

PERFORMANCE OF THERMALLY MODIFIED SCOTS PINE TREATED WITH COMBINATIONS OF SOME MODIFYING CHEMICALS

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Abstract. In this study, low-temperature thermally-modified (at 140°C) Scots pine (*Pinus sylvestris* L.) wood samples were impregnated with either one or more modifying agents, including the brown colorant, paraffin wax emulsion (PWE), and an organic preservative (4,5-dichloro-2-octyl-2H-isothiazol-3-one, DCOIT) microemulsion. All wood samples were assessed in dimensional stability, water absorption, the modulus of rupture (MOR), decay and mold resistance, and their weathering performance under both lab-scale and outdoor exposure. The results showed that 1) the treating groups with PWE showed obvious lower water absorption, and the lowest value appeared in the group treated with brown colorant and PWE (BC-PWE); 2) thermal modification improved the dimensional stability of wood, whereas all the further treatments except the group impregnated with PWE slightly counteracted the effect of thermal modification on dimensional stability after water soaking; 3) the MOR of thermally-modified sample showed little change after further treatments; 4) and thermally-modified wood treated with BC-PWE-DCOIT showed the optimal weathering performance with the least color change and the best mold resistance.

Keywords: Wood, thermal treatment, DCOIT, colorant, paraffin wax emulsion.

INTRODUCTION

Wood surface without protection exposed to outdoor conditions is susceptible to weathering and biological attack, which could cause a complex set of physical and chemical changes, including discoloration, decay, reduction in dimensional stability, and mechanical properties (Popescu et al 2011; Garcia et al 2014). Based on

these, protection methods were investigated extensively to improve the comprehensive properties of wood. Impregnation with chemicals is one of these methods. Unfortunately, some wood protection chemicals used previously were found to be highly toxic to humans and hence, the present focus has been to develop nontoxic approaches for the wood protection.

Thermal treatment under high temperature (180-260°C) can improve the dimensional stability and decay resistance of wood without requiring any

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toxic chemicals (Tolvaj et al 2014; Todaro et al 2015). Besides, the darkened color from thermal treatment wood presents another attractive aspect to some customers. However, most of the mechanical properties, such as the flexural properties of thermally-modified wood, including modulus of rupture (MOR) and modulus of elasticity, decrease with increasing thermal treatment temperature (Yildiz et al 2013; Tiryaki and Hamzaçebi 2014; Tomak et al 2014). To balance the reduced mechanical performance and the improved dimensional stability and decay resistance of thermally modified wood, thermal modification under a relatively low temperature (140°C) is considered to be an acceptable choice.

Although the comprehensive property of wood after low-temperature thermal modification was improved compared with the unmodified ones, researchers still found that thermal modification does not induce resistance to damage caused by UV radiation (Srinivas and Pandey 2012). Various weathering problems would appear when the thermally-modified wood is exposed outdoors for a long period of time, such as color fading, surface checking, decay, and mildew (Kocaefe et al 2008; Huang et al 2012; Tomak et al 2014), which definitely influence its service life in terms of aesthetic and mechanical considerations, and finally influence the commercialization of thermally-modified wood. To solve the weathering problem and further improve the biological performance, some nontoxic protection chemicals could be used in combination with thermal treatment on wood.

The combination used in this study is colorant, water repellent, and wood preservative. Adding colorant is a convenient method to darken surface color of low-temperature thermally-modified wood. Kielmann and Mai (2016) found that combined modification of resin with staining could improve the weathering performance and maintain the color stability of the modified wood. Because the surface color of thermally-modified wood with low temperature was not as dark as those modified at higher temperatures, brown colorant was chosen to make thermally-modified wood surface darker and possibly benefit the

weathering performance. In addition, paraffin wax emulsion (PWE) was found to be widely used in industrial applications for improving the water repellency of wood (Lesar et al 2011a). For example, Evans et al (2009) added 2.5% wax emulsion into wood and decreased its water absorption significantly. Meanwhile, addition of wax emulsion could also increase the photostability (Lesar et al 2011b) and decay resistance of wood (Lesar and Humar 2011). The applied wax emulsions can slow down wood fungi to grow because of the film/barrier formed in the cell lumina and on the surface of the specimens (Lesar and Humar 2011). As a nontoxic modifier, PWE was considered to be a good choice for modification combined with thermal treatment. For outdoor application of wood, copper-based preservatives are also commonly used to efficiently withstand the attacks by fungi and mold, but they turn the wood surface to green and somehow pose threats to the environment (Gadd and Griffiths 1977; Williams 2005). Kristin et al (2005) found that copper-based preservatives were inclined to copper leaching and therefore posed aquatic toxicity. In view of this, an organic preservative, 4,5-dichloro-2-octyl-2H-isothiazol-3-one (DCOIT), was used in its microemulsion formulation to increase the decay resistance of thermally-modified wood.

