

Evaluation of Color Changes, Wettability, and Moisture Sorption of Heat-Treated Blue-Stained Radiata Pine Lumber

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To improve the color and surface properties of blue-stained radiata pine, heat treatment was carried out at different temperatures and times. The color change, wettability, and dynamic moisture sorption of the wood were investigated. The results showed that, as the treatment temperature and duration time increased, the surface brightness of specimens gradually decreased, and the surface color became more reddish and blueish. In addition, the contact angle of distilled water at 0 s and 18 s gradually increased as the treatment temperature and time increased, which indicated a decrease in the surface wettability. The color of the blue-stained wood became more uniform with reduced surface wettability and hygroscopicity but increased sorption hysteresis after the treatment.

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INTRODUCTION

Radiata pine, a medium-density softwood, is susceptible to staining fungus infections during processing. Blue-stain fungi feed on simple sugars and starches in the wood. This can lead to bluish or greyish discolorations on the wood surface. The pigments of the blue-stain fungi are classified as melanin, and structurally heterogeneous polymers are formed *via* oxidative polymerization of phenolic compounds (Rättö *et al.* 2001). Although the blue-stain fungi do not influence the mechanical properties of the wood, the surface aesthetics are greatly affected, which severely reduces the added value of the wooden products (Humar *et al.* 2008).

Many studies have been reported to efficiently prevent the blue-staining of wood, which have involved chemical biocides, impregnation, and chemical modifications (Hsu *et al.* 2009; Kingsbury *et al.* 2012; Liu *et al.* 2012; Pries and Mai 2013; Bian *et al.* 2019). Nevertheless, it is necessary to alleviate the adverse effects of blue-staining on wood surfaces that have already been infected by the blue stain. Bleaching treatments for blue-stained lodgepole pine was carried out by Evans *et al.* (2007), who found that sodium hypochlorite was effective at removing the blue-color from blue-stained wood, but it was less effective at restoring the lightness of the treated wood. Hernandez *et al.* (2016) impregnated blue-stained pine wood using fungal pigments from spalting fungi and found that the fungal pigments were suitable for attenuating the appearance of the blue stain on the wood surface. Yu *et al.* (2010) reported that the combination of compression and heat

treatment can eliminate the adverse effects of blue-stain on the surface of Masson pine wood.

Heat treatment is commonly used to improve various wood properties, *e.g.*, dimensional stability and biological durability (Esteves and Pereira 2009). Heat treatment can also be used to improve the color uniformity of wood from its inner portion to its surface, hence diminishing the surface discoloration (Esteves *et al.* 2008; Cai and Cai 2012). Generally, heat treatment can make the color of the wood evenly darker in the thickness direction with reduced lightness and blue-yellow coordinate and increased green-red coordinate (Manoj *et al.* 2012; Cai *et al.* 2020). For example, Srinivas and Pandey (2012) reported that heat-treated rubber wood had a uniform color throughout. The lightness rapidly decreased as the time and temperature increased, while the red and yellow color values initially increased and later decreased with longer exposure at all temperatures. In addition to the surface color, the wettability of the wood is also altered by the heat treatment. Gao *et al.* (2013) studied the wettability of Southwestern birch through the dynamic contact angle and showed that the wettability of the wood surface decreased after heat treatment, with the contact angle increasing from 40° to 121°. Yang *et al.* (2010) demonstrated that the surface contact angle of heat-treated Douglas fir wood gradually increased as the treatment temperature and time increased, and the wettability of the wood surface decreased. Hakkou *et al.* (2005) investigated the wettability changes during heat treatment on the basis of chemical analysis. They found the wettability changes were attributable to the preferential degradation of amorphous cellulose and hemicelluloses, coupled with the plasticization of lignins and the desorption of residual water.

Therefore, heat treatment has a potential for eliminating the blemishes in blue-stained radiata pine wood. In addition, the properties of blue-stained wood could be further improved by heat treatment. Consequently, the application fields of blue-stained radiata pine wood will be expanded. In a previous study, it was confirmed that heat treatment could improve its quality by darkening and equalizing the color of the sap-stained wood (Lee *et al.* 2014). Hence, blue-stained radiata pine wood was subjected to heat treatment at various temperatures in the present study. The color difference, wettability, and water sorption values of blue-stained and normal radiata pine, before and after heat treatment, were analyzed.

