Wood coloring by reactive stains

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ABSTRACT

The appearance of interior wood products (e.g.: furniture and floors) is often the first criteria that affects customer's interests while making a purchase. One way to diversify the colors and appearances of wood products is by using reactive stains. These coloring systems, consisting of aqueous solutions of metal salts, can penetrate wood and react with its phenolic compounds by forming metal complexes. The color of wood obtained depends on the type of phenolic compounds, type of metal salt, wood surface preparation, temperature, wood humidity, and others. In order to determine the relationship between the structural characteristics of the phenolic compounds and the color developed on wood surface, the polyphenols of two North American hardwood species were extracted and analyzed by different spectrophotometric methods and by liquid state phosphorus-31 nuclear magnetic resonance (NMR) spectroscopy. The chromatic coordinates (CIELAB system) of wood colors obtained after application of reactive stains were compared for these hardwood species. A better knowledge of the reaction mechanisms and the factors influencing them, will allow the optimal use of these systems in wood finishing industries. Since the colored products are present in the wood structure, the wood grain appearance will be preserved or even be accentuated. Enhancing the natural and warm aspect of wood used in buildings interiors can contribute to the well-being of the consumers and promote furthermore the use of this biosourced material.

1. INTRODUCTION

Wood's natural appearance is perceived by the consumers as being warm, welcoming and relaxing (Rice 2006). Therefore, the wood finishing industries seek to diversify wood's color and appearance while preserving or enhancing its natural traits. Wood coloring systems that are most commonly used in industries consist of solvent- or water-based dispersions containing dyes and/or pigments that are applied to wood's surface.

Alternative dyeing systems that can modify wood's color instead of depositing colored substances on its surface, have been developed and discussed in the literature. Two well known methods for wood color's darkening are wood fuming with ammonia (Miklečić, Španić, and Jirouš-Rajković 2012; Weigl et al. 2012; Čermák and Dejmal 2013) and heat treatments of wood at high temperatures (Čermák and Dejmal 2013; Kučerová et al. 2016; Ayadi et al. 2003). The color modification of wood resulting from these treatments is related to changes in the chemical composition of the structural and extractables substances in wood (Barcík, Gašparík, and Razumov 2015; Matsuo, Umemura, and Kawai 2012; Weigl, Pöckl, and Grabner 2009; Kučerová et al. 2016). Both treatments are limited by the shifting of wood color toward brown and black shades (Ayadi et al. 2003; Weigl et al. 2012; Miklečić, Španić, and Jirouš-Rajković 2012). A larger variety of color hues have not been obtained on wood by these treatments.

Heat treatments influence several mechanical properties of wood (Tjeerdsma et al. 1998; Del Menezzi et al. 2009; Kučerová et al. 2016). Kučerová et al. (2016) have reported a slight increase in the bending strength (MOR) and the modulus of elasticity (MOE) with the initial increase of temperatures for heat-treated Fir wood but both properties showed reduced values following the treatment at higher temperatures. Esteves et al. (2008) have also investigated the changes in mechanical properties of heat-treated pine wood and have reported the decrease of the MOR and MOE. They also reported the decrease in wettability which influences the gluing and finishing of wood.

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The decrease in moisture content of heat-treated wood resulted in improved dimensional stability and durability. Ammonia treatment does not influence significantly the mechanical properties of wood while it improves the aesthetic properties and is considered therefore a milder treatment than heat treatment (Weigl et al. 2012). Due to the possible health and environmental risks associated to ammonia exposure, researchers have investigated alternative methods for wood color modification (Machová et al. 2019).