This study focuses on improving the performance of thermally-modified Scots pine (*Pinus sylvestris* L.) by using one or combinations of different modifying chemicals, including Brown colorant (BC), PWE, and DCOIT microemulsion. The weathering performance, dimensional stability, flexural properties, decay, and mold resistance were discussed.

MATERIALS AND METHODS

Samples and Chemical Preparation

Finland Scots pine (*Pinus sylvestris* L.) sapwood with an annual ring width of 2.2 mm and a density of $0.45 \text{ g}\cdot\text{cm}^{-3}$, was provided by Guangzhou Fengsheng Building Materials Co., Ltd. (Guangzhou, China). The dimension of the air-dried mother board is 500 mm (longitudinal) \times 110 mm

(tangential) \times 20 mm (radial). Half (ten boards) of the samples were thermally modified at 140°C for a total duration of 25 h under steam atmosphere with a pressure of 0.7 MPa heating by oil heat electric heater; the remaining ½ were not thermally modified. Samples with different dimensions that were used in following test and characterization as described below were cut from these mother boards, including 10 thermally modified wood and 10 unmodified wood.

Brown Colorant BF-TB 5 (pH = 3.5-5.5) was obtained from Rialco Co., Ltd. (Bradford, British), which is a dark brown dye formulated for pressure treatment of wood. The addition amount of colorant was 1% based on weight.

The water repellent, PWE, was prepared in a laboratory and the solid content of the emulsion was 40%, with an average particle size of 220 nm. Before the impregnation procedure, the emulsion was diluted to 2% (w/w) solid content at 20°C with distilled water.

The organic preservative (DCOIT) microemulsion was prepared in the laboratory and its addition amount was 0.1% based on weight.

Chemical Analysis

The acid-insoluble lignin and holocellulose content of thermally modified and unmodified wood powder were determined in accordance with the Chinese Standard GB/T 2677.8 (1994) and GB/T 2677.10 (1995), respectively. Nitric-acetic/ethanol method was used to obtain the cellulose content, and the hemicellulose content was calculated by subtracting the cellulose content from the holocellulose content. Polar and water soluble extractives were removed firstly using hot water followed by mixture of ethanol and acetone at a ratio of 1:2. After removal of extractives, the sawdust was air-dried until constant weight was reached. Holocellulose was determined by putting wood powder into conical flask containing mixture of glacial acetic acid, sodium chlorite, and distilled water. Reaction was carried out at 75°C for several hours until the powder became white. Lignin and cellulose

content were determined depending on the reaction of wood powder with sulphuric acid and nitric-acetic/ethanol solution, respectively.

Modification Process

The wood samples were impregnated using a full-cell process. The treating groups are listed in Table 1. The procedure started with a vacuum at -0.1 MPa for 30 min, and followed by pressure at 1.5 MPa for 45 min. After the pressure was removed, the samples were taken out and wiped with tissue paper before oven-drying (60°C) to a constant mass. All wood samples were then cut into different dimensions as required for the following characterizations and tests.

Weathering Test

Weathering performance of wood samples was investigated in both lab-scale and outdoor weathering conditions. In lab-scale weathering test, samples with a dimension of 70 mm (L) \times 50 mm (T) \times 5 mm (R) were tested using an accelerated weathering tester (QUV/Spray, Q-Lab Corporation) according to ASTM (2004). The weathering cycle consisted of UV radiation at 60°C for 8 h and condensation at 50°C for 4 h. The UVA-340 lamps gave a simulation of sunlight in the critical short wavelength region from 365 nm down to 295 nm. The UV-irradiation intensity was $0.89 \text{ W} \cdot \text{m}^{-2}$ at 340 nm wavelength. The UVA-340 lamps were calibrated every 500 h. The irradiation was interrupted after 72, 168, 240, 336, 672, and 1008 h of exposure, and each set of samples were taken out for evaluating surface properties, such as color parameters and surface

Table 1. Labeling of treating groups.

