Comparison among Parameters Affecting Vertical Vacuum Dyeing of *Populus cathayana* Rehd

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Abstract

Dyeing pale-colored plantation-grown hardwood is an effective way to meet the demand for richly colored tropical hardwoods used in decorative applications. The objective of this study was to investigate the influence of wood dyeing parameters such as vacuum pressure, vacuum time, dyeing time, and dye concentration regarding the dyeing of *Populus cathayana* Rehd using the vacuum dyeing system. As the height increased, the color change decreased gradually. Color change and dye uptake gradually increased with increasing vacuum pressure and dye concentration. As the vacuum time and dyeing time increased, color change and dye uptake first gradually increased until water began to be discharged, leading to a decrease of the pressure gradient in the log, and then decreased.

A lack of natural sources for high-quality, large-diameter timber has created a scenario in which the construction and decoration industries of China must rely on timber plantations as their primary source of material (Duan 2002). The majority of this fast-growing plantation timber is unusable in furniture or interior decoration industries because of its indistinct grain and uneven color, which is highly susceptible to change during storage, processing, and use. An effective method of amplifying both the visual characteristics and the value of plantation timber is through the use of wood dyeing technology, which can simulate the rich, attractive coloration of tropical hardwoods. Wood dyeing technology can aid in balancing the supply and demand of precious, large-diameter timber (Liu and Zhang 1996). There are many applications of dyed wood, such as decorative veneer, craftwork products, musical instruments, furniture, and decorative wood products (Duan 2004).

Recent studies on wood dyeing have mainly focused on the dyeing process of veneer and paneling rather than the log itself. Dyeing veneers shows distinct disadvantages, however, because they are easily damaged during the dyeing process, easily warped after dyeing, and involve a more complicated production process (Chang et al. 2009). The best way to use the wood is to dye the log directly, so that furniture or peeling veneer can be made using conventional processes.

Previous studies have reported a variety of wood dyeing methods, such as vacuum dyeing; pressure dyeing, which combines vacuum dyeing and pressure dyeing; and others. Applying a vacuum is one way to potentially achieve uniform color inside of the wood. Vacuuming is also useful for improving uptake because it discharges the free water and air from within the wood (Yu 1999, Li et al. 2011). If wood is immersed in a dye solution for extended periods of time during the dyeing process, pressure gradients cannot be created within the wood. Successful saturation with dye depends on transverse passageways in the wood, where only a few of the passageways are receptive to dye movement. To this effect, the dye solution does not easily permeate thicker timber; the wood, as a result, possesses uneven color and shallow dye penetration (Li 2005).

To form a theoretical foundation for optimizing the dyeing process to produce the highest quality product, tests were conducted using the vertical vacuum dyeing method on logs of *Populus cathayana* Rehd. There are two primary advantages to this method. First, we used axial direction passageways to dye the timber, because the permeability of the axial direction passageways of the wood are greater than

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the transverse passageways. Second, the log is not soaked in the dye solution, allowing for a pressure gradient to form in the wood, which results in an even penetration of dye solution. The objective of this study was to investigate the relationships among various factors as they affect the vacuum dyeing process and the effects of the dying processes on wood. We also investigate changes in color on the same log with varying height, using in the same dyeing process.

Materials and Methods

Materials and equipment

The fast-growing poplar wood of *Populus cathayana* was collected from Tong Zhou district, Beijing. The dyestuff used was reactive red X-3B, which was purchased from Shanghai Taopu dye factory; it was in powder form before being used. The main test equipment was an electronic balance (FA2204) temperature and humidity chamber (TERCHY MHGB-120CAUB), a wood vacuum dyeing system that was made uniquely for the present work. A schematic diagram of the vertical vacuum dyeing apparatus is shown in Figure 1. The system consisted of a dye vat, a vacuum tank, a residual liquid collection room vessel, and a vacuum pump. The analytical equipment used was an ultraviolet-visible (UV-vis) spectrophotometer (Unic UV-2000), and a color meter (Dataflash DF110).