EXPERIMENTAL

Materials

Quarter-sawn blue-stained and non-blue-stained radiata pine plank, with the dimensions of 2000 mm × 100 mm × 20 mm (L × R × T), was used as the materials in this study. The plank was subsequently sawn samples with a dimension size of 400 mm × 100 mm × 20 mm (L × R × T), and dried at a temperature of 65 °C (at a relative humidity (RH) of 50%) until the water content reached 12%.

Heat Treatment Process

The samples were heat treated at temperatures of 160, 170, 180, 190, 200, and 210 °C with superheated steam for 1, 2, and 3 h, respectively. Ten samples were used for each treatment group, including seven pieces of non-blue-stained wood and three pieces of blue-stained wood.

Surface Color Measurement

The color of the wood surface was measured with a colorimeter according to the CIE wood color characterization method promulgated by the International Commission on Illumination (CIE). A chroma meter (CR-400, Konica Minolta, Tokyo, Japan) was used to measure the L^* (lightness index), a^* (red-green index), and b^* (yellow-blue index), parameters at four specific positions on the tangential surface of each sample after regular intervals, and the average values were calculated. The ΔL^* , Δa^* , Δb^* parameters, and the ΔE were calculated according to Eq. 1 through Eq. 4,

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

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

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

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

where the L_i^* , a_i^* and b_i^* are the lightness, red-green index, and yellow-blue index of the untreated wood, respectively; L_t^* , a_t^* , and b_t^* are the lightness, red-green index, and yellow-blue index of the modified wood, respectively; and ΔL^* , Δa^* , Δb^* , and ΔE are the difference in the brightness, red-green index, yellow-blue index, and total color change, respectively (Dong *et al.* 2020).

Wettability Measurement

The contact angle was measured under constant temperature (25 °C) and humidity conditions (50%). The contact angle test reagents were distilled water (polar liquid) and diiodomethane (non-polar liquid). A micro-injector was used to take 2 μ L of distilled water and place it on the polished sample surface at different positions (the measuring method of diiodomethane was the same as that of distilled water). A wetting angle meter (DSA100, KRUSS Company, Germany) was used to measure the contact angle at 0 s and 18 s immediately after the drop fell onto the sample surface. Each sample was measured at 4 different positions.

After obtaining the contact angle value of θ , the Owens double-liquid method was used to calculate the surface free energy (Owens and Wendt 1969). According to the polar force (γ_s^p), the dispersion force (γ_s^d) (non-polar force), and the surface free energy (γ_s) (surface tension) of the distilled water and diiodomethane (Table 2), as well as the contact angle value of θ , the polar force, and the dispersion force (γ_L^d) can be obtained from Eq. 5 and 6, respectively,

$$\gamma_{L_1}(1 + \cos\theta_1) = 2(\gamma_s^d \gamma_{L_1}^d)^{1/2} + 2(\gamma_s^p \gamma_{L_1}^p)^{1/2} \quad (5)$$

$$\gamma_{L_2}(1 + \cos\theta_2) = 2(\gamma_s^d \gamma_{L_2}^d)^{1/2} + 2(\gamma_s^p \gamma_{L_2}^p)^{1/2} \quad (6)$$

where the surface free energy (γ_L) can be obtained according to Eq. 7,

$$\gamma_s^d + \gamma_s^p = \gamma_s \quad (7)$$

where γ_L , γ_L^d , and γ_L^p are the surface tension, dispersion force, and polar force of the test liquid (distilled water and diiodomethane), respectively; γ_s , γ_s^d , and γ_s^p are the surface free energy, dispersion force, and polar force of the test wood samples, respectively; and θ is the contact angle.

Sorption Measurement

Three pieces were randomly selected from the heat-treated (170 °C/3 h, 190 °C/3 h, and 210 °C/3 h) and untreated samples. A 30 mm × 100 mm × 20 mm piece was cut from 50 to 100 mm of the end of the plank, and then ground into a powder with a diameter of 0.425 mm. Approximately 20 to 40 mg of the powder was taken for sorption measurement using a dynamic vapor sorption instrument (Surface Measurement Systems, UK). The sorption test was carried out at a temperature of 25 °C, and the relative humidity (RH) range was 0% to 90% with an interval of 10%. The weight of the samples was automatically recorded every 60 s. The sample was considered to have reached the equilibrium moisture content (EMC) when the mass change rate between two consecutive measurements was less than 0.002 %/min. After the EMC was achieved, the instrument automatically adjusted the RH to the next stage for measurement.