Thermally-modified (T) or unmodified (U) ^a	Brown colorant	Paraffin wax emulsion	DCOIT microemulsion
T/U -Control	—	—	—
T/U -BC	✓	—	—
T/U -BC-PWE	✓	✓	—
T/U -BC-DCOIT	✓	—	✓
T/U -BC-PWE-DCOIT	✓	✓	✓

BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

pictures as discussed in the section below. All the samples were stored at room conditions until they were subjected to the characterization tests.

All samples for outdoor weathering test were machined into the rectangular boards of 200 mm (L) \times 100 mm (T) \times 20 mm (R) and placed on a south-facing and 45° inclined metal framework with a height of 10 cm above the ground on the Beijing Forestry University campus, Beijing, China (40°00'67" latitude and 116°35'19" longitude). The framework was installed on the outside and completely exposed to sun, rain, and wind etc. The samples were periodically collected for evaluating weathering performance at 10-d intervals over the whole 100-d period. At each interval, weathering performance of the samples was immediately determined by recording color parameters and surface pictures of the exposed surfaces before being put back.

Color Measurement

The surface color of samples was measured with a chroma meter (Datacolour DF 110, Shenzhen 3NH Technology Co., Ltd, Shenzhen, China) according to the CIE LAB color system. Parameters of L^* , a^* , and b^* were measured at five specific locations on the surface of each specimen at intervals and the average value was calculated. L^* represents the lightness coordinate and varies from 100 (white) to 0 (dark). Parameter a^* represents red (+ a) to green ($-a$) coordinate, whereas b^* represents yellow (+ b) to blue ($-b$) coordinate. Parameters of ΔL^* , Δa^* , Δb^* , and ΔE^* were calculated according to the following Eqs (1)-(4).

$$\Delta L^* = L_i^* - L_i^* \quad (1)$$

$$\Delta a^* = a_i^* - a_i^* \quad (2)$$

$$\Delta b^* = b_i^* - b_i^* \quad (3)$$

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

where L_i^* , a_i^* , and b_i^* are parameters measured before weathering; L_i^* , a_i^* , and b_i^* are parameters

measured after weathering; ΔL^* , Δa^* , Δb^* , and ΔE^* represent changes in lightness, red/green chromaticity, yellow/blue chromaticity, and the total color change, respectively.

Dimensional Stability and Water Absorption

Dimensional stability was determined by measuring volumetric swelling percentage and water absorption of the specimens (20 mm \times 20 mm \times 20 mm) in accordance with the Chinese Standards GB/T 1934.1 (2009), which were immersed in a water bath at a controlled temperature of 20°C. During the test, the water in the water bath was recirculated continuously to maintain the required temperature (20°C). The specimens were weighed at intervals until the weight changes of the last two test specimens that were placed in the tank were less than 0.1%, it was assumed that the specimens had reached equilibrium, which took 960 h. Then, the dimensions in length (longitudinal), width (tangential), and thickness (radial) directions were measured to an accuracy of ± 0.01 mm. The water absorption rate (WA, %) and volumetric swelling rate (S_{vol} , %) were determined by the Eqs (5) and (6), respectively.

$$WA = \frac{m - m_0}{m_0} \times 100\% \quad (5)$$

$$S_{vol} = \frac{V_{max} - V_0}{V_0} \times 100\% \quad (6)$$

where m_0 is the oven-dry weight of the specimens before water immersion (g) and m is the weight of the specimens after the water immersion (g). V_0 represents the oven-dry volume of the specimens before the water immersion (mm^3), and V_{max} represents the volume of the specimens after the water immersion (mm^3).

Flexural Property

The MOR in bending was determined at a test speed of $1 \text{ mm} \cdot \text{min}^{-1}$ according to the Chinese Standards GB/T 1936.2 (2009) with a universal testing machine. The specimens measuring

300 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) were conditioned in a humidity chamber at 20°C and 65% RH (RH) before testing.

Decay Resistance

The decay resistance of treated wood was determined by laboratory soil-block cultures according to AWWA (2008). Wood blocks measuring 10 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) were exposed to brown-rot fungi (*Gloeophyllum trabeum*) for a total duration of 4 mo. The mycelium twining around wood blocks were put off and the weight loss (WL, %) was calculated according to the following equation.

$$WL = \frac{W_1 - W_2}{W_1} \times 100\% \quad (7)$$

where W_1 and W_2 represents the oven-dry weight of wood specimens before and after exposure to fungi.

