Investigation into the Optimal Dyeing Method for Bluegum (*Eucalyptus globulus*) Veneer

Ngoc Nguyen,[a,*] Barbara Ozarska,[a] Mac Fergusson,[b] and Peter Vinden [a]

This study investigated the dyeing methods (soaking and vacuum-pressure), types of dye (direct dye and reactive dye), and dyeing parameters (dye concentration, dyeing time, and temperature) in the veneer dyeing process for *Eucalyptus globulus* grown on plantations in Australia. The dyed veneers were assessed in two ways: dye penetration, which was determined using ImageJ software, and visual veneer grading for identifying any damage (curves or cracks). Veneers with different moisture content (MC) levels were used and were called green veneer (80% ± 5% MC) and dried veneer (12% MC). The study showed that the reactive dye Procion Brown P2RN at a concentration of 2% resulted in a significantly higher dye penetration than the other dyes. Soaking was not recommended as the dyeing method for this species because the dyed samples were severely damaged by the pre-treatments and high temperatures. A dye penetration of 100% was achieved when using the vacuum-pressure method with a dyeing time of 120 min, a pressure of 1000 kPa, and the addition of 20 g/L of sodium chloride. The results of this study can be applied in further research on the veneer dyeing process for this species.

Keywords: Reactive dye; Direct dye; Eucalyptus globulus; Veneer dyeing

Contact information: a: University of Melbourne, School of Ecosystem and Forest Sciences, 500 Yarra Boulevard, Richmond, Victoria 3121, Australia; b: RMIT University, Centre for Materials Innovation and Future Fashion, 25 Dawson Street, Brunswick, Victoria 3056, Australia;
** Corresponding author: bnguyen1@student.unimelb.edu.au*

INTRODUCTION

Colour is a vital parameter in terms of the aesthetic and decorative value of wood veneers for most wood products used in interior applications, such as furniture, joinery, panelling, and architectural products. Many hardwood species have a nonhomogeneous colour and do not meet the requirements of most consumers. This colour disharmony can be homogenised with various techniques, such as staining and dyeing.

The art of dyeing dates back thousands of years to the use of natural dyes extracted from plants and animals. The modern dye industry started 150 years ago with the discovery of synthetic dyes. Thousands of dyes have since been developed to work with various types of materials (Katz 2003). To understand wood dyeing, many important aspects of this process need to be determined, such as the types of dye suitable for cellulose fibres, chemical interactions between wood and dyes, and dyeing parameters. Cellulose fibres can be dyed using several different dye types, such as reactive dyes, direct (substantive) dyes, vat dyes, sulphur dyes, and naphthol azoic dyes (Lacasse and Baumann 2004).

Large plantations of *Eucalyptus globulus* wood established in Australia have mainly been grown to produce pulpwood. This resource is not suitable for the production of decorative products, principally because of the low wood grades and unattractive colour

(McGavin *et al.* 2015). The manufacture of veneer-based products has recently been identified as having an unprecedented opportunity to promote higher value utilization of plantation resources. However, many uncertainties remain regarding the impacts of the inferior wood quality of young plantation trees on the product recovery and value, as well as the optimal processing techniques. Moreover, the quality of veneers and veneer-based products is far from optimal because these trees are young and have small diameters. The veneers also have substantial colour variation that affects the added value of the final products. Developing production methods that can enhance the appearance of low-quality veneers produced from young, small-diameter logs has great potential.

An innovative method for enhancing the appearance of low-quality veneers has been developed by ALPI, a company in Italy that is involved in the production of multilaminar veneers, which are also called “reconstructed veneers”. One of the most important stages in multilaminar production is dyeing the veneer throughout its entire thickness, which can be achieved by dyeing the veneer with dyes of different colours, depending on the appearance of the products, their design, and market demand.

In the current multilaminar veneer manufacturing process, veneer dyeing is normally applied to give veneers a colour similar to that of another timber or to minimise variations and make the veneers more homogeneous in colour (Castro and Zanuttini 2004). Basically, veneer dyeing methods are based on the deep colouration of wood, which is known as wood impregnation or dyeing, as opposed to surface colouration (varnishing and painting). The method used depends on the applications of the veneers (Kwiatkowski 2007). The benefit of dyeing is that the colour cannot be removed by sanding because the veneer is dyed throughout its entire thickness (Wagenführ *et al.* 2012).

Although veneer dyeing technology has been well advanced in Italy, it has focused on poplar veneers, using plantation wood that is characterised by a low density, even colour, small number of defects, and high permeability. Conversely, the majority of plantation eucalypts have a medium to high density, many defects, uneven colour, and low permeability. Therefore, a detailed study is required to investigate the veneer dyeing process for eucalypt veneers.

The aim of this study was to investigate the dyeing methods, types of dye, and dyeing parameters that would be the most suitable for dyeing *Eucalyptus globulus* veneers. The results of this study can be applied in further research on the veneer dyeing process of this species.

EXPERIMENTAL

Materials

Samples

The *Eucalyptus globulus* wood was obtained from a commercial plantation in Ballan, Victoria, Australia. The plantation was established in June 2000 by Australian Bluegum Plantations Company, and the trees were harvested in December 2015. The trees used in this study were felled at an average stump height of 0.5 m. From each tree, three 1.8-m long billets were cut from the bottom of the logs. The billets were then rotary peeled using a spindleless veneer four-foot-lathe. The lathe was operated and set with parameters determined for peeling *Eucalyptus globulus* to provide the optimal veneer quality in relation to veneer thickness variation along and cross the grain, surface roughness, and flatness (McGavin *et al.* 2014).

Veneers made from sapwood and heartwood with two different moisture content (MC) levels were used to conduct the dyeing experiments and were labelled green veneer (80% ± 5% MC) and dried veneer (12% MC). The sample dimensions were 100 mm x 50 mm x 2.6 mm, which was based on the dimensions of the equipment used in the experiments. The veneer thickness was selected because this represents the most common veneer thickness used for wood veneer-based products in Australia. There were 12 samples for each combination of the experiments.

The quality of the wood veneer was assessed by visual grading based on Australian and New Zealand standard *AS/NZS 2269.0:2012* (Standard Australia 2012). D-grade was the visual grade quality for the selected veneers.

Dyes

In this study, two different types of dye were used: direct (substantive) dye and reactive dye. The dark brown colour of the dyes allowed for easy observation of dye penetration into the wood veneers. Direct and reactive dyes were selected because they are the most common types used for dyeing cellulose fibres, as they are known to be compatible (Shore 1995; Lacasse and Baumann 2004). Materials dyed with these dye types have also been reported to have a very good wash fastness (Shore 1995).

Two different dyes in the category of the reactive dye were used: Procion H-EXL and Procion Brown P2RN. One direct dye was used, which was the mixture of Sirius Red F-4BL, Sirius Yellow K-GRL, and Sirius Grey K-CGL. All of the dyes were purchased from Dyechem Australia Pty Limited (Melbourne, Australia).

Soaking and vacuum-pressure methods were used in this study to compare the results and select the suitable method for veneer dyeing. These processes were conducted at RMIT University, Centre for Materials Innovation and Future Fashion and the University of Melbourne, Creswick Campus, Victoria, Australia.

Equipment

The equipment used for the soaking method were as follows:

- Atlas Launder-Ometer (Rock Hill, SC, USA):
  This piece of equipment is also called a “shaker”. The shaker comes with an easy opening door and quick-lock retention bars for easy loading and unloading of the containers (20 container capacity).
- Magnet stirring device (also known as a magnetic stir plate or magnetic stirrer) (Industrial Equipment & Control Pty., Ltd, Thornbury, Victoria, Australia):
  This piece of equipment is very common in experimental chemistry. Using a magnetic stirrer rather than manual stirring is critical for consistent, reproducible mixing or mixing over long periods of time. Solutions are mixed with the magnetic stirrer using an external magnetic field that rotates a small magnetic bar that has been placed in the mixture.
- AHIBA TurboColor (Crewe, United Kingdom):
  This piece of equipment is a state-of-the-art dyeing unit for dyeing cotton and is located at RMIT University, Centre for Advanced Materials and Performance Textiles. The TurboColor moves the liquid in a circular rotation over a wide temperature range.
- Ultrasonic bath (UNISONICS MODEL FXP12DH, Australian Scientific Pty., Ltd, KOTARA, NSW, Australia):

This piece of equipment is an Australian made ultrasonic bath, which is designed for medical, dental, and chemical laboratories and where all forms of contamination are lightly deposited. The manufacturer states that the thermostat control on the bath can go up to 120 °C, but recommends a maximum temperature of 60 °C.