RESULTS AND DISCUSSION

The Effect of the Heat Treatment Process on the Surface Color of Radiata Pine

As shown in Fig. 1, within the untreated groups, there was an obvious color difference between the blue-stained and the non-blue-stained samples. The surface color of the blue-stained radiata pine became more uniform after heat treatment. With the increase of the heat treatment temperature and time, the color of the wood surface showed a darkening trend, and the discoloration due to the blue stain gradually became invisible *via* macrography.



Fig. 1. Surface color of the different heat treated radiata pine samples

According to Fig. 2 the L^* (lightness index) values of both the blue-stained and non-blue-stained radiata pine samples showed a decreasing trend after heat treatment. The L^* decreased by 3.12% to 39.70% in the blue-stained samples, and a larger decrease of 6.26% to 48.15% was found for the normal non-blue-stained ones. After heat treatment, the L^* value of the blue-stained sample ranged from 64.30 and 40.02, while the L^* value ranged from 69.01 and 38.17 for the non-blue-stained samples. The brightness of both the blue-stained and non-blue-stained samples tended to be similar. For the non-blue-stained

samples, the a^* (red-green index) increased after heat treatment. The a^* value of the blue-stained sample ranged from 49.26 and 68.29, while the (a^*) value of the non-blue-stained sample ranged from 67.90 and 15.20. The blue change part (a^*) value was between 5.06 to 10.82 for the normal color part. When the (a^*) ranged from 8.11 to 11.82, the (a^*) of the two parts tended to be similar. The b^* value of the blue-stained sample ranged from 20.82 to 24.95, while it ranged between 23.20 and 26.64 for the non-blue-stained samples, the (b^*) of the two parts tended to be contrary. The increase of the blue change material ranged from 3.27% to 23.76%, and the decrease of the normal part ranged from 3.06% and 15.57%. The (b^*) values of the two parts tended to be distinctive. The above analysis shows that the blue change material had been heat-treated. The colors of the blue change part and the normal color part were close to each other. Figure 1 also shows the test results.

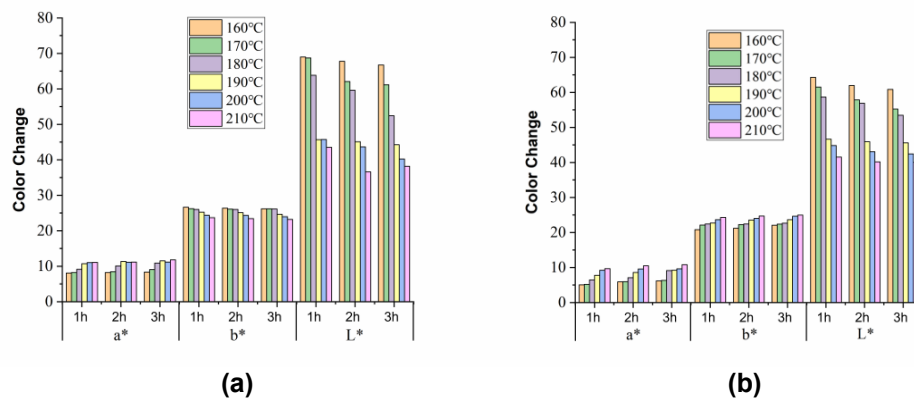


Fig. 2. Color change of the *Pinus radiata* wood samples after heat treatment: a) blue-stained samples; and b) nonblue-stained samples

The Effect of the Heat Treatment on the Surface Wettability and Surface Free Energy

Table 3 shows the change in the contact angle of the radiata pine surface. The initial contact angle of the untreated radiata pine was 62.39° , 39.46° and the angle decreased after 18 s. The contact angle difference between 0 and 18 s decreased for the heat-treated samples as the treatment temperature and time increased, which indicated that the surface wettability decreased and hydrophobicity increased after heat treatment. These results were consistent with previous research, which reported that the longer the droplet stays on the surface, the smaller the contact angle, and as the treatment temperature and time increases, the decreasing trend in contact angle became obvious (Yang *et al.* 2016).

The solid surface free energy is an important theoretical data value for explaining the wettability of a material surface. When the surface tension of a liquid was equal to or lower than the free energy of the wood surface, the liquid can be completely spread on the wood surface. The higher the free energy of the solid surface, the better the wetting performance. Figure 3 showed the results of the surface free energy of the both heat-treated and untreated the blue-stained radiata pine samples. The surface free energy (γ_s) of the untreated blue-stained sample was 53.5° . As the treatment temperature increased, the surface free energy of the sample showed a downward trend. The surface free energy ranged from 52.3° to 44.0° , and the surface free energy of the treated samples decreased to a range of 2.4° to 17.9° . Therefore, the higher the free energy of the solid surface, the higher

the wetting. Since the surface free energy of the heat-treated wood was reduced, its wettability was reduced.