Mold Resistance

The mold (*Aspergillus niger*) resistance tests were carried out in accordance with the Chinese Standards GB/T 18261 (2000). After incubation in a conditioning chamber at $26 \pm 2^\circ\text{C}$ and 85% RH for 4 wk, the infection index of wood specimen was evaluated by visual inspection as showed in Table 2.

RESULTS AND DISCUSSION

Chemical Analysis of Thermally-Modified Wood

Changes of wood chemical composition after thermal modification are shown in Table 3. Slight

Table 2. Evaluation methods of infection index on wood surface in accordance with the Chinese standard GB/T 18261 (2000).

Infection index	Description
0	No hyphae on wood surface
1	Hyphae infected ratio is smaller than 1/4
2	Hyphae infected ratio is between 1/4 and 1/2
3	Hyphae infected ratio is between 1/2 and 3/4
4	Hyphae infected ratio is larger than 3/4

changes of wood components can be found after thermal modification at 140°C. Content of cellulose and hemicellulose decreased after thermal modification, whereas acid-insoluble lignin increased slightly because of the degradation of polysaccharide during high temperature (Esteves et al 2013), thus leading to an increase in extractives and other components. In consideration of these changes, mechanical property of thermally-modified wood is expected to be influenced insignificantly.

Color Changes of Wood Surface during Lab-Scale Weathering Test

Figure 1 depicts the tangential surface images of control and modified Scots pine during lab-scale artificial weathering test. Compared with T-control group, brown colorant-treated thermally-modified sample (T-BC) had a darker color before weathering. It can be observed that the color of T-control darkened, whereas all dyed groups became lighter-colored with increasing exposure time. After 1008 h of irradiation, the combination of brown colorant and PWE presented the most significant improvement on color stability during weathering, followed by the group of T-BC-DCOIT and T-BC-PWE-DCOIT. This phenomenon was due to the presence of water-proof PWE, which can adhere to the interior wood surfaces and hinder the degradation products from leaching out (Wang et al 2015).

To statistically analyze the color change of wood surface during weathering, CIE Lab system was used and the main parameters are shown in Fig 2. Lightness (L^*) of T-control sample decreased sharply from 74 to 48, suggesting that the surface of thermally-modified wood darkened significantly, as can be seen in Fig 1. In contrary, the lightness change of thermally-modified wood with further modifications (T-BC; T-BC-PWE;

Table 3. Chemical composition of wood before (*U*-Control) and after thermal modification (*T*-Control).

	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Others (%)
<i>U</i> -Control	48.62	22.67	24.75	3.96
<i>T</i> -Control	47.24	22.01	25.09	5.66