Another possible method for wood coloring is the application of metal salts aqueous solutions to wood's surface (Figure 1) (Leach 1988). These solutions, when applied on wood's surface, can react with the polyphenols present naturally in wood by forming colored complexes (Krilov and Gref 1986; Andjelkovic et al. 2006; McDonald, Mila, and Scalbert 1996). Therefore, the color of wood is modified due to the formation of new colored products and the resulting color depends on the structures of the complexes formed (Malesev and Kuntic 2007; Perron and Brumaghim 2009; Hider et al. 1981). According to Malesev and Kuntic (2007), the intensity and the hue of the colored complexes depend on the chemical structure of the polyphenols and the properties of the metal ion. More precisely, the number and the position of the hydroxyl groups present in polyphenols structure influence the length of the conjugated Pi-bond system and consequently influence the absorption intensity of the complexes. Due to the variation of the quantity and type of polyphenols from one species to another (Stevanović and Perrin 2009), the color of wood obtained by metal salt treatment is strongly dependent on the type of species treated. Other factors, such as wood's surface preparation and drying temperatures can also influence the resultant colors of wood.

Figure 1: Wood coloring by reactive stains

The objective of this study was to determine the phenolic substitution patterns of the polyphenols present in two North American hardwood species, that can be responsible of the differences in colors obtained following the treatment by metal salts solutions. Liquid state phosphorus-31 nuclear magnetic resonance (NMR) spectroscopy was used to analyze the crude ethanolic extracts of the heartwood and sapwood of White oak and Yellow birch.

2. MATERIAL AND METHODS

2.1 EXTRACTIONS

Lumber of two North American species White oak (*Quercus alba* Linnaeus) and Yellow birch (*Betula alleghaniensis* Britton) that do not include any defects were used for these experiments. Sapwood and heartwood of the two wood species were separately milled with a Fritsch cutting mill (Model PULVERSISETTE 19) then sieved. The fractions with particle size between 250 µm and 425 µm were selected and then stored in darkness at -5 °C.

For each type of wood, 15 g of ground wood were extracted at room temperature with 150 mL of 95% aqueous ethanol with continuous shaking (250 RPM) on an orbital shaker (Barnstead Lab Line model 4633) for 24 h. After filtration on a Büchner funnel by vacuum through a Whatman No. 4 paper, the wood powder was additionally washed with 75 mL of 95 % aqueous ethanol. Ethanol was evaporated by vacuum on a rotatory evaporator at 40 °C and the extract was then dried in a vacuum oven at 40 °C until the weight remained constant. All extracts were stored at -20 °C in darkness prior to experiments. The moisture content was determined following the standard method by oven drying at 104°C.

2.2 PHENOL CONTENT

The total phenol content was quantified by the spectrophotometric method of Folin Ciocalteu as described by Royer et al. (2011). The extract solutions were prepared at 200 µg/mL in methanol and the Folin Ciocalteau reagent was diluted 10 times in distilled water. 2.5 mL of Folin reagent was added to 500 µL of the extract solution followed by 2 mL of sodium carbonate solution (75 g/L). The mixture was heated to 50 °C for 10 min. A Varian Cary 50 UV-Vis spectrophotometer was used to read the absorbance at 760 nm. Gallic acid was used to prepare the calibration

curve (y = 0,0103x + 0,0263; R^2 = 0,9982). The results are expressed as mg equivalent of gallic acid per gram of dry extract (mg GA/g dry extract).

2.3 TANNIN CONTENT

The polyvinyl polypyrrolidone (PVPP) method was used to quantify the total tannin content (Royer, Diouf, and Stevanovic 2011). The PVPP was added to the extract solutions (200 µg/mL in methanol) to precipitate the tannins. The mixture was centrifuged (3000 g, 10 min, 4 °C) and the supernatant was collected and analyzed by the Folin Ciocalteu method. The total tannin content corresponds to the difference between the total phenol content of the extract and the phenols remaining in the supernatant.