The equipment used for the vacuum-pressure method was a wood treatment plant with a high pressure (up to 1500 kPa = 1.5 MPa) that is located at the Creswick Campus of the University of Melbourne.

Methods

Dye solution preparation

The brown reactive dye of Procion H-EXL was a mixture of Procion Red H-EXL, Procion Yellow H-EXL, and Procion Blue H-EXL. The brown direct dye was a mixture of brand dyes manufactured by DyStar Global Headquarters (Singapore), (Sirius Red F-4BL, Sirius Yellow K-GRL, and Sirius Grey K-CGL).

Each of the dyes (red, yellow, and blue/grey) was prepared by dissolving 1 g of the dye in 100 mL of water in a standard flask. Therefore, a 2% total dye concentration was made using the following dye concentrations: 1% yellow, 0.5% red, and 0.5% blue/grey. A 2% concentration of the reactive Procion Brown P2RN was prepared by dissolving 2 g of the dye in 100 mL of water in a standard flask. All of the dye solutions were made at ambient temperature (20 °C ± 3 °C) in the laboratories.

Soaking (dipping) method

Several dyeing schedules were used in the experiments (Fig. 1). After each stage of the experiments and based on the results, some dye types, dyeing parameters, and equipment were eliminated.

The suitable equipment, dyes, and dyeing parameters used in this study were selected based on the dye penetration percentage, which was determined with ImageJ software (developed by Wayne Rasband at the National Institute of Mental Health, Bethesda, Maryland, USA), and visual veneer grading to assess any damage (curves or cracks).

The first three stages were applied to the following dyeing process:

- A liquor ratio of 10:1 (water:veneer sample weight) was used in all pots,
- 1.0 g/L of sequestering agent (10% solution) was added to the solution,
- The veneers were dyed for half of the required time, 20 g/L of sodium carbonate were added, and then the veneers were dyed for the other half of the required time.

Therefore, the total dyeing time shown in the experimental framework is the sum of the two time periods. In the first stage of the study, only heartwood samples were used. This decision was based on the knowledge that heartwood is less permeable than sapwood (*i.e.*, if heartwood can be dyed, then sapwood can also be dyed).

Detailed dyeing schedules for each of the stages from Fig. 1 are given in Tables 1 through 5.

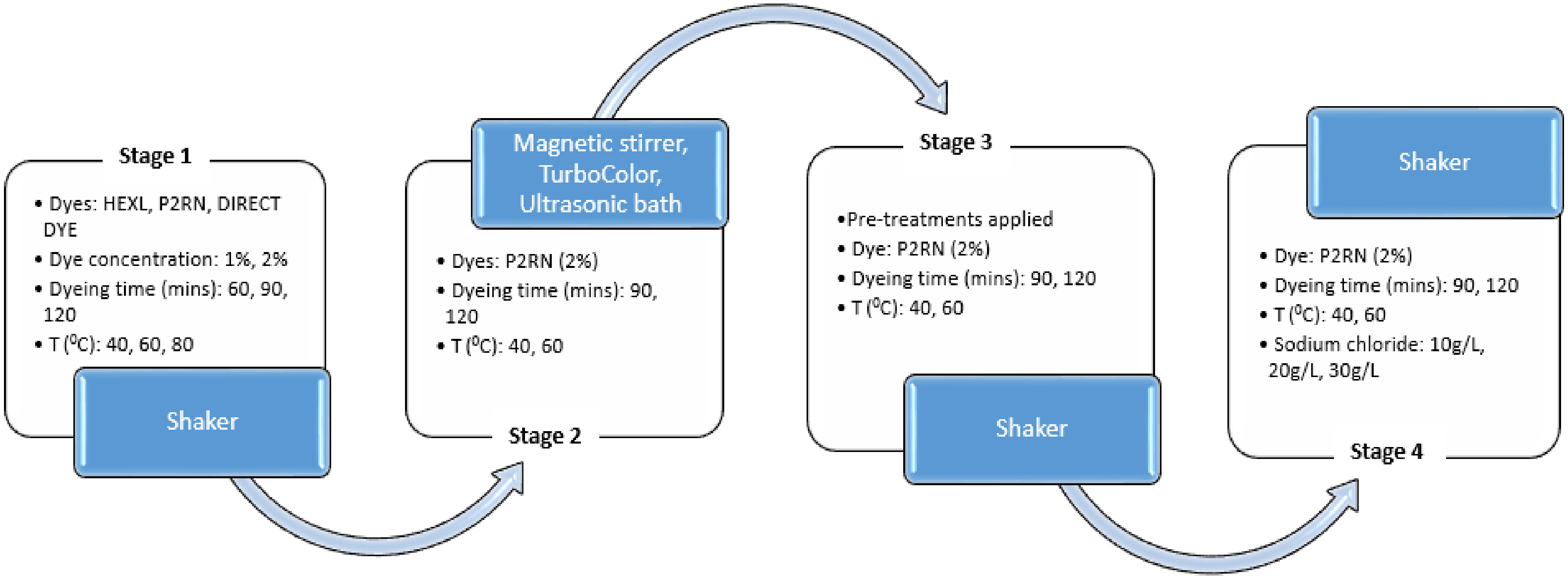

Fig. 1. Overall stages of the dyeing schedules for the soaking method

Table 1. First Stage of the Dyeing Schedules

Moisture content (%)	Green Veneer (80% ± 5%)	Dry Veneer (12%)	
Sample	Heartwood		
Type of dye	HEXL	P2RN	Direct dye
Dye concentration (%)	1	2	
Dyeing time (min)	60	90	120
Dyeing temperature (°C)	40	60	80
Dyeing method	Soaking		
Type of equipment	Shaker		

Based on the dye penetration results from the first stage (details in Table 1), the most suitable dye parameters (dye concentration, dyeing time, and dyeing temperature) were selected and then used in the second stage (Table 2), which mainly compared different types of equipment to determine if these dyeing devices had better results than the shaker.

Table 2. Second Stage of the Dyeing Schedules

Moisture content (%)	Green Veneer (80% ± 5%)	Dry Veneer (12%)	
Sample	Heartwood		
Type of dye	P2RN		
Dye concentration (%)	2		
Dyeing time (min)	90	120	
Dyeing temperature (°C)	40	60	
Dyeing method	Soaking		
Type of equipment	Magnetic stirrer	TurboColor	Ultrasonic bath

It was found that the use of different dyeing equipment did not provide better results than the shaker. The dyed samples did not obtain full penetration at any of the parameters. Therefore, the shaker was used again in the dyeing experiments in Stage 3. Apart from using only the shaker instead of the other equipment, the difference between Stages 2 and 3 was that pre-treatments were applied in Stage 3 prior to the veneer dyeing process. There were two steps in the veneer pre-treatments (Table 3), which were also performed using the shaker.

Table 3. Schedules Used for the Veneer Pre-treatments

Step One				Liquor ratio = 10:1 (water:total sample weight)
Sodium hydroxide (g/L) (10% solution)	0.1	1.0	2.0	
Triton X100 (g/L) (10% solution)	0.1			
Time and temperature	60 min at 40 °C			
Step Two				
Acetic acid (%)	0.5			
Time and temperature	30 min at 40 °C			

The reason for applying sodium hydroxide was to slightly swell the wood fibres, which should make them more accessible to the dye. The details of Stage 3 are provided in Table 4.