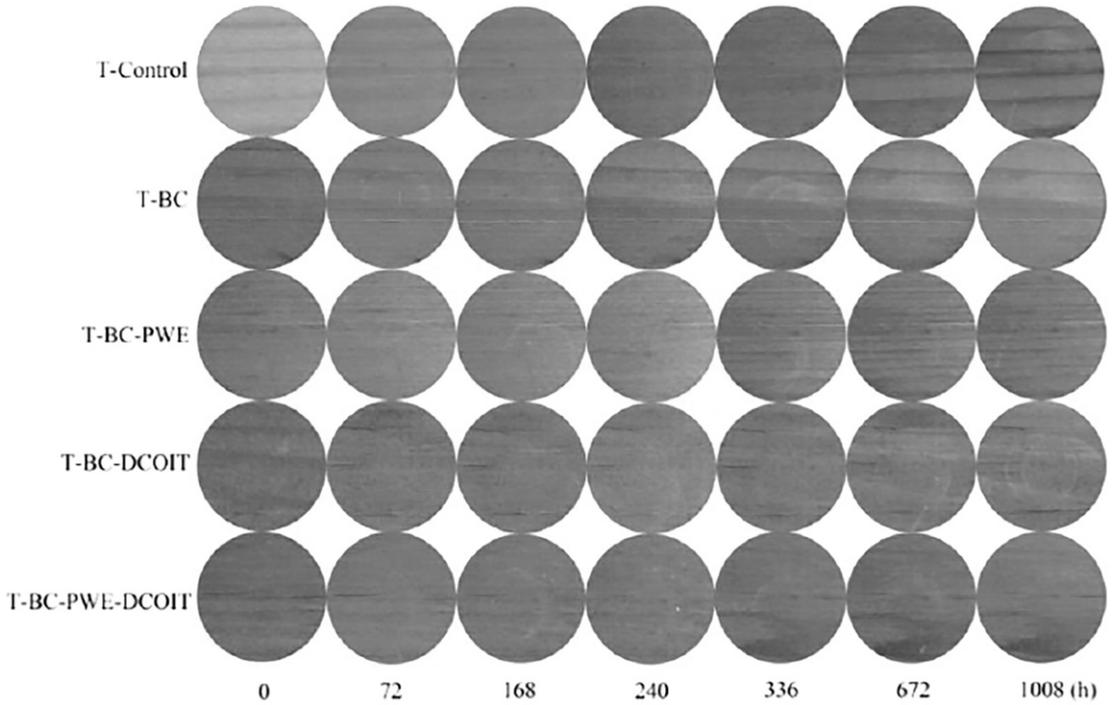


Figure 1. Surface image of thermally-modified wood during laboratory-scale artificial weathering test. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

T-BC-DCOIT; T-BC-PWE-DCOIT) was insignificant. The total color change (ΔE) is the most important parameter to evaluate color stability. Addition of colorant and wax to thermally

modified wood proved to be the most effective way in improving color stability of treated wood. In accordance with the visual observation results, the total surface color change of brown colorant

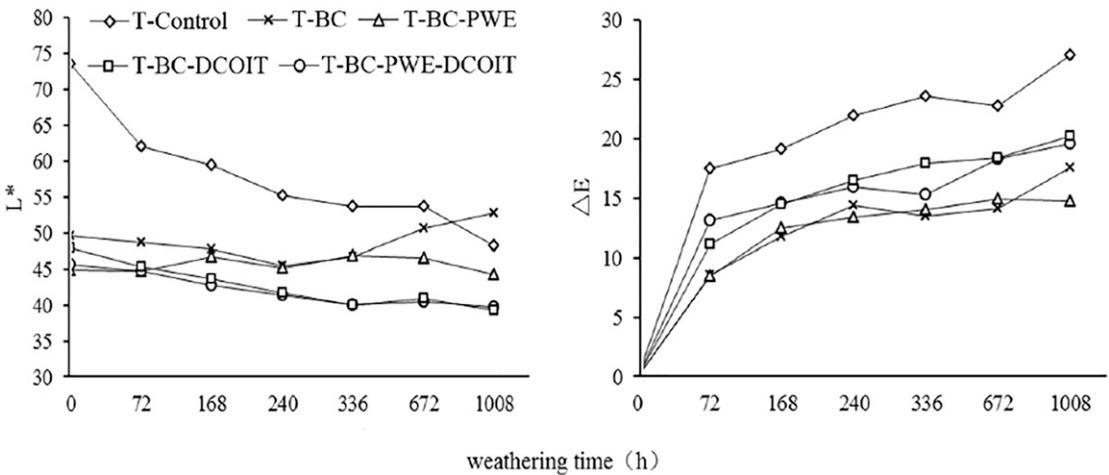


Figure 2. Color changes of thermally-modified wood during laboratory-scale artificial weathering test. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

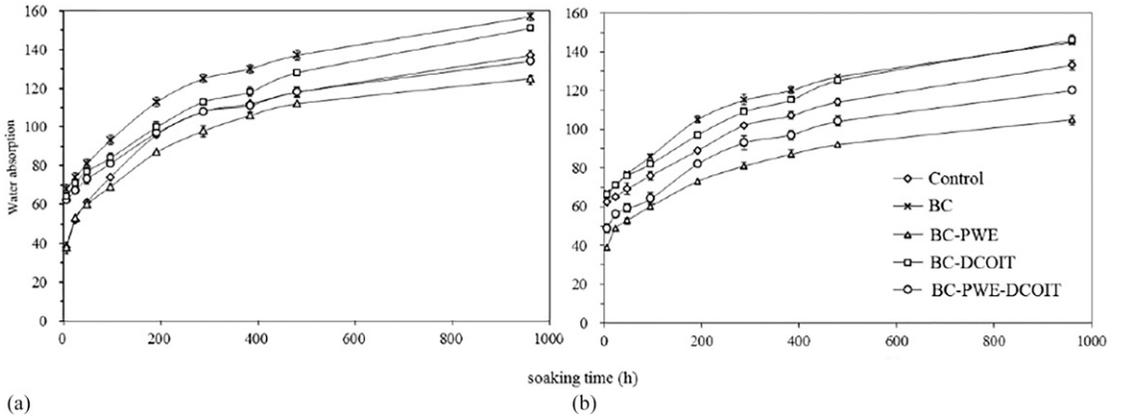


Figure 3. Water absorption rate (WA) of untreated (a) and thermally-modified (b) Scots pine treated with different modifying chemicals during water soaking. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

and PWE treated thermally modified group was the most insignificant.