Table 1. Contact Angle of the Wood Surface with Three Reagents

Groups	Initial Contact Angle Average Value			Contact Angle at 18 s Average Value			Differential (0s and 18s)		
	Distilled water	Methy- lene iodide	Formamide	Distilled water	Methy- lene iodide	Formamide	Distilled water	Methy- lene iodide	Formamide
Ref.	62.39	25.14	29.28	39.46	1.06	3.17	22.93	24.08	26.11
1	62.77	27.20	29.41	41.17	3.70	3.86	21.07	23.50	25.55
2	64.19	27.88	29.97	43.74	4.90	4.95	20.45	22.98	25.02
3	64.28	28.08	30.31	45.51	5.76	6.22	18.76	22.32	24.09
4	65.35	28.65	31.64	47.37	6.37	7.47	17.98	22.28	24.17
5	65.92	29.58	32.95	48.35	7.44	8.68	17.57	22.14	24.27
6	66.08	30.56	33.06	49.34	8.73	9.92	16.74	21.83	23.14
7	67.59	31.28	34.27	50.73	9.94	10.98	16.86	21.34	23.29
8	71.12	32.33	34.80	54.54	11.33	11.92	16.58	21.00	22.88
9	72.46	32.51	35.16	56.10	12.12	12.17	16.36	20.39	22.99
10	72.69	32.79	35.65	56.60	12.15	13.30	16.09	20.64	22.35
11	73.22	33.36	35.92	57.69	12.61	14.70	15.53	20.75	21.22
12	75.15	33.80	36.49	61.67	12.85	14.96	13.48	20.95	21.53
13	76.54	34.25	37.48	61.45	13.25	15.23	15.09	21.00	22.25
14	77.41	35.00	38.37	61.99	13.53	15.52	15.41	21.47	22.85
15	77.99	35.97	39.08	62.61	13.15	15.99	15.38	22.82	223.09
16	78.18	36.09	40.01	63.53	13.89	16.36	14.65	22.20	23.65
17	78.25	37.10	41.55	63.71	14.38	16.74	14.54	22.72	24.81
18	79.27	38.43	42.73	64.34	14.78	16.97	6.93	23.65	25.76

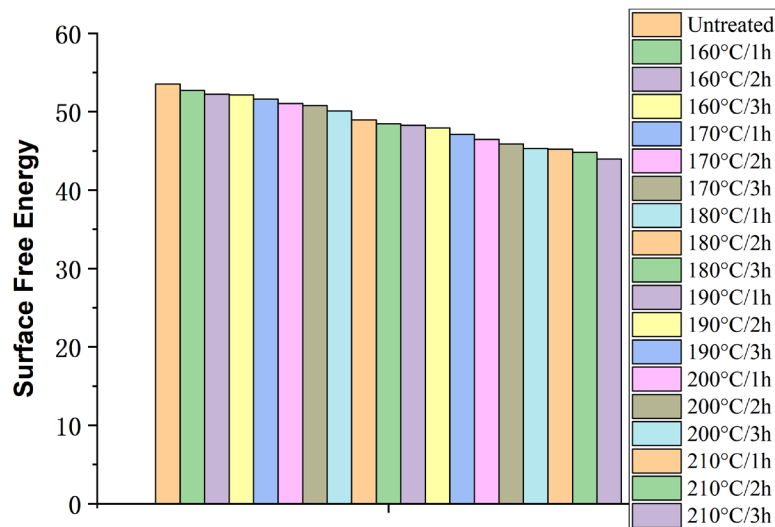


Fig. 3. Surface free energy of heat-treated and untreated radiata pine

The Influence of Heat Treatment on the Water Sorption Characteristics

Figure 4 shows the moisture adsorption and desorption isotherms of the heat-treated blue-stained and non-blue-stained radiata pine powder at an ambient temperature of 25 °C.

For the adsorption stage (from 0% RH to 90% RH), the moisture content of the treated samples showed an upward trend. The slope of the moisture adsorption isotherm was first steep, and then flat, followed by becoming steep again. During the desorption process, the slope of the curve first became steeper. Then it decreased approximately in a straight line, and finally became steeper again. Under the same RH conditions, the EMC for the adsorption and desorption gradually decreased as the treatment temperature increased, which indicated that heat treatment can reduce the equilibrium moisture content of wood. With the decrease of the relative humidity, the desorption equilibrium moisture content of the samples treated at the same temperature gradually decreased.