The preparation of dyed wood

In this test, 38 310 by 105-mm (length by diameter) log sections were used. The weights of the log sections, as well as their heartwood and sapwood content, were determined to be comparable prior to experimentation in order to maintain uniformity among samples to which the same dyeing process was applied. Dye concentrations of 0.5, 1, and 1.5 percent were made by weighing the dyestuff m_0 (g) in a beaker and diluting the dye with distilled water. The temperature of the dye solution was 30°C. Experiments were conducted in accordance with Table 1, and two test samples were used in each experiment.

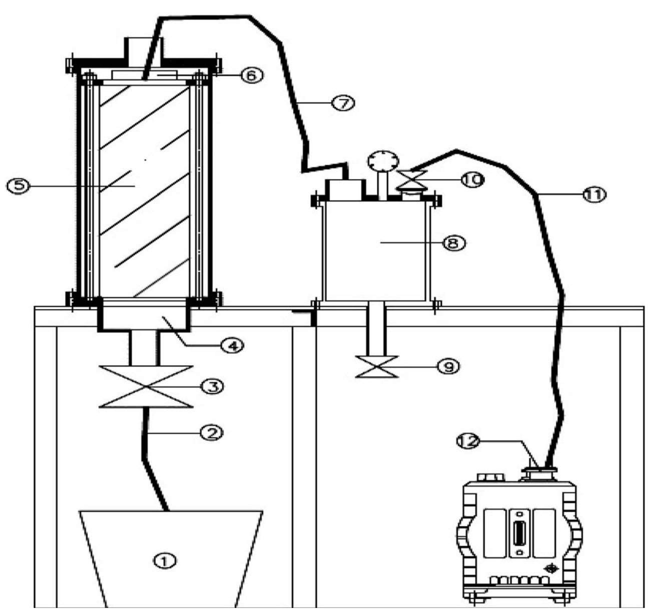

Figure 1.—Schematic diagram of vertical vacuum dyeing apparatus: 1 = dye vat; 2, 7, and 11 = rubber tube; 3, 9, and 10 = ball valve; 4 = intake chamber; 5 = log; 6 = exhaust chamber; 8 = residual liquid collection vessel; 12 = vacuum pump.

Placing the log sections into the wood vacuum dyeing system, we filled the dye vat (Fig. 1, no. 12) with dye solution and immersed the rubber tube (Fig. 1, no. 2) into the bottom of the dye vat. We closed ball valves 3 and 9 (Fig. 1, no. 3 and 9) to ensure the vacuum system is properly sealed and then turned on the vacuum pump (Fig. 1, no. 12) to make logs under vacuum treatment, setting vacuum pressure and vacuum time. After vacuum treatment, we turned on ball valve 3 to place dye solution from the bottom of the logs to the top of the logs. The dye solution, which accumulated on the top surface of logs, could be discharged to residual liquid until the height of the dye solution covered rubber tube 7. When the dyeing time was sufficient, we turned off the vacuum pump and turned on ball valve 3.

The vacuum pressure, vacuum time, dyeing time, and dye concentration all were in accordance with Table 1. The end of the sample that was in contact with the intake chamber was regarded as the bottom of the log, while the end of the sample that was in contact with the exhaust chamber was regarded as the top of the log. Disks were cut from the dyed logs at the heights of 8.2, 69.6, 131, 225.4, and 286.8 mm and were dried in open air for 1 month. The size of the disks was 105 by 15 mm (diameter by height). The disks were then put into a temperature and humidity chamber to dry until the moisture content of their respective surfaces was 6 to 8 percent.

Dyeing effect evaluation

The measurement of dye uptake.—Dye use is the percentage of dyestuff permeating the wood as a function of the percentage of total dyes in the dye solution before being applied. Since the dye solution partly displaces the sap in the log, the amount of dye absorbing directly into the log cannot be measured by weight change. Dye uptake was therefore calculated according to the Lambert-Beer law to determine the relationship between dye concentration and absorbance. The absorbance could be measured by spectrophotometry. With the relationship between the dye concentration and absorbance determined, the dye concentration was obtained by measuring the absorbance. The UV-vis absorption spectrometer was used to measure the spectral absorption curve of the reactive red X-3B from 200 to 700 nm. The maximum absorption wavelength was 538 nm within a given period. Dye solutions were prepared with a concentration between 0.003 and 0.007 percent. The absorbance of these dye solutions was measured at 538 nm. Then a linear regression equation was set up to draw the standard curve of dye concentration and absorbance.