Dimensional Stability

Water absorption and volume swelling rate of untreated and thermally-modified wood were

shown in Figs 3 and 4. The treating groups with PWE showed obvious lower water absorption, and the lowest value appeared in T-BC-PWE group. This phenomenon indicates that PWE improves water resistance of wood surface significantly, which may result in improvement of color stability by hindering the degradation

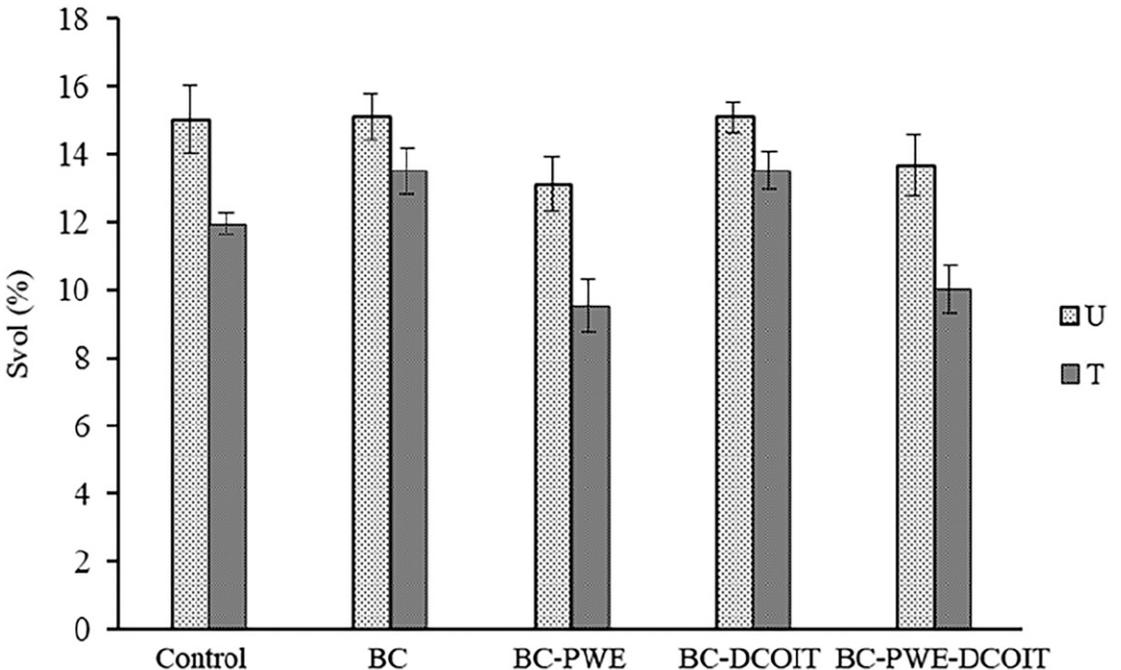


Figure 4. Volumetric swelling rate (S_{vol}) of untreated (*U*) and thermally-modified (*T*) Scots pine-treated with different modifying chemicals after 24 h of water soaking. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