Table 4. Third Stage of the Dyeing Schedules

Moisture content (%)	Green Veneer (80% ± 5%)		Dry Veneer (12%)
Sample	Heartwood		
Type of dye	P2RN		
Dye concentration (%)	2		
Dyeing time (min)	90		120
Dyeing temperature (°C)	40		60
Sodium hydroxide (g/L)	0.1	1.0	2.0
Dyeing method	Soaking		
Type of equipment	Shaker		

According to the results of the third stage, 1.0 g/L and 2.0 g/L of sodium hydroxide provided better dye penetration than 0.1 g/L. However, the dyed samples were badly damaged, especially after using 2.0 g/L of sodium hydroxide. Therefore, sodium hydroxide was not used in the fourth stage of the experiments, in which sodium chloride was added (Table 5).

The dyes were all anionic compounds, which have a negative charge in water. Cellulosic fibers when wet also has a negative charge. Therefore, sodium chloride was used to induce a positive charge on the surface of the fibres. Sodium chloride should cause the negative dye to be attracted to the positive cellulose fibres.

In this stage, sapwood samples were also used to confirm that the dyeing parameters would provide satisfactory results for both sapwood and heartwood.

Table 5. Fourth Stage of the Dyeing Schedules

Moisture content (%)	Green Veneer (80% ± 5%)		Dry Veneer (12%)
Sample	Heartwood		Sapwood
Type of dye	P2RN		
Dye concentration (%)	2		
Dyeing time (min)	90		120
Dyeing temperature (°C)	40		60
Sodium chloride (g/L)	10	20	30
Dyeing method	Soaking		
Type of equipment	Shaker		

The use of 20 g/L of sodium chloride in Stage 4 resulted in better dye penetration than the other amounts. The dye penetration was higher and the colour of the dyed samples was brown and evenly distributed on the veneer surface because of the addition of sodium chloride. However, the samples still did not obtain full dye penetration. The dyed samples were again damaged slightly (curved) because of the high dyeing temperatures (detailed results are provided in the Results and Discussion section). Thus, the vacuum-pressure method (Stage 5) was tested.

Vacuum-pressure method

Based on the results of the tests conducted with the soaking method using different dyes and equipment (Stages 1 to 4), a 2% concentration of the reactive dye Procion Brown P2RN was used in Stage 5, which tested the vacuum-pressure method using the wood treatment plant at Creswick Campus. The vacuum-pressure method was performed without and with pre-treatment.

The details of the vacuum-pressure method without the veneer pre-treatment (Stage 5.1) are given in Table 6.

Table 6. Dyeing Schedules for the Vacuum-pressure Method Without Pre-treatment (Stage 5.1)

Moisture content (%)	Green Veneer (80% ± 5%)	Dry Veneer (12%)
Type of dye	P2RN	
Concentration (%)	2	
Sample	Heartwood	
Vacuum time (min)	10	15
Vacuum level (kPa)	-100	
Pressure time (min)	60	120
Pressure (kPa)	500	1000

For the vacuum-pressure method with a veneer pre-treatment (Stage 5.2), several pre-treatments were tested:

- Samples were submerged in 1.0 g/L of sodium hydroxide and 20 g/L of sodium chloride for 1 h prior to the dyeing process (HC1),
- Samples were submerged in 1.0 g/L of sodium hydroxide and 20 g/L of sodium chloride for 24 h prior to the dyeing process (HC24),
- Samples were submerged in 20 g/L of sodium chloride for 1 h prior to the dyeing process (C1),
- A mixture of 1.0 g/L of sodium hydroxide, 20 g/L of sodium chloride, and the dye (HCD) was used,
- A mixture of 20 g/L of sodium chloride and the dye (CD) was used.

All of the pre-treatments were conducted at ambient temperature (20 °C ± 3 °C). Heartwood samples with two MC levels (green sample and dry sample) were used. The dyeing process was similar to the process without a pre-treatment, except the samples were maintained at a vacuum level for 15 min during the vacuum treatment and a pressure of 1000 kPa was maintained for 2 h during the pressure treatment. These optimal dyeing parameters were based on the results of the vacuum-pressure method without a pre-treatment. After the dyeing process, the samples were removed and dried to a 12% MC.

Determination of the Dye Penetration

It is very difficult to measure the cross-section of the veneers using a calliper as the veneers are thin and, in most cases, irregular areas in which the dyes have penetrated cannot be visually observed. Therefore, ImageJ software was used to compute the areas. A dyed veneer was cut using a sharp knife. Then, a 5MP-USB Microscope (distributed by TechBrands by Electus Distribution Pty., Ltd, Rydalmere, NSW, Australia) was used to take pictures of the cross-section of the dyed veneer. The ImageJ software was then used to compute its area and the dye penetration percentage, which is an effective and accurate method. An example how to calculate the percentage of dye penetration using ImageJ software is shown in Fig. 2.

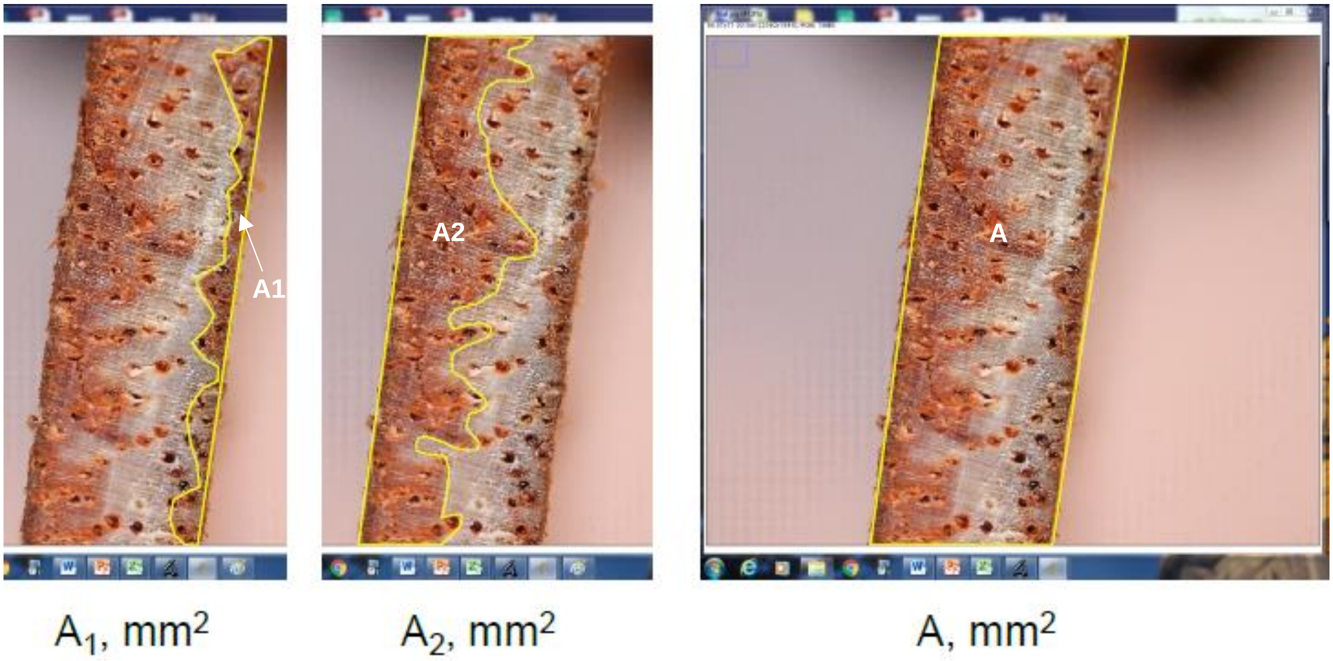

Fig. 2. An example of how to measure the dye penetration percentage by ImageJ

As can be seen from Fig. 2, irregularly shaped selections of areas A, A1, and A2 are defined by a series of line segments. The selected areas were automatically calculated. The percentage of dye penetration was then calculated as (A1+A2)*100/A, %.