Distilled water was used to clean the vacuum tank, the residual liquid collection vessel, and the dyed wood. The cleaning fluid was then mixed with the collected residual dye solution after dyeing. A pipette was used to move 1 mL of dye solution into a beaker, and then the dye solution was diluted with 100 times distilled water. The absorbance was measured three times, and its average value was calculated. The value was put into the standard curve, and the weight of the dyestuff in the collected dye solution was calculated. The value of the dye uptake was the result, as follows,

$$\text{dye uptake } (\%) = (m_0 - m_1)/m_0 \times 100\% \qquad (1)$$

Table 1.—Design of experiment, indicating levels of factors.

Sample combination	Test factors			
	Vacuum pressure (MPa)	Vacuum time (min)	Dyeing time (min)	Dye concentration (%)
A	0.01, 0.03, 0.05, 0.07, 0.09	5	60	1
B	0.09	0, 5, 15, 30, 60	60	1
C	0.09	15	5, 15, 30, 45, 60, 75	1
D	0.09	15	60	0.5, 1, 1.5

where m_0 is the initial concentration and m_1 is the concentration observed for the test condition.

The measurement of color change.—The color change of the *Populus* was the main observation indicator, and dye uptake was the reference indicator. ''Color change value'' means the difference of the color between undyed control samples and experiment samples. The Datacolor 110 automatic color change meter was adopted to measure the color change between contrast samples and experiment samples whose moisture content was 6 to 8 percent after dyeing. The standard illuminant used was D65, correlated color temperature was 6504 K, the lighting and observation geometry condition was o/d (vertical incidence/diffuse), view was 10°, and the measurement range was 20 mm in diameter. The transection of each sample was measured four times, and then its average value was calculated. The chromaticity index deviation in terms of the color parameters L^*, a^*, and b^* was calculated according to the specimen data previously acquired. To calculate the color change, the following equation was used (Yu 1999):

$$\Delta E = \text{sqrt}\left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right] \quad (2)$$

where L^* is the luminancy, a^* is the red/green chrominancies, and b^* is the yellow/blue chrominancies.

Results and Discussion

Effects of vacuum pressure on dye uptake and color change

The following parameters remained constant: 5 minutes of vacuum time, 60 minutes of dyeing time, and 1 percent

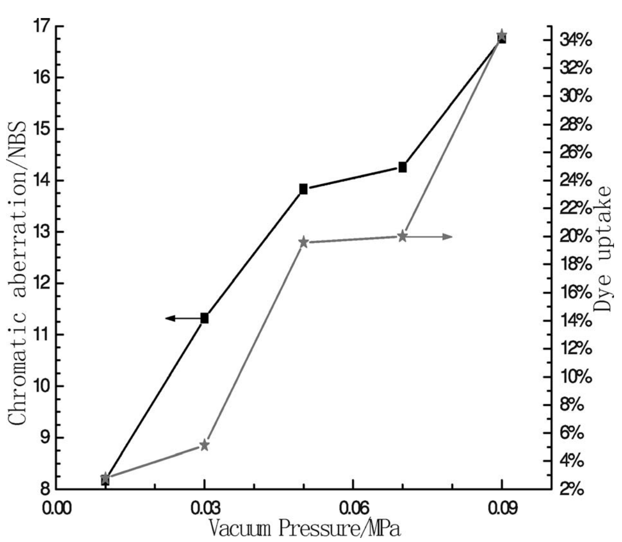

Figure 2.—Effect of vacuum pressure on color change and dye uptake.

dye concentration. The effects of different vacuum pressures to the dye uptake and color change were studied (shown in Fig. 2). Dye uptake and color change increased as the pressure in the vacuum decreased. When the vacuum pressure reached 0.09 MPa, the dye uptake and color change both reached their greatest value. The values of color change and dye uptake were 16.8 and 34.3 percent, respectively. When the vacuum pressure increased from 0.01 to 0.09 MPa, the color change and dye uptake increased by 8.6 and 31.6 percent, respectively.

Free water and air within the log is discharged through the application of negative pressure. Using the pressure gradient and concentration gradient of the log allows the dye solution to permeate and diffuse into the log uniformly. By releasing free water and air in the log, the air resistance of the log decreases so that the passageways of the log are less obstructed and the dye solution permeates into the wood more easily (Yu et al. 2002). Many capillary interstices are also present in the log, allowing the dye solution to permeate easily into the log by capillarity and diffusion.