2.4 ^{31}P NMR ANALYSIS

^{31}P NMR analysis were performed according to the method described by Melone et al. (2013b). The solvent used was a mixture of pyridine d_5 and $CDCl_3$ (1.6/1, v/v). For each extract, 10 to 15 mg is weighed and dissolved in 500 µL of the solvent mixture. 100 µL of the standard solution was added followed by addition of 100 µL of the phosphorylating agent 2-chloro-4,4,5,5-tetramethyl-1,3,2-dioxaphospholane (Cl-TMDP). The internal standard used was endo-*N*-hydroxy-5-norbornene-2,3-dicarboximide (0.1 mol/L). Cr (III) acetylacetonate (5 mg/mL) was added as a relaxation agent. The liquid phase ^{31}P NMR analyses were performed on a NMR Agilent DD2 spectrophotometer 500 MHz with a relaxation time of 15 s, a pulse width of 45° and application of line broadening of 4 Hz. The signal at 132.2 ppm corresponding to the reaction product of water with Cl-TMDP was used as a reference for all the chemical shifts reported in this study.

2.5 COLOR MEASUREMENTS

Color measurements were performed with a X-rite spectrophotometer (model Ci6x) used in SPIN mode. The standard illuminant D65 was chosen with an angle of observation of 10°. Colors were expressed in the CIELab color space (Figure 2) with the coordinates a*, b* and L* respectively defining the red/green, yellow/blue and lightness values.

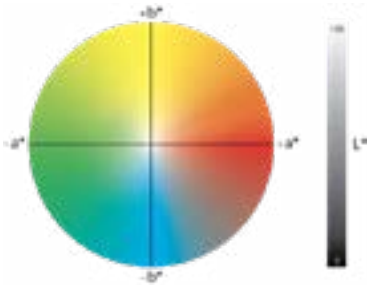

Figure 2 : CIELab color space (Image prepared by Jeremy Winninger)

Ten color measurements were taken on each wood sample. The following equations were used to calculate the color differences of wood surface before and after application of metal salts:

$$\Delta a^* = a_1 - a_2 \quad (1)$$

$$\Delta b^* = b_1 - b_2 \quad (2)$$

$$\Delta L^* = L_1 - L_2 \quad (3)$$

Where a_1, b_1 et L_1 are the chromatic coordinates of the untreated wood samples. The a_2, b_2 and L_2 are the chromatic coordinates of the samples after treatment with reactive dyes. The total color change of the sample after treatment with reactive dyes compared to the colors of the untreated wood are expressed as ΔE:

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

3. RESULTS AND DISCUSSION

3.1 COLOR MEASUREMENTS

The application of reactive dyes on the surface of wood boards of white oak and yellow birch modified the natural wood color for both wood species. The values of the chromatic coordinates L*, a* and b* (Figure 3) decreased for both wood species. The decrease of L* (ΔL* >0) indicates a darker wood color after application of reactive dyes. The decrease of a* (Δa* >0) indicates a shift in wood's color to green shade and the decrease of b* (Δb* >0) value indicates a shift toward blue shades. The total color change is higher for white oak (ΔE = 29.44 ± 2.71) than for yellow birch (ΔE = 8.31 ± 0.66).