Statistical Analysis

An analysis of variance was conducted on the dye penetration percentage with various factors from different stages of the dyeing process as explanatory variables. Main effects and two-factor interactions were included in the model.

The assumption of constant variance was checked with a plot of residual values *vs.* fitted values. Main effects were considered to be significant if the P value was less than 0.05, and two-factor interactions were reported for $P < 0.01$ or close to it. Least Significant Difference (LSD) values at P 0.05 were used to estimate the variability between the means of the samples for each combination of factors. The software GENSTAT (16th Edition, VSN International Ltd, Hemel Hempstead, UK) was used for the statistical evaluation of the data.

RESULTS AND DISCUSSION

Analysis of the Effect of Various Factors on the Dye Penetration during Different Dyeing Stages of the Soaking Method

Based on the data analysis, the effect of different dyes and dye concentrations on the dye penetration at different temperatures, times, and moisture contents of the sample in Stage 1 was determined (Fig. 3).

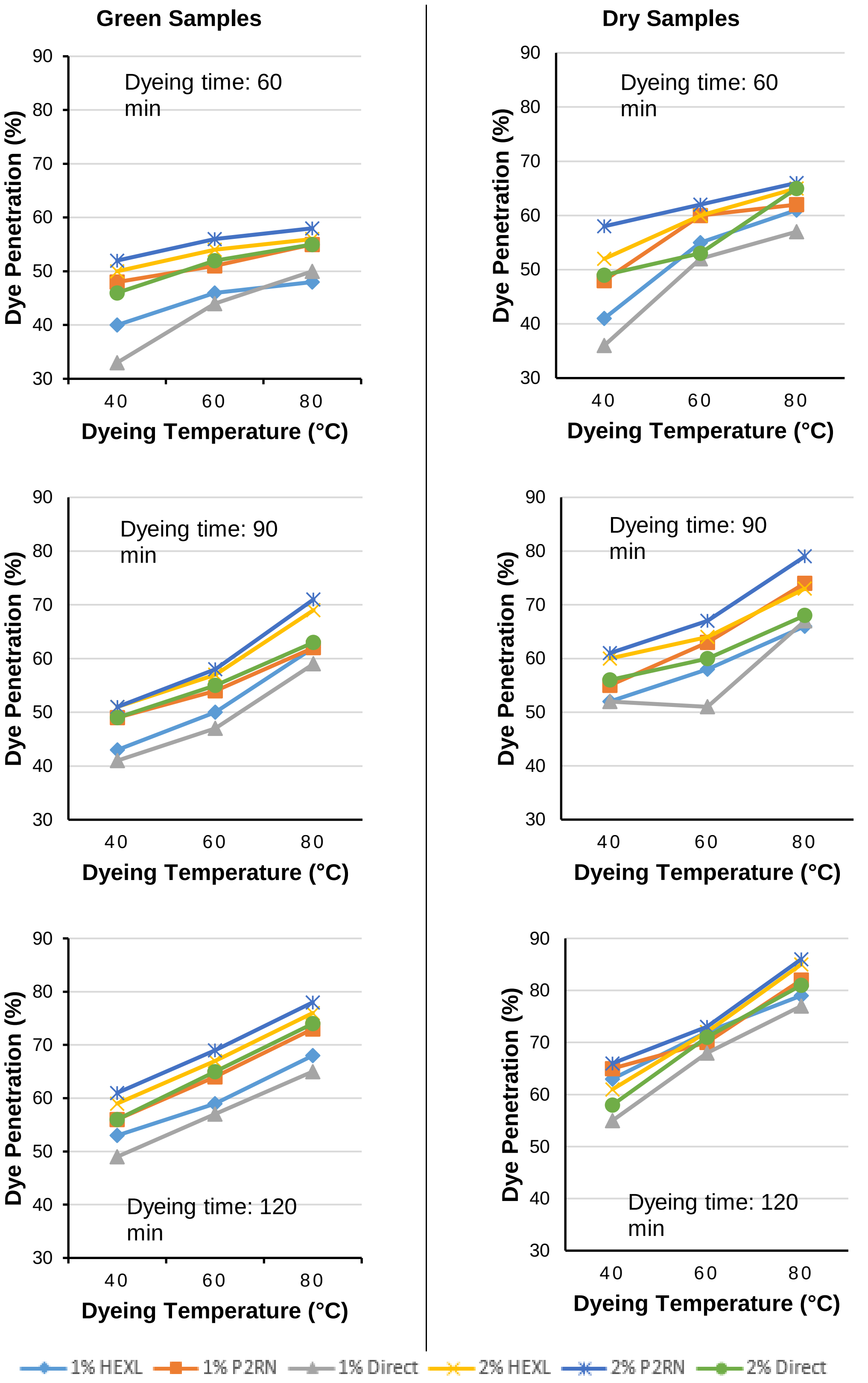

Fig. 3. Effect of the different dyes and dye concentrations on the dye penetration at different temperatures, times, and sample MCs (Stage 1). Note: 1% HEXL: 1% concentration of Procion H-EXL; 1% P2RN: 1% concentration of Procion Brown P2RN; 1% Direct: 1% concentration of the direct dye; 2% HEXL: 2% concentration of Procion H-EXL; 2% P2RN: 2% concentration of Procion Brown P2RN; and 2% Direct: 2% concentration of the direct dye

All main effects – moisture content, dye concentration, and type of dye – were highly significant (P < 0.001). The factors other than the dyeing time and dyeing temperature are shown in Table 7, together with the LSDs.

Table 7. The Influence of the Main Effects on Dye Penetration

Factor	Level	Mean	LSD
MC (%)	Green veneer (80% ± 5%)	56.19	0.84
	Dry veneer (12%)	63.19	
Dye concentration (%)	1	56.80	0.84
	2	62.57	
Type of dye	Direct dye	56.56	1.02
	HEXL	59.64	
	P2RN	62.86	

The interaction between dyeing time and dyeing temperature was highly significant (P < 0.001). The means of the factor combinations and the associated LSD are shown in Table 8.

Table 8. Influence of the Interaction between Dyeing Time and Temperature on Dye Penetration

Dyeing temperature (^{0}C)	Dyeing time (min)			Mean
	60	90	120	
40	46.08	51.67	58.50	52.08
60	53.75	57.00	67.25	59.33
80	58.17	67.75	77.00	67.64
Mean	52.67	58.81	67.58	**59.69**
LSD	**1.77**			

The parameters that resulted in the highest dye penetration were the same for both the green and dry veneers. The samples that were dyed with the reactive dye Procion Brown P2RN at a 2% concentration at 80 °C for 120 min provided the best results in relation to the dye penetration (86% for the dry samples and 78% for the green samples). The dye penetration of the dry samples was 8% higher than that of the green samples. Both the reactive dyes, Procion Brown P2RN and the Procion HEXL mixture, resulted in a higher dye penetration than the direct dye did. For the 2% dye concentration, the percentage of dye penetration reached 86%, which was higher compared with 82% when using the 1% dye concentration.

The reactive dye Procion Brown P2RN gave more favourable results than the other two dyes because it is a monochlorotriazine reactive dye; therefore its reactivity is lower than the Procion HEXL type. This means it should have better penetration through the wood veneer. Moreover, the Procion HEXL type has two reactive groups and therefore may not penetrate easily, as it would react more rapidly than the P2RN. The molecular size of P2RN dye is relatively small compared to the direct dye. The direct dye generally is large molecules and therefore may not penetrate easily through the wood veneer.

The level of dye penetration in all of the specimens significantly increased when the dyeing time increased from 60 min to 120 min. Time is an important factor in veneer dyeing because adsorption occurs on the veneer surface if the dyeing time is too short and it is very difficult to achieve full penetration of the veneer. This effect is reasonable because the wood dyeing process is a dynamic sorption and desorption equilibrium process that transports dye molecules into the wood surface and internal structure (Yu *et al.* 2002; Liu *et al.* 2015).