Freshly felled *Populus* with a density of 0.386 g/cm^3 was used in this study. There were significant amounts of free water and air inside of the log prior to experimentation. The greater the vacuum pressure, the faster free water and air were released, allowing for a greater amount of the dye to permeate into the log (Yu et al. 2002).

There is a pressure gradient between the bottom and the top of the log during the vacuum state. As the vacuum pressure decreased, so did the pressure gradient of the log and the permeation rate. This resulted in an increase of dye permeating into the log, so that the dye uptake demonstrably increased and the color grew darker than a nondyed log.

As shown in Figure 3, the vacuum pressure and color change value simultaneously decreased with increase of height and reached its greatest color change value at a height of 8.2 mm from the bottom of the log. Because the bottom of the sample came in contact with the dye solution for extended periods of time during the dyeing process, the bottom of the log absorbed greater amounts of dye.

Effect of vacuum time on dye uptake and color change

The following parameters remained constant: a vacuum pressure of 0.09 MPa, 60 minutes of dyeing time, and a 1 percent dye concentration. The effect of varying vacuum times on dye uptake and color change was explored, as shown in Figure 4. The vacuum time that achieved the greatest color change and dye uptake in the sample was about 15 minutes. The values for the color change and dye uptake were 18.455 and 33.79 percent, respectively. When time increased from 0 to 15 minutes, the color change and dye uptake increased by 7.786 and 10.52 percent, respectively. With an increase of vacuum time from 15 to 60

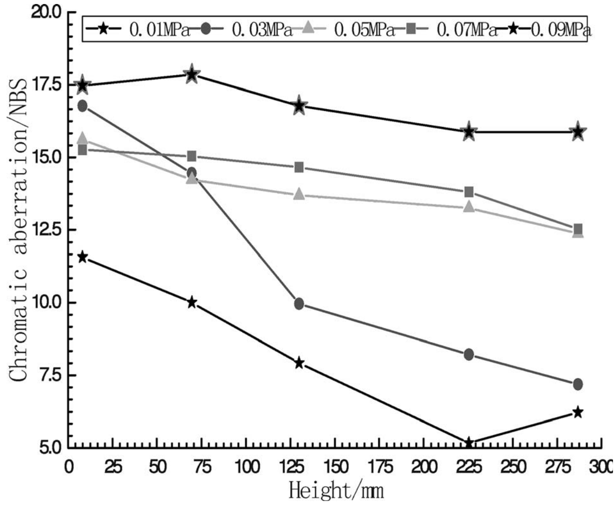

Figure 3.—Effect of vacuum pressure on color change at different heights.

minutes, the color change decreased by 5.863 percent, and the dye uptake decreased by 11.37 percent.

To summarize, vacuum times up to 15 minutes showed more free water and air discharged from the log with increased time, allowing the dye solution to permeate into the log more easily. Color change and dye uptake increased with longer vacuum time.

When vacuum time was above 15 minutes, we observed discharged water on the top of the log that increased with vacuum time. Because the *Populus* used in this study was fresh, the content of free water in these samples was very high, and an increase in vacuum time allowed for a significant discharge of this abundant free water from the log. Because the discharged water could not reach rubber tube 7 (Fig. 1, no. 7) it did not fully discharge from the exhaust chamber (Fig. 1, no. 6) into the residual liquid collection vessel (Fig. 1, no. 8) and instead accumulated on the top surface of the log, which prevented the free water and air in the log from flowing out. This led to a decrease of the pressure gradient in the log and reduced the amount dye that permeated into it, resulting in reduced color change and dye uptake with longer vacuum time. The color change of the dyed sample reached its greatest value at a height of 8.2 mm from the bottom of the log, as shown in Figure 5. Because the bottom of the sample was in contact with the dye for an extended period of time during the dyeing process, these portions of the sample absorbed greater amounts of dye.

Increased vacuum time allowed for more free air and water to be discharged from the log, thus increasing void space in the log. However, if vacuum time was too long, the free water was not able to fully discharge into the residual liquid collection vessel, because the discharged water could not reach the exhaust chamber. This led to a decrease of the pressure gradient in the log and reduced the amount dye that permeated into the log. Color change decreased as a result.