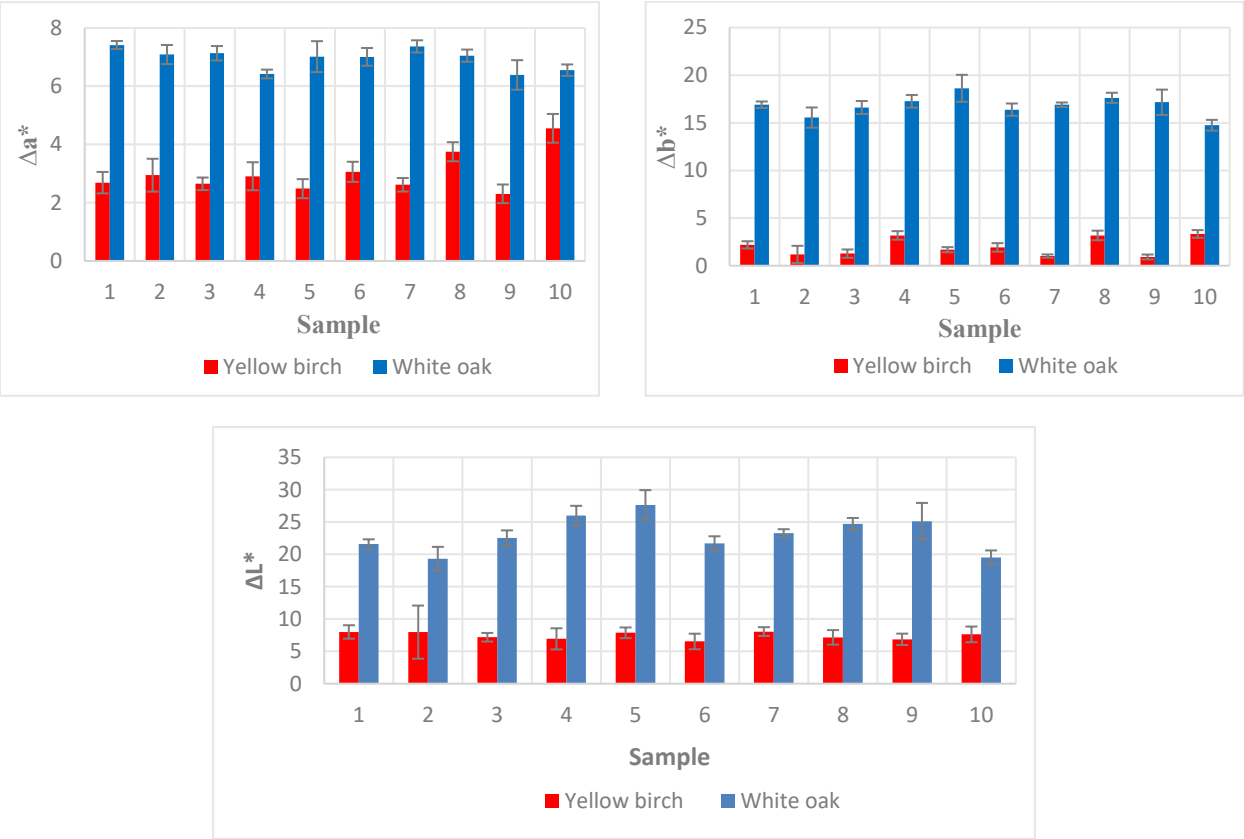

Figure 3 : Chromatic coordinates difference for wood's color before and after application of reactive dyes

3.2 POLYPHENOLS ANALYSIS

The extraction yields and the results of the total phenol and tannin content of the heartwood and the sapwood of white oak and yellow birch are presented in Table 1. The yield obtained for the heartwood of yellow birch was higher than for the sapwood. As for white oak, the yield obtained was higher for the sapwood than the heartwood. Although it was mostly demonstrated in the literature that the heartwood contains a higher amount of extractives than the sapwood (Panshin and Zeeuw 1980), multiple factors can influence the results for different wood species. In the case of white oak, the presence of tyloses in the heartwood (Pallardy 2008) can limit the penetration of the solvent and therefore result in a lower yield of extractives.

Table 1: Extraction yield and polyphenolic composition of extracts of the heartwood and sapwood of white oak and yellow birch

Extracts		Yield (%)	Phenol content (mg GA/g dry extract)	Tannin content (mg GA/g dry extract)
White oak	Heartwood	2.6 ± 00	443 ± 21	367 ± 23
	Sapwood	3.9 ± 0.1	208 ± 14	155 ± 12
Yellow birch	Heartwood	1.7 ± 0.0	258 ± 12	128 ± 7
	Sapwood	1.4 ± 0.0	153 ± 6	82 ± 7
Oligopin ®		-	682 ± 4	625 ± 1

Results are presented as means of triplicates with standard deviation

The ethanolic extracts of the heartwood and the sapwood of the two wood species were analyzed by ^{31}P NMR after phosphorylation with Cl-TMDP. This technique has been widely used to study the structure of lignin (Pu, Cao, and Ragauskas 2011; Argyropoulos 1994; Koumba-Yoya and Stevanovic 2017; Crestini and Argyropoulos 1997). Melone et al. (2013) have demonstrated the possibility of using this analytical technique for the chemical characterization of tannins. In this study, we have applied this method to analyze the crude extracts of wood and to determine quantitatively the different OH substitutions in the structures of the polyphenols present in the extracts. The signals obtained in the spectra were attributed to aliphatic OH, phenolic OH and OH from carboxylic acids according to the chemical shifts presented in Table 2. The quantitative analysis was done by the integration of different signals and the calculation according to the precise quantity of internal standard added to the sample.