It was found that increasing the dyeing temperature enhanced the dyeing penetration of the veneer. Temperature is an important factor that affects the reactive performance of reactive dyes. According to the principle of proliferation, when the temperature is higher, then the diffusion coefficient is greater and the diffusion velocity is faster. If the molecular motion of the dye liquor increases with an increasing temperature, the wood dyeing rate and dye uptake increase (Deng and Liu 2010). At 80 °C, the dye penetration of the dry samples dyed for 120 min with a 2% concentration of Procion Brown P2RN was 13% higher than at 60 °C, and 20% higher than at 40 °C. However, the dyed samples were severely damaged (Fig. 4). This was because the wood fibers were swollen at high temperature during the dyeing process and when the dyed veneers were dried, the number of hydroxyl groups diminishes. Diminished hydroxyl groups may cause a curve or damage in the dyed veneers. Therefore, a temperature of 80 °C is not recommended for the veneer dyeing process.

Fig. 4. Samples dyed at 80 °C

For Stage 2 of the dyeing schedules, the obtained results are given in Fig. 5. According to the analysis of variance, all main effects were highly significant ($P < 0.001$) while interactions between the factors were not significant. The factor means are shown in Table 9, together with the LSDs.

Three different dyeing devices were used in Stage 2 of the experiments, and it was evident that the dye penetration of the samples dyed using the AHIBA TurboColor was higher than for those using the magnetic stirrer and ultrasonic bath. However, the results showed that there was little difference between the dye penetration of both the green and dry samples dyed with 2% P2RN at 60 °C for 120 min using the TurboColor (67% and 72%, respectively) and shaker (69% and 73%, respectively).

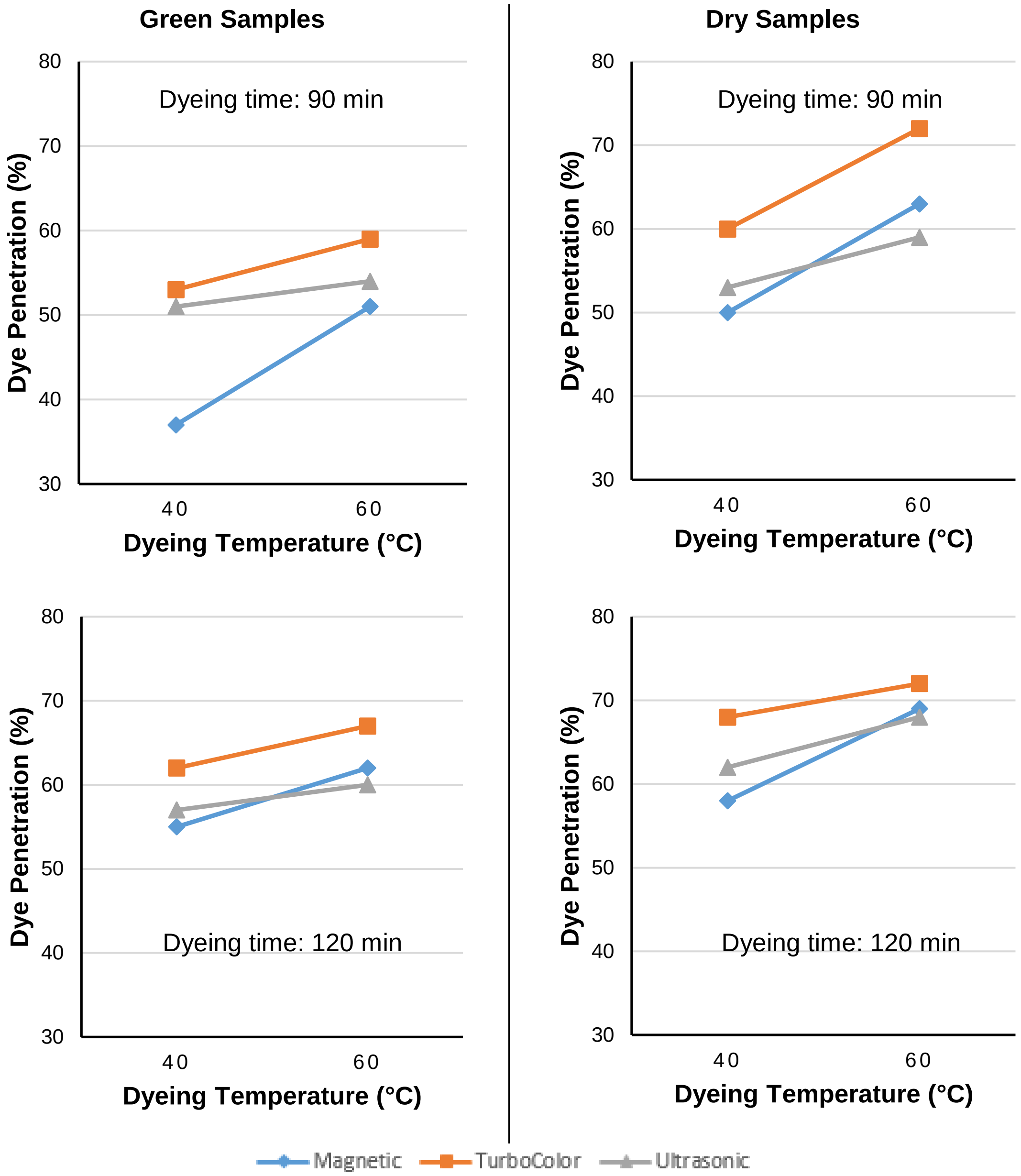

Fig. 5. Effect of different types of dyeing equipment (magnetic stirrer, AHIBA TurboColor, and ultrasonic bath) on dye penetration at different temperatures, times, and sample MCs (Stage 2)

Table 9. Influence of the Main Effects on Dye Penetration

Factor	Level	Mean	LSD
MC (%)	Green veneer (80% ± 5%)	55.67	2.08
	Dry veneer (12%)	62.83	
Dyeing time (min)	90	55.17	2.08
	120	63.33	
Dyeing temperature (^{0}C)	40	55.50	2.08
	60	63.00	
Equipment	Magnetic	55.62	2.55
	TurboColor	64.12	
	Ultrasonic	58.00	

As was expected, the samples dyed at 60 °C for 120 min resulted in a greater dye penetration than those dyed at 40 °C for 90 min. However, the dyed veneers did not obtain full dye penetration at these dyeing parameters. Therefore, pre-treatments were used in the following stage and the results of the optimal dyeing factors are shown in Fig. 6.

