Colorimetric and Chemical Changes in Pre-Hydrolyzed Strand Board Particles of Pine and Eucalyptus

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Changing wood color with physical treatment is an alternative method to avoid the use of chemicals harmful to the environment. However, despite this advantage, there are few studies on the chemistry and colorimetry of pre-hydrolyzed wood. The objective this research was to evaluate the content of extractives, lignin and holocellulose, lightness (*L*), red matrix (*a**), yellow matrix (*b**), saturation (*C*), and tonality angle (*H*) on strand board particles of pre-hydrolyzed pine and eucalyptus. Strand board particles measuring 23 x 90 x 0.30 mm in width, length, and thickness, respectively, were pre-hydrolyzed at 130, 150, and 170 °C during 7 or 21 minutes. The temperature had a significant effect on the chemical composition of wood. The pre-hydrolysis temperature of 170 °C resulted in higher chemical modifications. The pre-hydrolysis reduced the equilibrium moisture content of pine and eucalyptus particles, especially at 170 °C. Color modification due to pre-hydrolysis varied according to treatment conditions. Pine particles were more resistant color changing than the eucalyptus particles.

Keywords: Colorimetry; Oriented strand board (OSB); Pre-hydrolysis

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INTRODUCTION

Pre-hydrolysis is used to degrade hemicellulose in wood for the manufacture of dissolving pulp (Li *et al*. 2010; Liu *et al*. 2013), and cellulosic ethanol (Vila *et al*. 2013). The hemicellulose unfavorably affects the dimensional stability of the wood and can cause problems in the quality of the final product. Some treatments, such as heat treatment, are employed to degrade hemicellulose and increase the dimensional stability of wood and panels (Paul *et al*. 2007; Del Menezzi *et al*. 2009; Mendes *et al*. 2013; Severo *et al*. 2012; Zanuncio *et al*. 2014), however the use of pre-hydrolysis to achieve the same effect has been scarcely studied.

Pre-hydrolysis, in common with other treatments that heat the raw material, changes the color and the chemistry of the wood, which is related to the degradation of hemicellulose (Sundqvist 2004) and phenolic extractives (McDonald *et al*. 1997). The formation and oxidation of compounds such as quinones are also mentioned as causes of colorimetric changes (Bekhta and Niemz 2003). This color change is often regarded as a positive effect, increasing the potential of other wood species to replace expensive hardwoods. The darkening with physical treatments, such as pre-hydrolysis of wood, can add value and extend its use. Such treatment also can prevent inks that emit toluene and xylene, harmful to human health and the environment, for staining wood (Korkut 2012).

The color of wood products influences consumer preferences. Wood of darker tonality is generally regarded as preferable for decorative functions. The color of the treated wood, in addition to the visual aspect, may be used to predict physical and mechanical properties of the solid wood (Bekhta and Niemz 2003) and panels (Del Menezzi 2008).

CIE-*L**a**b** (*Commission International de L' Eclairage*) is an efficient system to determine wood color with colorimeters or spectrophotometers. In this system, *L* indicates lightness and varies between 0 and 100, with zero representing pure black and 100 a perfect white. A positive value of the *a** parameter indicates a red matrix, while a negative value indicates green. A positive value of *b** indicates a yellow matrix, while a negative value indicates blue. The saturation (*C*) indicates the color purity, and the tonality angle (*H*), the dominant hue of a color.

The objective was to evaluate the chemical, colorimetric, and equilibrium moisture content modification, of pre-hydrolyzed particles of pine and eucalyptus.