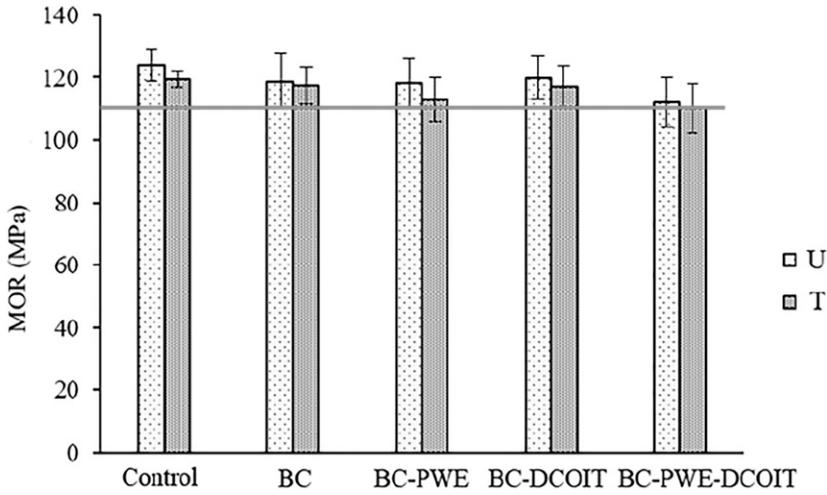


Figure 5. Modulus of rupture (MOR) strength of untreated (*U*) and thermally-modified (*T*) Scots further treated with modifying chemicals. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

products from leaching out (Wang et al 2015). Thermal modification was beneficial to increase the dimensional stability of wood, which was in accordance with the previous study (Esteves and Pereira 2008; Kocaefe et al 2008). Although further treatment with other chemicals, such as brown colorant, PWE and DCOIT, decreased the

dimensional stability of thermally-modified wood, the volume swelling rate of thermally-modified Scots pine treated with chemicals was still lower than that of untreated ones. This phenomenon indicates that thermal modification combined with further treatment with colorant, water repellent, and organic preservative microemulsion

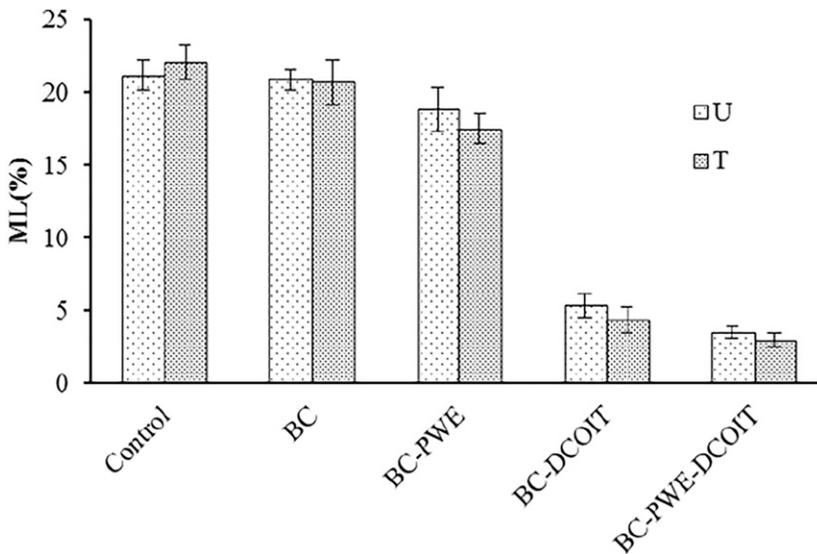


Figure 6. Mass loss (ML) of untreated (*U*) and thermally-modified (*T*) Scots pine treated with protection chemicals after fungal (*Gloeophyllum trabeum*) attacks for 4 mo. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

Table 4. Infected index of wood surface after exposure to mold fungi (*Aspergillus niger*) for 4 wk by visual inspection.

	Control	BC	BC-PWE	BC-DCOIT	BC-PWE-DCOIT
<i>U</i>	4	4	3	0	0
<i>T</i>	3	3	3	0	0

BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

had a positively comprehensive effect on improving the dimensional stability of wood.

Flexural Property

Both single thermal modification and combined thermal and chemical modification cause a slight decrease in MOR strength of wood (Fig 5). The decrease caused by thermal modification can be explained by the degradation of wood components, such as cellulose and hemicellulose, as shown in Table 2. In the further treated groups, MOR of thermally-modified wood still maintain a value of above 110 MPa, indicating that combined modification has insignificant effects on flexural MOR of thermally-modified wood. The slight decrease was probably due to destruction of wood structure in the process of high-pressure impregnation.

Decay and Mold Resistance

The effect of modifying chemicals on decay resistance of wood was conducted and the results were showed in Fig 6. Thermal modification at 140°C had little influence on decay resistance of wood, which was evidenced by similar mass loss in untreated and thermally-modified groups. This is reasonable because 140°C is a rather low temperature for thermal modification and only slight chemical components have been degraded as indicated in Table 3. The treating groups with DCOIT showed excellent decay resistance with mass loss of lower than 5%, whereas the value for control group reached up to 22% after exposed to fungal attacks for 4 mo, suggesting that addition of 0.1% DCOIT can prevent attacks from *Gloeophyllum trabeum* effectively, whereas the addition of BC or PWE has little effect on improving the decay resistance of wood.