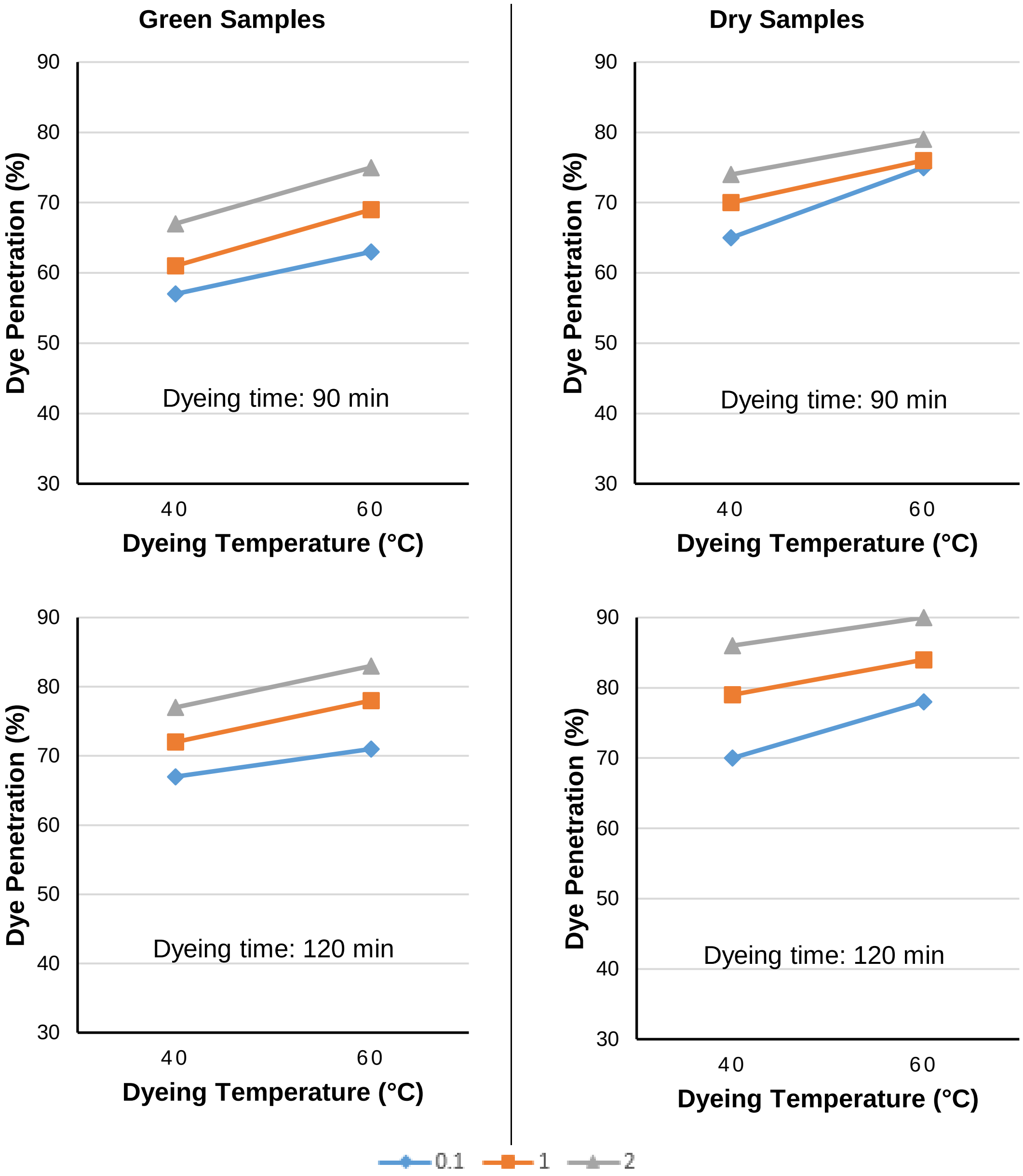

Fig. 6. Effect of the different amounts of sodium hydroxide (0.1 g/L; 1.0 g/L, and 2.0 g/L) on the dye penetration at different temperatures, times, and sample MCs (Stage 3)

The analysis of variance showed a highly significant effect of all factors ($P < 0.001$). None of the interactions were significant. The factor means are shown in Table 10, together with the LSDs.

Table 10. Influence of the Main Effects on Dye Penetration

Factor	Level	Mean	LSD
MC (%)	Green veneer (80% ± 5%)	70.00	1.60
	Dry veneer (12%)	77.17	
Dyeing time (min)	90	69.25	1.60
	120	77.92	
Dyeing temperature (^{0}C)	40	70.42	1.60
	60	76.75	
Sodium hydroxide (g/L)	0.1	68.25	1.95
	1.0	73.62	
	2.0	78.88	

There was a significant increase in the dye penetration when sodium hydroxide was used, especially when the amount was 2 g/L, which resulted in dye penetrations of 90% in the dry veneers and 83% in the green veneers. However, the dyed samples were badly curved (Fig. 7). The pre-treatment with sodium hydroxide causes the cellulose to swell and this increases the ability of the dye to penetrate more easily. The presence of the alkali also promotes the dye fixation as the dye forms a covalent bond with the cellulose via the hydroxyl groups. The dye penetration was improved with an increase in the dyeing time and temperature.

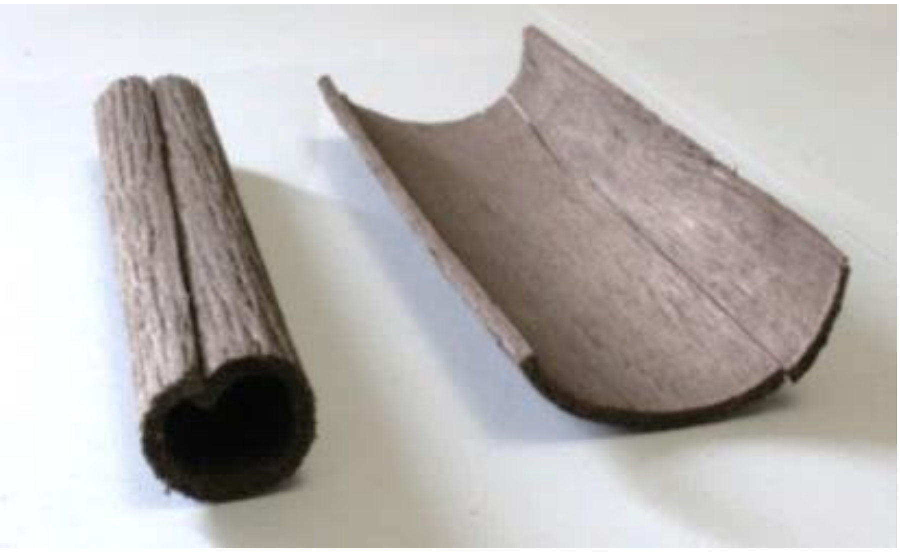

Fig. 7. Samples dyed with sodium hydroxide

After the first three stages, the dyed veneers did not obtain full penetration with any of the dyeing parameters. Thus, Stage 4 was performed with sodium chloride (Fig. 8) and 2% Procion Brown P2RN.