Effect of dyeing time on dye uptake and color change

The following parameters remained constant: a vacuum pressure of 0.09 MPa, 15 minutes of vacuum time, and a 1 percent dye concentration. The effects of different dyeing time on dye uptake and color change were studied, as shown in Figure 6. The dyeing time that produced the greatest color change and dye uptake in the sample was about 60 minutes. The values of color change and dye uptake were 20.782 and 32.11 percent, respectively. When dyeing time increased from 5 to 60 minutes, the color change increased by 8.044 percent and the dye uptake increased to 20.47 percent. The color change and dye uptake tended to reach a balanced state when the dyeing time was increased above 60 minutes.

Owing to the larger size of the sample, dye permeated and diffused into the log over a more gradual process. If the dyeing time was too short, dye did not easily permeate into the log. With an increase of dyeing time, the amount of dye that entered into the log increased to where the wood fiber could absorb more dye altogether and allow the dye to migrate throughout the log more thoroughly (Liao and Deng 2006). When the dyeing time was above 60 minutes, the color change and dye uptake tended to reach a balanced level. Significant empty capillary interstices were produced

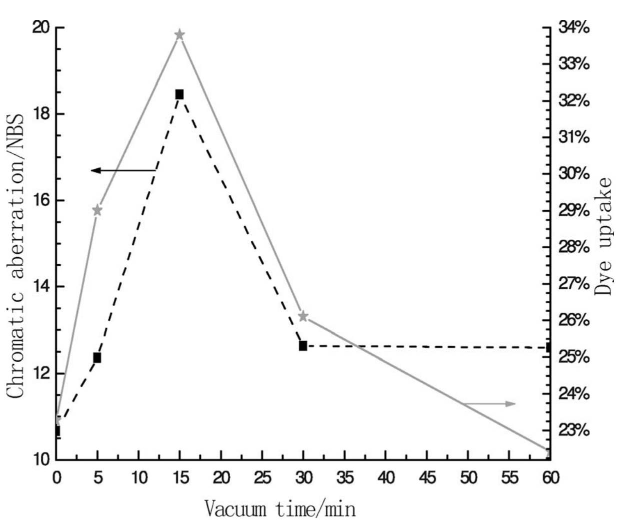

Figure 4.—Effect of vacuum time on color change and dye uptake.

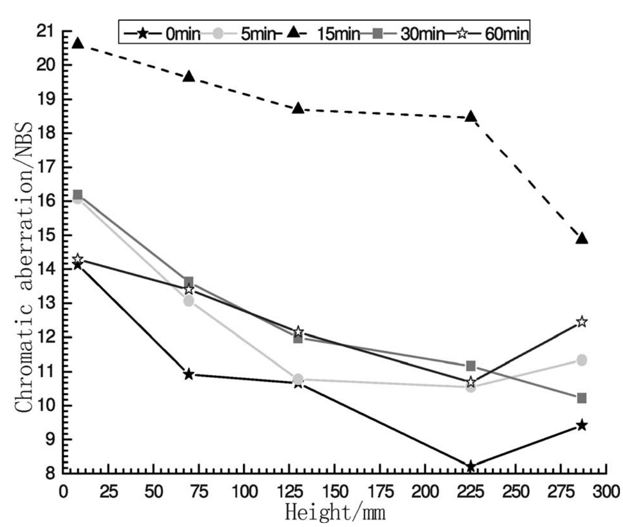

Figure 5.—Effect of vacuum time on color change at different heights.

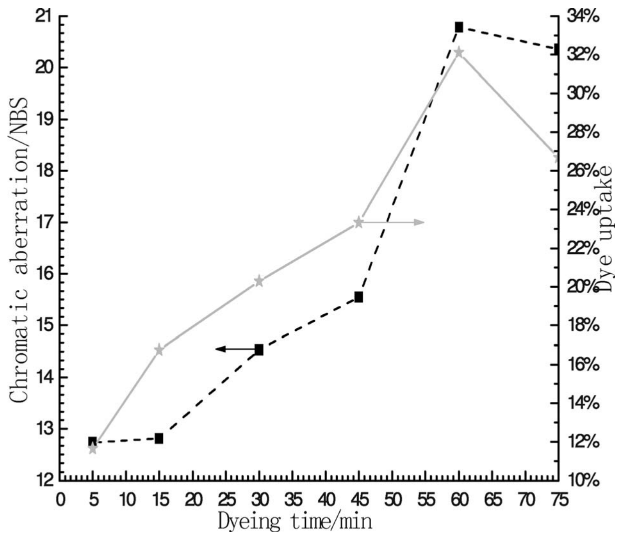

Figure 6.—Effect of dyeing time on color change and dye uptake.

