | 34.9 | <0.05 | 54.9 | <0.05 |
| Specimen type*Impregnating treatment | 173.5 | <0.05 | 85.6 | <0.05 | 2.6 | 0.12 | 7.8 | <0.05 |

Table 11 shows the comparison of cracks on exposed surfaces of all tested specimens for impregnating treatment, indicating that the treated specimens significantly generated more cracks than control specimens.
Table 11: Comparison of cracks on exposed surfaces of all specimens.

| Specimen type          | Impregnating treatment | Number of cracks >5 mm | Total length of cracks (mm) |
|------------------------|------------------------|-------------------------|-----------------------------|
| Quarter-sawn southern pine | Control                | 1 (1.67) Bb             | 25.98 (47.47) Bb            |
|                        | Treated                | 8 (2.58) Ac             | 735.80 (454.66) Ac          |
| Flat-sawn southern pine | Control                | 16 (4.28) Ba            | 531.69 (202.71) Ba          |
|                        | Treated                | 24 (2.77) Aa            | 1230.67 (261.58) Aa         |
| Quarter-sawn sweetgum  | Control                | 0 Bc                    | 0 Bc                        |
|                        | Treated                | 21 (3.06) Ab            | 861.08 (136.16) Ab          |

Note: The value in the parentheses is coefficient of variations (COV); the values in the same column of the same specimen type with the same uppercase letters are the same; the values in the same column of the same treatment not followed by a common lowercase letter are significantly different one from another.

Table 11 also shows the comparison of cracks on exposed surfaces of all tested specimens. Concerning control specimens, in terms of the number of cracks longer than 5 mm and the total length of cracks, the quarter-sawn sweetgum was the lowest, followed by the quarter-sawn southern pine and then the flat-sawn southern pine. Concerning treated specimens, quarter-sawn had the lowest the numbers of the cracks longer than 5 mm and total length of cracks, similar to the others’ previous studies (Coupe and Watson 1967). These show that untreated sweetgum might be a good species for exterior application and quarter-sawn southern pine a good candidate. Further research is needed to reach a conclusive statement.

Table 12 shows the comparison of cracks at exposed ends of all tested specimens, indicating that the effect of impregnating treatment on cracks on exposed ends of all specimens were varied. For sweetgum, the number of crack with a length longer than 5mm and the total length of the treated quarter-sawn specimens were significantly greater than those of control quarter-sawn specimens, but for quarter-sawn southern pine, low MW PF treatment can reduce the cracks at exposed ends significantly. In flat-sawn southern pine specimens, treated specimens had significantly longer cracks than control ones. These indicate that the low MW PF resin treatment might be effective in reducing the end cracks of quarter sawn southern pine. Further research is needed to reach a conclusive statement.

Table 12: Comparison of cracks at exposed ends of all specimens.

| Specimen type          | Impregnating treatment | Number of cracks > 5 mm | Total length of cracks (mm) |
|------------------------|------------------------|-------------------------|-----------------------------|
| Quarter-sawn southern pine | Control                | 6 (3,13) Ac             | 52.30 (22.78) Ac            |
|                        | Treated                | 3 (4,10) Bc             | 28.17 (38.48) Bc            |
| Flat-sawn southern pine | Control                | 8 (1,79) Bb             | 73.04 (14.65) Bb            |
|                        | Treated                | 11 (2,07) Ab            | 122.97 (20.17) Ab           |
| Quarter-sawn sweetgum  | Control                | 12 (1,90) Ba            | 128.65 (27.53) Ba           |
|                        | Treated                | 16 (2,71) Aa            | 179.76 (26.90) Aa           |

Note: The value in the parentheses is coefficient of variations (COV); the values in the same column of the same specimen type with the same uppercase letters are not significantly different; the values in the same column not followed by the same lowercase letters are significantly different one from another.

Table 12 also shows the comparisons of cracks at exposed ends of all specimens for specimen type, indicating that the number of cracks longer than 5 mm, and total length of cracks of quarter-sawn sweetgum were significantly bigger and longer than those of control quarter-sawn southern pine and treated quarter-sawn southern pine both for control and treated specimens. This showed that the sweetgum generated cracks at the end more easily than southern pine. In addition, the flat-sawn southern pine specimens had more cracks at exposed ends than those of quarter-sawn southern pine specimens, which matched with others’ findings (Coupe and Watson 1967).

In summary, the low MW PF resin treatment seemed to have a negative effect on anti-cracking property.
which means that the low MW PF resin treated specimens generated more cracks than control specimens subjected to accelerated weathering tests.