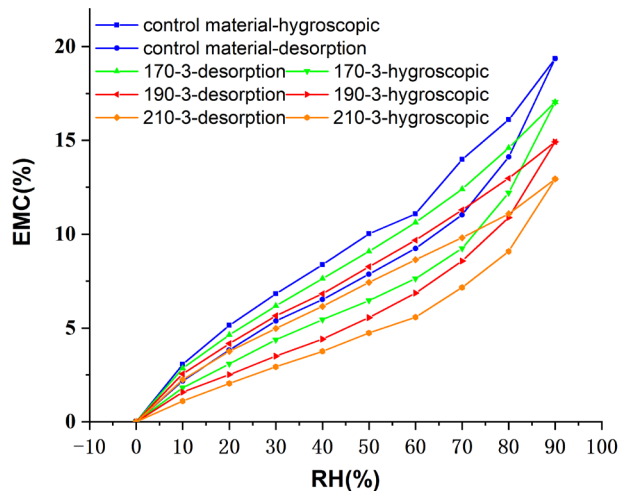


Fig. 4. The sorption isotherm of radiata pine at a temperature of 25 °C

Table 2. Hysteresis Coefficient of the Radiata Pine Samples at Different Relative Humidity

Relative Humidity (%)	Untreated	170 °C 3 h	190 °C 3 h	210 °C 3 h
10	0.74	0.65	0.60	0.51
20	0.76	0.69	0.62	0.55
30	0.79	0.70	0.62	0.59
40	0.78	0.72	0.65	0.60
50	0.79	0.72	0.67	0.63
60	0.78	0.72	0.70	0.66
70	0.80	0.75	0.76	0.73
80	0.88	0.84	0.83	0.80

Table 4 shows the hysteresis coefficient ($X_{HC} = W_{Ead}/W_{Edc}$, where X_{HC} means the moisture absorption hysteresis coefficient, and W_{Ead} and W_{Edc} represent moisture absorption EMC and desorption EMC of the samples at the same temperature and relative humidity condition respectively) of the radiata pine samples at different RH conditions. It was found that the hysteresis coefficient of the heat-treated samples was dramatically smaller than the hysteresis coefficient of the untreated ones at the same RH condition. In addition, it was found that the hysteresis coefficient was obviously reduced as the heat treatment temperature increased. Compared with the untreated samples, the reduction in

the hysteresis coefficient ranged from 8.4% to 30.8%. Under the same heat treatment temperature, the hysteresis coefficient of the samples gradually increased as the RH increased.

When the wood started to adsorb moisture, part of the free hydroxyl groups combined with each other *via* hydrogen bonding, so that the availability of the sorption site was reduced, and consequently the adsorbed moisture content was reduced. During desorption, the moisture inside the wood began to be removed, and the fiber surfaces were close to each other until only one monomolecular water layer remained between the surfaces of the two cellulose microfibrils. During this process, the free water molecular movement was hindered due to the internal resistance of the cellulose network structure, making it difficult for the absorbed water to escape. Part of the hydroxyl groups in the amorphous region reform the intra- or inter-molecular hydrogen bonds of the fiber, but the formation of hydrogen bonds is irreversible. Since fewer hydrogen bonds were reformed, there were more sorption centers, which led to a greater amount of absorbed water, resulting in hysteresis (Qi *et al.* 2010). Heat treatment reduced the EMC and the hygroscopicity of the wood.

CONCLUSIONS

1. After heat treatment with superheated steam, the lightness index and yellow-blue index of the surface of radiata pine gradually decreased, while the red-green index gradually increased. The color difference between the blue-stained and non-blue-stained surface decreased as the temperature increased. When the samples were treated using the 190 °C/3 h method, the colors of the blue-stained and non-blue-stained samples became similar, and the discoloration was eliminated.
2. With the increase of heat treatment temperature and time, the initial contact angles of distilled water, formamide, and diiodomethane and the contact angles at 18s increased gradually, but their difference decreased with the increasing temperature. So the heat treatment reduced the surface free energy of the wood, and thus improved its hydrophobicity.
3. In addition, heat treatment can considerably reduce the EMC of radiata pine wood. As the treatment temperature increased, the EMC of adsorption decreased, and the hysteresis coefficient of the moisture sorption gradually decreased.

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