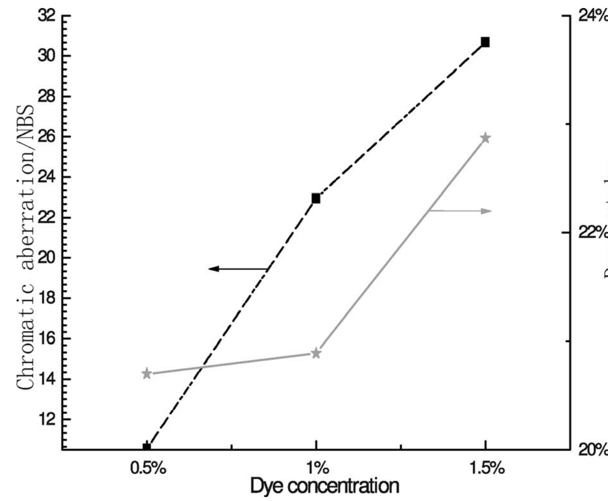

Figure 8.—Effect of dye concentration on color change and dye uptake.

in the log after its exposure to the vacuum. At the beginning of the dyeing process, the concentration and pressure gradient of the sample were high, as was the rate of diffusion—this allowed dye to permeate and diffuse into the log quite rapidly. With a prolonged dyeing time, the concentration gradient of the log gradually decreased, resulting in a gradual decrease of diffusion, as well. At the same time, the dye that entered the log first blocked the passageways of the log, leading to the decrease of effective flow area, which reduced the amount of dyestuff immersed in the log. When the dyeing time was above 60 minutes, the color change and dye uptake tended to balance out. Sixty minutes was thus determined the most appropriate dyeing time.

The color change either decreased, or decreased and then increased, under the same dyeing time, shown in Figure 7. The color change of the dyed sample reached its greatest value at the height 8.2 mm from the bottom of the log. The color change increased and then balanced higher up the log under the same dyeing time. At a dyeing time of 60 minutes, the color change of the dyed sample reached its value. Because the discharged water could not reach the height of the exhaust chamber, the free water could not discharge into the residual liquid collection vessel, which caused dye solution to accumulate in the exhaust chamber. The dye then began to permeate and diffuse backward. This backward diffusion resulted in an increase of dye immersion at the top and bottom of the log, where the dye was closest to the sample.

Effect of dye concentration on dye uptake and color change

The following conditions remained constant: a vacuum pressure of 0.09 MPa, a 15-minute vacuum time, and a 60-minute dyeing time. The effects of different dye concentrations on the dye uptake and color change were explored, as shown in Figure 8. The dye uptake and color change both increased sharply with an increase of dye concentration. The dye concentration that achieved the most favorable change

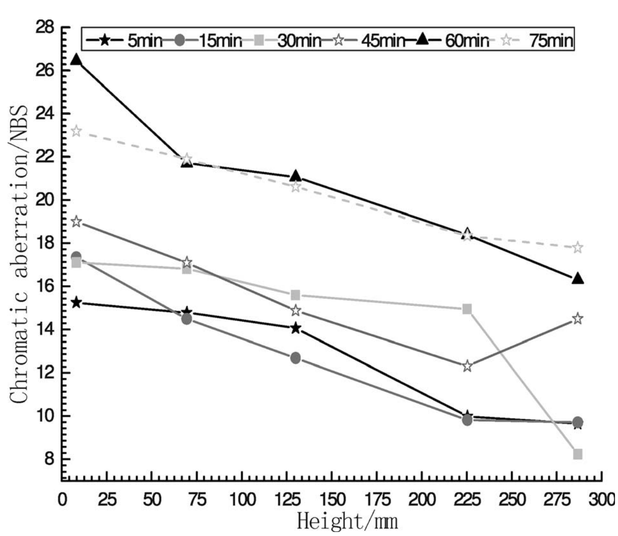

Figure 7.—Effect of dyeing time on color change at different heights.