Infection index of wood surface after exposure to mold fungi (*Aspergillus niger*) for 4 wk (Table 4) presents similar results with decay test. Only the groups with DCOIT showed a complete control of mold growth on wood surface, suggesting that DCOIT microemulsion is a good mold inhibitor.

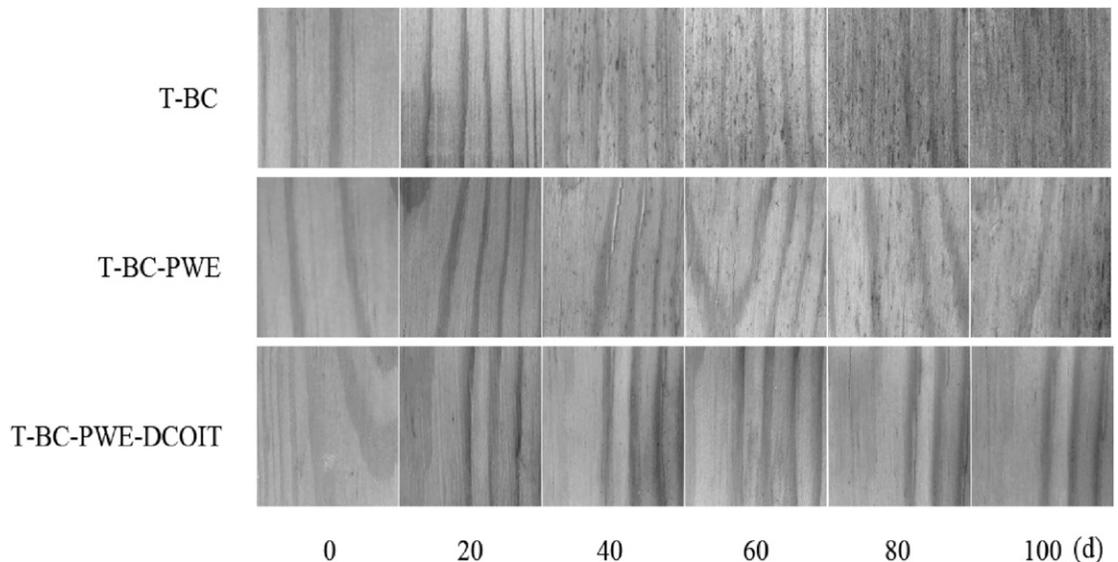


Figure 7. Surface images of thermally-modified Scots pine further treated with colorant (BC) or combination of colorant (BC), PWE, and DCOIT during outdoor weathering test. BC, brown colorant; DCOIT, 4,5-dichloro-2-octyl-2H-isothiazol-3-one; PWE, paraffin wax emulsion.

Thermal modification and addition of PWE can slightly decrease the infected rate of Scots pine surface against *Aspergillus niger* down to about 75%.

Figure 7 shows the surface image of thermally-modified samples further treated with BC, BC-PWE, and BC-PWE-DCOIT during outdoor weathering tests. Compared with artificial weathering process (Fig 1), more severe discoloration and cracks could be observed during outdoor weathering test. It was because the lab condition cannot simulate exactly the rain, wind, and also the biological threats, which would also affect the degradation behavior of wood. It is clear that the surfaces of brown colorant-treated thermally modified wood and brown colorant/PWE-treated thermally modified wood were infected with mold during outdoor weathering test, whereas T-BC-PWE-DCOIT group showed little sign of mold infection, which agreed well with lab data of mold resistance test in Table 4.

CONCLUSIONS

The treating groups with water repellent showed obvious lower water absorption, and the lowest water absorption appeared in the group treated with the combination of brown colorant and water repellent (BC-PWE). Thermal modification improved the dimensional stability of wood, whereas all the further treatments except the group impregnated with water repellent showed slight negative effect on dimensional stability after water soaking. The MOR of thermally-modified Scots pine showed little change after further treatments. Thermally-modified wood further treated with BC-PWE-DCOIT showed the best weathering performance by showing the least color change and the least occurrence of mold and fungi and addition of colorant in combination with wax and organic preservative can significantly improve the weathering performance of thermally treated wood when exposed to natural weathering.

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