CONCLUSIONS

Impregnation of low MW PF resin improved the volumetric dimensional stability of sawn sweetgum and southern pine specimens. The low MW PF resin treated quarter-sawn sweetgum had higher ASE than treated quarter-sawn southern pine. Impregnation of low MW PF resin into wood has varied results. The treated sweetgum and southern pine species had more surface cracks than control ones. Concerning the cracks at the ends of specimens, quarter-sawn sweetgum had the longest cracks. Within the southern pine specimens, quarter-sawn specimens had fewer and shorter cracks than flat-sawn specimens. Among treated specimens, quarter-sawn southern pine had the least and shortest cracks.

This may show that sweetgum is a better wood species for impregnating low MW PF to improve wood dimensional stability. Impregnation low MW PF resin helped reducing the end crack of quarter-sawn southern pine. Further research should work on how to reduce wood surface cracks.

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REFERENCES

Altgen, M.; Adamopoulos, S.; Viikari, J.A.; Hukka, A.; Teri, T.; Militz, H. 2012. Factors influencing the crack formation in thermally modified wood. In Proceedings of the 6th European Conference on Wood Modification, Ljubljana, Slovenia. pp: 149-158.

American Society for Testing and Materials. ASTM. 2017. Standard Practice for Accelerated Weathering of Fire-Retardant-Treated Wood for Fire Testing. ASTM D2898-10. ASTM: West Conshohocken, PA, USA. https://www.astm.org/Standards/D2898.htm

Biziks, V.; Bicke, S.; Militz, H. 2019. Penetration depth of phenol-formaldehyde (PF) resin into beech wood studied by light microscopy. *Wood Science and Technology* 53(1): 165-176. http://doi.org/10.1007/s00226-018-1058-2

Cai, Y.; Wu, Y.; Yang, F.; Gan, J.; Wang, Y.; Zhang, J. 2021. Wood sponge reinforced with polyvinyl alcohol for sustainable oil-water separation. *ACS Omega* 6(19): 12866-12876. https://doi.org/10.1021/acsomega.1c01280

Coupe, C.; Watson, R.W. 1967. Fundamental aspects of weathering. In Proceedings from the Annual Seventeenth Convention of British Wood Preservation Association, London, England. pp: 37-49.

Forest Products Laboratory. 2010. *Wood handbook-Wood as an engineering material*. General Technical Report FPL-GTR-190. Centennial Edition, Department of Agriculture, Forest Service: Madison, WI., USA. Chapter 2, pp: 508. https://www.fpl.fs.fed.us/docs/fplgtr/fpl_gtr190.pdf

Furuno, T.; Imamura, Y.; Kajita, H. 2004. The modification of wood by treatment with low molecular weight phenol-formaldehyde resin: a properties enhancement with neutralized phenolic-resin and resin penetration into wood cell walls. *Wood Science and Technology* 37(5): 349-361. http://doi.org/10.1007/s00226-003-0176-6

Gabrielli, C.P.; Kamke, F.A. 2010. Phenol-formaldehyde impregnation of densified wood for improved dimensional stability. *Wood Science and Technology* 44(1): 95-104. http://doi.org/10.1007/s00226-009-0253-6
Comparative study on weathering durability property: Hu and Wan

Ghani, A.; Ashaari, Z.; Lee, S.H.; Bakar, E.S.; Bawon, P. 2017. A comparison between the properties of low and medium molecular weight phenol formaldehyde resin-treated laminated compreg oil palm wood. *International Forestry Review* 19: 1-11. http://doi.org/10.1505/146554817828562305

Hansmann, C.; Deka, M.; Wimmer, R.; Gindl, W. 2006. Artificial weathering of wood surfaces modified by melamine formaldehyde resins. *European Journal of Wood and Wood Products* 64(3): 198-203. http://doi.org/10.1007/s00107-005-0047-y

Huang, Z.; Wang, Z.; Xiao, Y.F.; Xie, Y.J. 2019. Weathering performance of wood modified with an agent containing N-methylol resin/sucrose. *Journal of Forestry Engineering* 4(5): 60-69. http://doi.org/10.13360/j.issn.2096-1359.2019.05.009

Islam, M.S.; Hamdan, S.; Rusop, M.; Rahman, M.R.; Ahmed, A.S.; Idrus, M.A.M.M. 2012. Dimensional stability and water repellent efficiency measurement of chemically modified tropical light hardwood. *BioResources* 7(1): 1221-1231. https://ojs.cnrs.edu/index.php/BioRes/article/view/BioRes_07_1_1221_Islam_HRRAMI_Dimen_Stabil_Water_Repell_Modified_Wood/1393

Jirous-Rajkovic, V.; Mikletic, J. 2019. Heat-treated wood as a substrate for coatings, weathering of heat-treated wood, and coating performance on heat-treated wood. *Advances in Materials Science and Engineering* 2019:1-9. http://doi.org/10.1155/2019/8621486