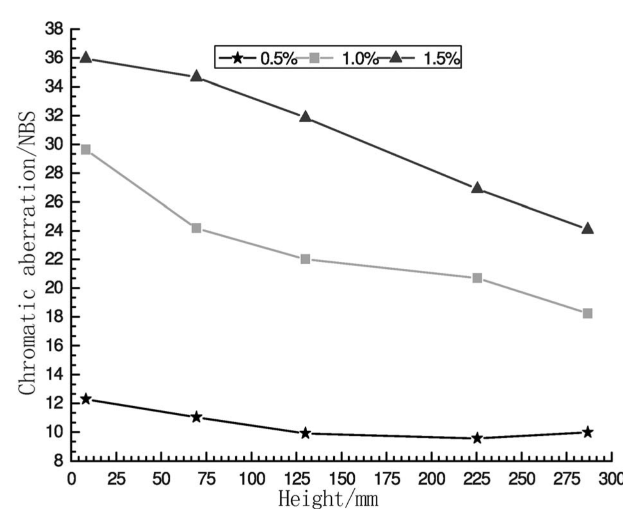

Figure 9.—Effect of dye concentration on color change at different heights.

and dye uptake in the sample was about 1.5 percent. The values of the color change and dye uptake were 30.684 and 22.87 percent, respectively. The amount of dye in the dye solution increased alongside increasing dye concentration—this process creates more opportunity for the dye to contact the wood fiber surface, thus quickly increasing color change and dye uptake.

As Figure 9 shows, color change decreased with an increase in the height of the sample within the same dye concentration. The color change of the dyed sample reached its greatest value at the height 8.2 mm from the bottom of the log. The color change increased at the same height of the dyed sample with increased dye concentration.

Conclusions

1. Using the vertical vacuum dyeing system to dye *Populus* logs produces quite favorable results.
2. Color change and dye uptake increased gradually corresponding to decreased vacuum pressure and dye concentration.
3. The effects of vacuum time and dyeing time on color change and dye uptake of the log was affected significantly by the amount of free water discharged from the log. The vertical vacuum dyeing process allows for free water to discharge to the top of the log, reducing the amount of dye that permeated into the log. As vacuum time and dyeing time increased, color change and dye uptake first gradually increased, then decreased once this discharged water appeared at the top of log.
4. From the bottom to the top of the log, the color change either decreased gradually or decreased and then increased.

Acknowledgment

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Literature Cited

Chang, D. L., N. Liu, W. H. Hu, W. H. Huang, Y. L. Zhang, Q. Xie, and F. H. Li. 2009. Deep dyeing technology for woods from three fast-growing tree species. *J. Northeast Forestry Univ*. 37(12):22–24.

Duan, X. F. 2002. Color of Wood and Its Controlling Technology. Building Materials Press, Beijing. pp. 89–92.

Duan, X. F. 2004. Wood microstructure effects on Chinese white poplar dyeing. *Res. Inst. Wood Ind. Chin. Acad. Forestry* 3(4):59–64.

Li, H. 2005. Study on dyeing technology of *Populus tomentosa*. Beijing Forestry University, Beijing. pp. 29–31.

Li, Y. F., Y. X. Liu, F. H. Wang, and G. J. Jiang. 2011. Controlling factors of wood permeability and its improving measures. *Sci. Silvae Sinicae* 47(5):131–139.

Liao, Q. and H. Deng. 2006. Dyeing of the padauk-imitated veneer of paulownia wood with reactive dyes. *J. Central South Forestry Univ*. 26(1):82–85.

Liu, Y. L. and X. P. Zhang. 1996. Introduction to wood dyeing. *Forest Sci. Technol*. 21(6):48–50.

Yu, Z. M. 1999. The Research of Wood Dyeing Technology and Permeable Mechanism of Dyestuffs. Beijing Forestry University, Beijing. pp. 36–38.

Yu, Z. M., L. Zhao, and W. J. Li. 2002. Study on permeable mechanism with dyestuff during wood dyeing. *J. Beijing Forestry Univ*. 24(1):79–82.