Table 2: Signals attribution for ^{31}P NMR spectra (Melone et al. 2013a, 2013b; Pu, Cao, and Ragauskas 2011)

Signals	Chemical shifts (ppm)
Aliphatic OH	150.0 – 145.4
Phenolic OH	144.0 – 137.0
Ortho di-substituted OH	142.46 – 141.06
Ortho substituted OH	140.6 – 137.59
Ortho substituted OH (catechol)	140.2 – 138.3
Ortho unsubstituted OH	137.72 – 137.4
COOH	135.5 – 134.0

The total phenol content determined by the spectrophotometric method of Folin Ciocalteau (Table 1) and by ^{31}P NMR (Table 3) showed that the heartwood of oak wood has a higher phenol content than the heartwood of yellow birch. The sapwood of white oak also has a higher phenol content than the sapwood of yellow birch. The phenol content obtained for the sapwood was lower than the heartwood for the two wood species. The ^{31}P NMR spectra of the ethanolic extracts of the heartwood and the sapwood of the two species (Figures 4 and 5) showed signals in the aliphatic OH, phenolic OH and COOH regions. The comparison of the signals corresponding to the specific types of substitutions of OH showed remarkable differences between the two wood species. The ortho-disubstituted OH signals were present intensely in the spectra of the heartwood of white oak (Figure 4a) and barely present in the spectra of the heartwood of yellow birch (Figure 5a). As for the non-substituted OH, the signals were more intense in the heartwood of yellow birch (Figure 5a) than for white oak (Figure 4a). The signals corresponding to the substituted OH signals were present in the spectra of the two wood species.

The quantitative analysis (Table 3) of the signals showed that the quantity of di-substituted OH present in the ethanolic extract of the heartwood of white oak wood (1.42 ± 0.09 mmol/ g extract) was almost 10 times higher than in the heartwood of yellow birch (0.15 ± 0.03 mmol/ g extract). As for the non-substituted OH, the quantity present in the ethanolic extract of yellow birch (1.01 ± 0.16 mmol/ g extract) was almost 10 times higher than for white oak (0.11 ± 0.01 mmol/ g extract). These results show the presence of di-substituted phenolic compounds in white oak's extracts that can stabilize the metal complexes formed and be responsible of the more intense color change compared to yellow birch (Fiuza et al. 2004).

Moreover, the comparison of the signals in the phenolic OH region of the ^{31}P NMR spectra obtained for the crude ethanolic extract of the heartwood of white oak with the signals obtained for different model compounds in the study of Melone et al. (2013b) showed that the two most intense signals in this region at 139.15 ppm and 141.6

ppm correspond to the ortho-substituted and di-substituted OH specific to the ellagic phenolics. The signals at 138.4 ppm and 141.4 ppm are typical of gallate residues. These results can confirm the presence of ellagitannin, a specific type of hydrolysable tannin in the heartwood of white oak.

Table 3: OH content of phosphorylated ethanolic extracts of the heartwood and sapwood of white oak and yellow birch

		OH (mmol / g extract)					
Extracts		**Aliphatic**	**Phenolic**	**Ortho di-substituted**	**Ortho-substituted catechol**	**Ortho-unsubstituted**	**COOH**
White oak	Heartwood	7.41 ± 0.7	4.74 ± 0.34	1.42 ± 0.09	1.54 ± 0.11	0.11 ± 0.01	0.74 ± 0.18
	Sapwood	14.88 ± 0.1	2.84 ± 0.12	0.57 ± 0.02	0.97 ± 0.03	0.06 ± 0.01	0.42 ± 0.01
Yellow birch	Heartwood	5.43 ± 0.42	3.61 ± 0.45	0.15 ±0.03	0.56 ± 0.08	1.01 ± 0.16	0.52 ± 0.06
	Sapwood	8.77 ± 0.27	2.11 ± 0.07	0.19 ± 0.00	0.79 ± 0.04	0.14 ± 0.01	0.55 ± 0.01