EXPERIMENTAL

Methods

Production and treatment of particles

Eucalyptus grandis and *Pinus elliottii* logs were obtained on a commercial plantation in the town of Coimbra, Minas Gerais, Brazil, and were processed with a band saw on boards of 23 mm thick. The boards were re-sawn using a circular saw into blocks of 90 mm length. These blocks were submerged in water until completely saturated and processed in a disc flaker with knives adjusted to generate flakes with thickness around 0.30 mm. Resulting flakes measuring approximately 23 x 90 x 0.30 mm (width x length x thickness, respectively) that were heated at temperatures and pressures (Table 1) in a 18,75 L digester (reactor Parr - model 4555) equipped with a heat exchanger (Parr 4848M) using a 10 L / 1.5 kg liquid / particle ratio.

Table 1. Treatments

Treatment	Species	Temperature (°C)	Pressure (bar)	Time (min)
Control 1	Pine	-	-	-
Control 2	Eucalyptus	-	-	-
T1	Pine	130*	2.28	7
T2				21
T3		150	4.48	7
T4				21
T5		170	8.62	7
T6				21
T7	Eucalyptus	130	2.28	7
T8				21
T9		150	4.48	7
T10				21
T11		170	8.62	7
T12				21
* Time to reach the temperature: 130°C – 60min / 150°C – 70min / 170°C – 90min				

The material was inserted in the digester at room temperature and the time to reach each temperature was different. To achieve 130, 150, and 170 °C, required durations of 60, 70, and 90 min, respectively. The liquid used was regular water. Due a high pressure into the digester, the water remained in liquid form. After each treatment time, cooler was turned on and the liquid was removed by a hose located on the inside of the cooler. After removing the liquid, the digester was opened. Then the material was removed and washed.

The control samples were randomly obtained, after being produced in the disk flaker. Treated and non-treated particles were inserted into a climatic chamber adjusted at 65% ± 2% relative humidity and 20 °C ± 2 °C temperature until mass stabilization. Then the equilibrium moisture content (EMC) was determined. Five repetitions were used to determine the EMC, each one with 10 particles.

Colorimetric analysis was carried out for the surface of the wood particles with a Konica Minolta spectrophotometer CM-2500D. Lightness (*L*), red-green or red matrix (*a**), and coordinated blue-yellow or yellow matrix coordinate (*b**), based on the CIE-LAB color system in 1976 were measured. Twenty five samples of each treatment were used to obtain the colorimetric parameters.

The saturation (*C*) was calculated by Eq. 1, and the tonality angle or "hue" (*H*) according to Eq. 2,

$$C = [(a^*)^2 + (b^*)^2]^{0.5} \tag{1}$$

$$H = \arctan\left(\frac{a^*}{b^*}\right) \tag{2}$$

where *C* is the saturation, a* indicates the value along the red/green color dimension, *b** indicates the value of the yellow/blue coloration, and *H* indicates the tonality angle or hue.

Samples of strand board particles were ground in a Wiley mill. The fraction that passed through a 40 mesh and was retained on a 60 mesh sieve was used to determine the chemical composition (ASTM 1982). Three replicates were used for these analyses. Wood ash content was determined according to ABNT NBR 8112 (ABNT 1986), replacing the platinum crucible by porcelain and using a temperature of 600 °C instead of 750 °C. The content of total extractives was determined according to TAPPI standard 204 om-88 (TAPPI 1996), insoluble lignin, following the procedure proposed by Gomide and Demuner (1986), and lignin spectrometry according to Goldschimid (1971). The total lignin content was obtained by the sum of the amounts of soluble and insoluble lignin. Percentage of ash in wood was determined according to ABNT NBR 8112 (ABNT 1986). The holocellulose content was determined by subtracting the lignin, extractives, and ash from 100%.

Statistical analysis

In a preliminary way to assess the analyses of variance, the homogeneity of variance test (Bartlett test at 5% significance), and normality test (Shapiro Wilk test at 5% significance) were performed. The Scott-Knott mean test at 5% significance was used for differentiation among the means of each treatment.

RESULTS AND DISCUSSION

The mass loss of eucalyptus particles was higher than that observed in pine particles for all treatments (Table 2). The particles mass loss for the two species increased with an increase in temperature. Keeping the temperature constant, the longer heating time also caused higher mass loss. The average equilibrium moisture content of treated particles was numerically smaller than the observed value in untreated particles. The lowest equilibrium moisture content was observed on particles treated at 170 °C.