Kielmann, B.C.; Mai, C. 2016a. Application and artificial weathering performance of translucent coatings on resin-treated and dye-stained beech-wood. *Progress in Organic Coatings* 95: 54-63. http://dx.doi.org/10.1016/j.porgcoat.2016.02.019

Kielmann, B.C.; Mai, C. 2016b. Natural weathering performance and the effect of light stabilizers in water-based coating formulations on resin-modified and dye-stained beech-wood. *Journal of Coatings Technology and Research* 13(6): 1065-1074. http://dx.doi.org/10.1007/s11998-016-9818-0

Li, J.; Ma, E. 2021. Influence of heat treatment and delignification on hygroscopicity limit and cell wall saturation of southern pine wood. *Journal of Forestry Engineering* 6(3): 61-68. http://doi.org/10.13360/j.issn.2096-1359.201911036

Lu, H.; Zhang, Y.; Zhang, S.; Huang, Y.; Pan, M. 2021. Preparation of flame-retardant plywood by PEI/APP modified urea-formaldehyde resin and its properties. *Journal of Forestry Engineering* 6(01): 44-49. http://dx.doi.org/10.13360/j.issn.2096-1359.201911036

Nasir, V.; Nourian, S.; Avramidis, S.; Cool, J. 2019. Prediction of physical and mechanical properties of thermally modified wood based on color change evaluated by means of “group method of data handling” (GMDH) neural network. *Holzforschung* 73(4): 381-392. http://doi.org/10.1515/hf-2018-0146

Sandberg, D. 1999. Weathering of radial and tangential wood surfaces of pine and spruce. *Holzforschung* 53(4): 355-364. http://doi.org/10.1515/HF.1999.059

Sandberg, D.; Söderström, O. 2006. Crack formation due to weathering of radial and tangential sections of pine and spruce. *Wood Materials Science and Engineering* 1(1): 12-20. http://doi.org/10.1080/17480270600644407

Silva, B.C.; Trevisan, H.; Garcia, R.A. 2020. Effect of the thermal modification and nano-ZnO impregnation on the deterioration of Caribbean pine wood. *Maderas-Ciencia y Tecnologia* 22(4): 569-576. https://dx.doi.org/10.4067/S0718-221X2020005000415

Statistical Product and Service Solutions Software. SPSS. 2013. SPSS Version 22. IBM: USA. https://www.ibm.com/analytics/spss-statistics-software

Yan, X.; Yin, T.; Li, H. 2021. Preparation of urea-formaldehyde resin coated fluororesin microcapsules and its effect on the performance of water-borne coatings on wood surface. *Journal of Forestry Engineering* 4(5): 167-173. http://doi.org/10.13360/j.issn.2096-1359.202005030
Tu, J.C.; Zhao, D.; Zhao, J. 2020. Experimental study for determining method of cracking load of wooden beams with LT crack. *Journal of Forestry Engineering* 5(3): 149-154.  http://doi.org/10.13360/j.issn.2096-1359.201907006

Wan, H.; Dahlen, J.; Mao, A.; Sites, L.; Rowlen, A.; Miller, G.; McClendon, B.; Liu, M.; Liu, X.; Nicholas, D. 2017. Evaluation of the performance of composite wood decking bonded with phenol resorcinol formaldehyde and polyurethane adhesives after accelerated aging tests. *Forest Products Journal* 67(1): 112-119.  http://doi.org/10.13073/FPJ-D-16-00020

Wan, H.; Kim, M. 2008. Distribution of phenol-formaldehyde resin in impregnated southern pine and its effects on wood stabilization. *Wood and Fiber Science* 40(2): 181-189.  https://wfs.swst.org/index.php/wfs/article/view/111

Wang, X.; Meng, J.; Cheng, Z.; Guang, H. 2020. Research process of durable superhydrophobic wood surface. *Journal of Forestry Engineering* 5(3): 13-20.  http://doi.org/10.13360/j.issn.2096-1359.201910013

Wu, S.S.; Tao, X.; Xu, W. 2021. Thermal conductivity of poplar wood veneer impregnated with graphene/polyvinyl alcohol. *Forests* 12: 777.  https://doi.org/10.3390/f12060777

Yan, X.; Li, W.; Han, Y.; Yin, T. 2022. Preparation of melamine/rice husk powder coated shellac microparticles and effect of different rice husk powder content in wall material on properties of wood waterborne primer. *Polymers* 14(1):56.  https://doi.org/10.3390/polym14010072

Zivkovic, V.; Prsa, I.; Turkulin, H.; Sinkovic, T.; Rajkovic, V.J. 2008. Dimensional stability of heat treated wood floorings. *Drvna Industrija* 59(2): 69-73.  https://hrnak.srce.hr/file/40067