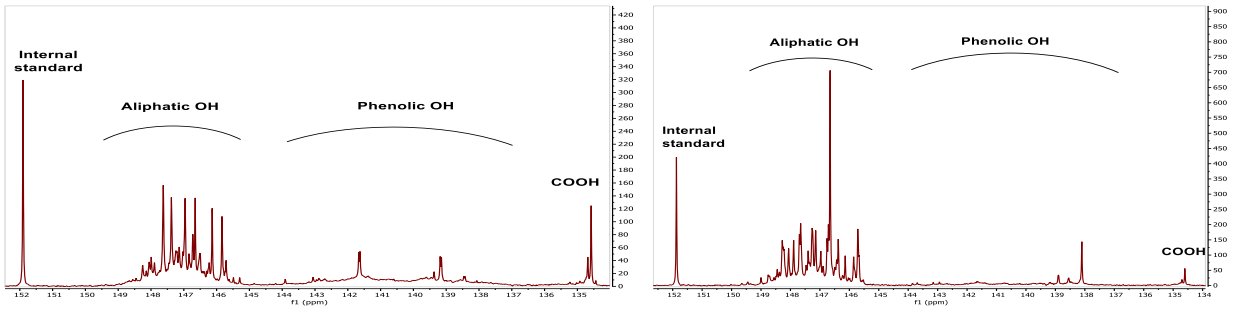

Figure 4: ^{31}P NMR spectrum of the phosphorylated ethanolic extract of the heartwood (a) and softwood (b) of white oak

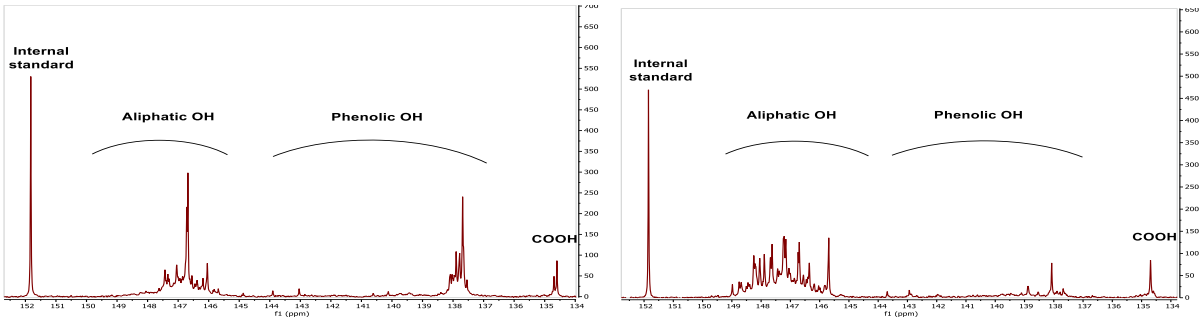

Figure 5: ^{31}P NMR spectrum of the phosphorylated ethanolic extract of the heartwood (a) and softwood (b) of yellow birch

4. CONCLUSION

The chemical characterization of ethanolic extracts of Yellow birch and White oak wood, after phosphorylation with Cl-TMDP by ^{31}P NMR spectroscopy, indicate the differences in the OH substitution patterns of their polyphenols. The presence of OH ortho di-substituted, OH ortho-substituted or OH ortho-unsubstituted can influence the capacity of polyphenols in forming metal complexes. Therefore, the intensity and hue obtained on wood's surface following the application of reactive stains will be dependent on the wood species treated. The better

understanding of the reaction of polyphenols with metal salts and the factors influencing them, will allow the optimal control of the colors obtained in wood finishing industries while using reactive stains.

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