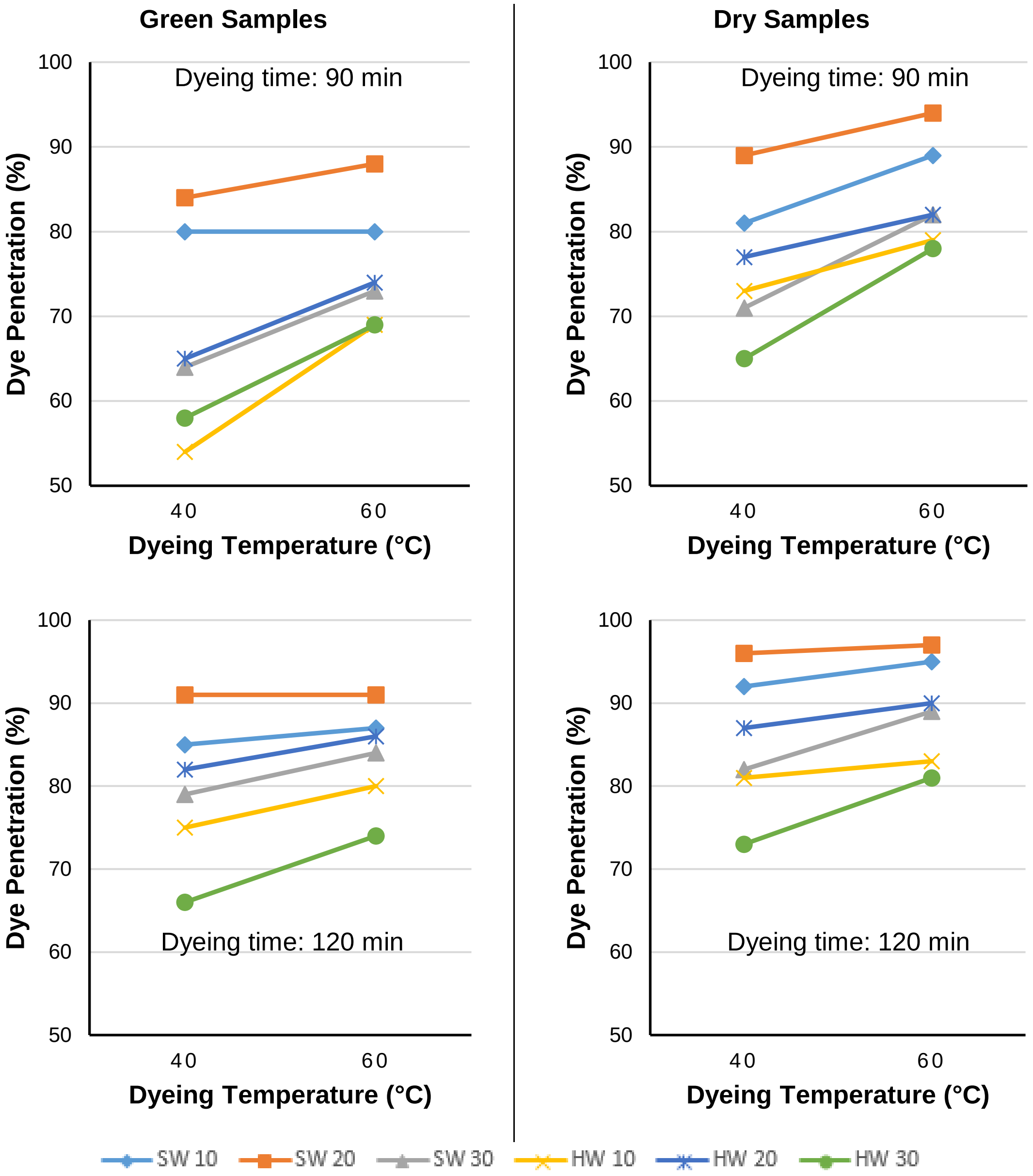

Fig. 8. Effect of sapwood (SW), heartwood (HW), and different amounts of sodium chloride (10 g/L, 20 g/L, and 30 g/L) on the dye penetration at different temperatures, times, and sample MCs (Stage 4)

All main effects were highly significant ($P < 0.001$). The factors other than the dyeing time and dyeing temperature are shown in Table 11, together with the LSDs.

The interaction between dyeing time and the dyeing temperature was significant ($P = 0.013$). The means of the factor combinations and the associated LSD are shown in Table 12.

Table 11. Influence of the Main Effects on Dye Penetration

Factor	Level	Mean	LSD
MC (%)	Green veneer (80% ± 5%)	76.58	1.54
	Dry veneer (12%)	83.58	
Sample	Heartwood	75.04	1.54
	Sapwood	85.12	
Dyeing time (min)	90	75.75	1.54
	120	84.42	
Dyeing temperature (^{0}C)	40	77.08	1.54
	60	83.08	
Sodium chloride (g/L)	10	80.19	1.88
	20	85.81	
	30	74.25	

Table 12. Influence of the Interaction between Dyeing Time and Temperature on Dye Penetration

Dyeing temperature (^{0}C)	**Dyeing time (min)**		**Mean**
	90	**120**	
40	71.75	82.42	77.08
60	79.75	86.42	83.08
Mean	75.75	84.42	**80.08**
LSD	**1.06**		

The highest dye penetration for the dry sapwood and heartwood samples of this stage was obtained at 60 °C and 120 min with 20 g/L of sodium chloride. Almost full dye penetration was obtained for both the dry sapwood and heartwood samples, and were 97% and 90%, respectively.

There were similar trends in the percentage of dye penetration in the green sapwood and heartwood samples. The dye penetration reached 91% for the green sapwood samples and 86% for the green heartwood samples when they were dyed at 60 °C for 120 min with 20 g/L of sodium chloride.

Almost full dye penetration was obtained with the soaking method (Stage 4) at 60 °C and 120 min with 20 g/L of sodium chloride. However, the dyed samples were still slightly damaged (curved) because of the high temperatures applied.

Effect of Different Dyeing Parameters on the Dye Penetration during the Vacuum-pressure Method

The effect of the vacuum time, pressure time, and pressure level during the vacuum-pressure method without pre-treatment was explored and the results are shown in Fig. 9. The Procion Brown P2RN dye was used at a concentration of 2%.

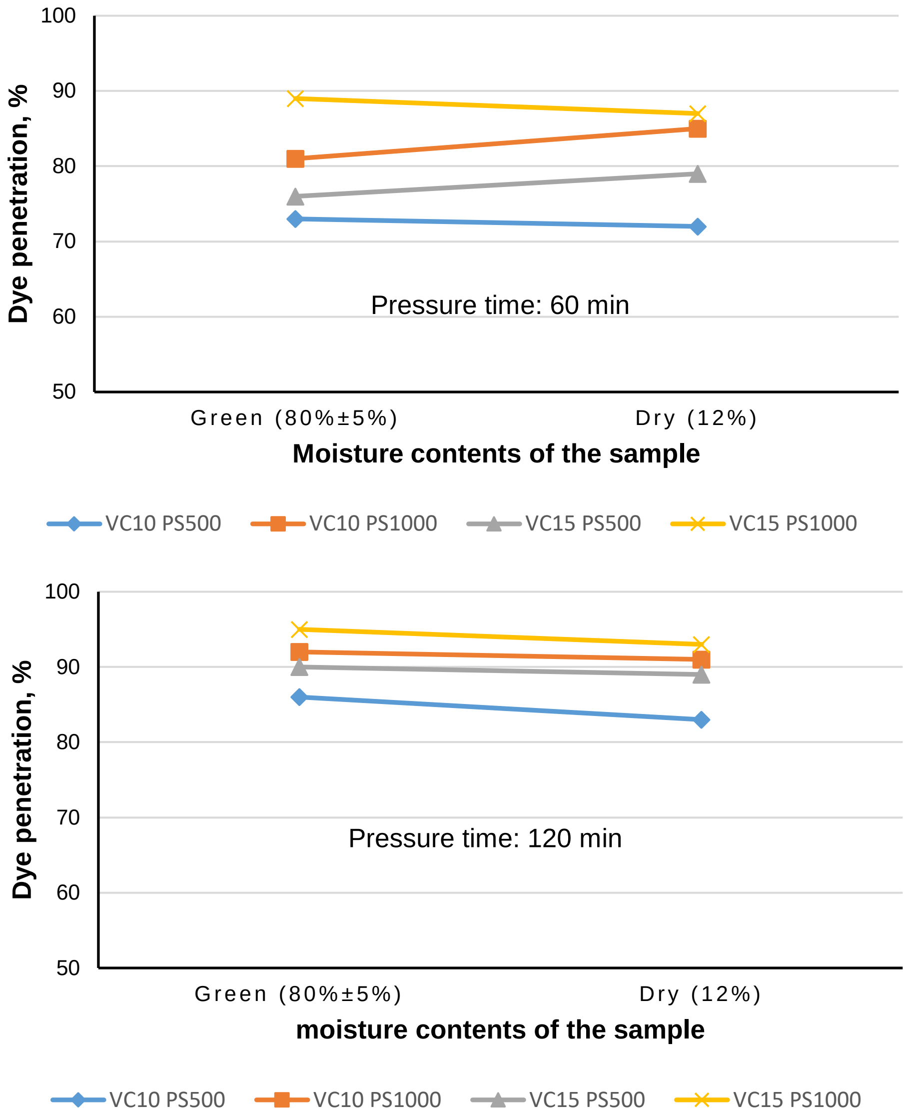

Fig. 9. Effect of the different vacuum times (VC10 and VC15) and pressure levels (PS500 and PS1000) on the dye penetration at different times and sample MCs (Stage 5.1)

Table 13. Influence of the Main Effects on Dye Penetration

Factor	Level	Mean	LSD
Vacuum time (min)	10	82.88	2.29
	15	87.25	
Pressure time (min)	60	80.25	2.29
	120	89.88	
Pressure (kPa)	500	81.00	2.29
	1000	89.12	
MC (%)	Green veneer (80% ± 5%)	85.25	2.29
	Dry veneer (12%)	84.88	

In the analysis of variance, three factors (vacuum time, pressure time, and pressure levels) were highly significant ($P < 0.01$) while the effect of moisture content was not significant ($P = 0.69$). The factor means are shown in Table 13, together with the LSDs.

The highest dye penetration was obtained at a vacuum time of 15 min, pressure time of 120 min, and pressure of 1000 kPa. The dye penetration of the green and dry samples reached 95% and 93%, respectively.

An increased vacuum time allowed more free air and water to be discharged from the veneer, and thus increased the void space in the samples. It was also observed that when the pressure time and level were higher, the results of dye penetration were better.

The effect of various pre-treatments applied in the dyeing process of the vacuum-pressure method on the dye penetration was determined (Fig. 10). All of the experiments were conducted at ambient temperature (20 °C ± 3 °C). The dyeing process with pre-treatment was similar to the process without pre-treatment, except that the samples were maintained at the vacuum level for 15 min and a pressure of 1000 kPa was maintained for 2 h. The dye Procion Brown P2RN at a concentration of 2% was used.

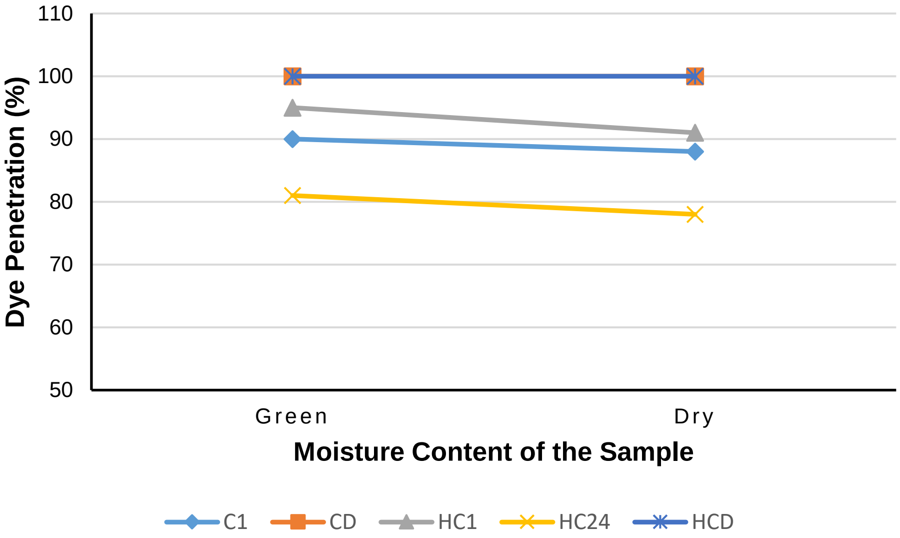

Fig. 10. Effect of different pre-treatments on the dye penetration at different sample MCs (Stage 5.2). Note: C1: samples were submerged in 20 g/L of sodium chloride for 1 h prior to the dyeing process; CD: a mixture of 20 g/L of sodium chloride and the dye was used; HC1: samples were submerged in 1.0 g/L of sodium hydroxide and 20 g/L of sodium chloride for 1 h prior to the dyeing process; HC24: samples were submerged in 1.0 g/L of sodium hydroxide and 20 g/L of sodium chloride for 24 h prior to the dyeing process; and HCD: a mixture of 1.0 g/L of sodium hydroxide, 20 g/L of sodium chloride, and the dye was used

Compared with the soaking method and vacuum-pressure method without pre-treatment (Stages 1 to 5.1), the use of the HCD and CD mixtures had noticeable effects on the dye penetration. Both the green and dry heartwood samples obtained 100% dye penetration. However, because the samples dyed with the HCD mixture were curved, it is not recommended to use this schedule for further experiments. The schedule with the CD mixture resulted in 100% dye penetration and veneer samples with a very good quality without any damage; therefore, it is recommended to use this schedule in further research on the veneer dyeing process of this species.

CONCLUSIONS

1. The shaker resulted in better penetration than the other equipment. The dye penetration was higher and the colour of the dyed samples was evenly distributed on the veneer surface because of the use of sodium chloride. The dyed samples obtained almost full penetration at 60 °C and 120 min with 20 g/L of sodium chloride. However, the dyed samples were still slightly damaged (curved) because of the high temperatures applied.
2. The vacuum-pressure method improved the dye penetration and the effect was greatly strengthened by the use of a 15-min vacuum time at a pressure of 1000 kPa with a mixture of 20 g/L of sodium chloride and the reactive dye P2RN. Both the green and dry heartwood samples obtained full penetration (100%) with the reactive dye (Procion Brown P2RN) at a concentration of 2%, and the dyed samples were not damaged.
3. It is not recommended to use elevated temperatures or sodium hydroxide in the wood veneer dyeing process because the samples were damaged. Therefore, an ambient temperature is recommended for further experiments.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Australian Centre for International Agricultural Research (ACIAR); the University of Melbourne, School of Ecosystem and Forest Sciences; and RMIT University, Centre for Advanced Materials and Performance Textiles. Australian Bluegum Plantations of Victoria is also acknowledged for providing plantation resources, assistance with labour and equipment, and access to the trial site. The authors are indebted to those working at the Salisbury Research Facility Centre at the Department of Agriculture and Fisheries, Queensland, Australia for their great help with specimen preparation and their guidance.

REFERENCES CITED

Castro, G., and Zanuttini, R. (2004). "Multilaminar wood: Manufacturing process and main physical-mechanical properties," *Forest Prod. J.* 54(2), 61-67.

Deng, H., and Liu, Y. (2010). "Study on environmental-friendly dyeing processes of fast-growing plantation veneers," in: *2010 4th International Conference on Bioinformatics and Biomedical Engineering (iCBBE)*, Chengdu, China.

Katz, D. A. (2003). "Dyes and dyeing," (http://www.chymist.com/Dyes.pdf), Accessed 07 April 2016.

Kwiatkowski, A. (2007). *Impregnation of Wood with Stains*, Ph.D. Thesis, University of Melbourne, Melbourne, Australia.

Lacasse, K., and Baumann, W. (2004). *Textile Chemicals: Environmental Data and Facts*, Springer Science and Business Media, New York, NY.

Liu, Y., Hu, J., Gao, J., Guo, H., Chen, Y., Cheng, Q., and Via, B. K. (2015). "Wood veneer dyeing enhancement by ultrasonic-assisted treatment," *BioResources* 10(1), 1198-1212. DOI: 10.15376/biores.10.1.1198-1212

McGavin, R. L., Bailleres, H., Lane, F., Fehrmann, M. R., and Ozarska, B. (2014). "Veneer grade analysis of early-age plantation *Eucalyptus* species," *BioResources* 9(4), 6562-6581.

McGavin, R. L., Bailleres, H., Hamilton, M., Blackburn, D., Vega, M., and Ozarska, B. (2015). "Variation in rotary veneer recovery from Australian plantation *Eucalyptus globulus* and *Eucalyptus nitens*," *BioResources* 10(1), 313-329. DOI: 10.15376/biores.10.1.313.329

Standard Australia (2012). "AS/NZS 2269.0:2012, Plywood-structural," Australian Standard/New Zealand Standard distributed by SAI Global Limited, www.saiglobal.com

Shore, J. (1995). *Cellulosics Dyeing*, Society of Dyers and Colourists, Bradford, UK.

Wagenführ, A., Tobisch, S., Emmler, R., Buchelt, B., and Schulz, T. (2012). "Veneer in interior work," *Initiative Furnier+Natur e.V.*, (http://www.furnier.de/fileadmin/editor/files/englisch/IFN-Bro-Furnier_eng_final_kl.pdf), Accessed 24 December 2017.

Yu, Z., Zhao, L., and Li, W. (2002). "Study on permeable mechanism with dyestuff during wood dyeing," *Journal of Beijing Forestry University* 24, 79-82.

Article submitted: March 8, 2018; Peer review completed: June 5, 2018; Revised version received: July 5, 2018; Accepted: July 6, 2018; Published: July 11, 2018.
DOI: 10.15376/biores.13.3.6444-6464