Table 2. Mass Loss and Equilibrium Moisture Content of Pre-Hydrolysed Particles Strand

Temperature (°C)	Time (min)	EMC Pine (%)	EMC Eucalyptus (%)	Mass Loss Pine (%)	Mass Loss Eucalyptus (%)
-	-	$13.6^{(0.03)**}$a	$12.8^{(0.91)}$ a	-	-
130^{*}	7	$12.6^{(0.37)}$ b	$11.9^{(0.02)}$ a	$1.5^{(0.94)}$ a	$3.7^{(1.74)}$ a
	21	$12.7^{(0.44)}$ b	$12.0^{(0.26)}$ a	$2.6^{(1.6)}$ a	$4.5^{(0.99)}$ a
150	7	$12.5^{(0.23)}$ b	$11.5^{(0.35)}$ b	$5.9^{(1.47)}$ b	$6.3^{(0.78)}$ b
	21	$12.6^{(0.06)}$ b	$10.4^{(0.77)}$ b	$6.8^{(2.39)}$ b	$7.9^{(1.89)}$ b
170	7	$10.7(^{0.23)}$ c	$9.5^{(0.09)}$ c	$12.2^{(2.95)}$ c	$13.5^{(2.61)}$ c
	21	$9.8^{(0.04)}$ d	$9.2^{(0.12)}$ c	$14.9^{(1.85)}$ c	$16.3^{(2.4)}$ c
* Mean values followed by the same letter, for each species; in each column, do not differ by the Scott-Knott test at 5 % significance. **The standard deviation are in parentheses.					

Pre-hydrolysis increased the extractives content in the pine particles, probably due to the formation of soluble products in alcohol/toluene (Brito *et al*. 2008). In eucalyptus particles, a significant reduction was observed in particles extractives heated at 130 °C, with an increase at higher temperatures. The reduction in the content of extractives in eucalyptus particles that occurred at the temperature of 130 °C is probably due to volatilization of polar extractives found in greater amounts in hardwoods, since hardwoods decompose between 130 °C and 250 °C (Miranda and Pereira 2002; Meszaros *et al*. 2007; Society and Pereira 2012).

The holocellulose content for the wood of the two species heated at 170 °C was smaller than the holocellulose content under all other experimental conditions. This reduction is probably due to its degradation, which, according to (Barneto *et al*. 2011; Musinguzi *et al*. 2012), occurs at 170 °C and higher temperatures. A reduction of wood hemicelluloses was also observed by Musinguzi *et al*. (2012) and Barneto *et al*. (2011). This explains the increase of the amount of extractives and the decrease in holocellulose content at 170 °C.

The insoluble lignin content increased only in pine particles heated to 170 °C. On the other hand, the soluble lignin content for the particles of both species was greater in the particles heated to 170 °C and further increased as a function of treatment time (Table 3). The total lignin content of the two species was not affected by experimental conditions except for pine particles heated at 170 °C, for which higher values were observed. The more condensed lignin structure with of coniferous woods may explain the better resistance to thermal degradation (Fengel and Wegener 1984).

Table 3. Chemical Composition of Pre-Hydrolyzed Wood of Pine and Eucalyptus

Species	Temp (°C)	Time (min)	Extractives content (%)	Insoluble lignin content (%)	Soluble lignin content (%)	Total lignin (%)	Holocellulose (%)
Pine	-	-	$1.7^{(0.01)**}$ a	$29.66^{(1.38)}$ a	$0.27^{(0.13)}$ c	$29.93^{(1.52)}$ a	$68.37^{(1,52)}$ b
	130^{*}	7	$3.37^{(0,12)}$ b	$28.61^{(0.09)}$ a	$0.24^{(0,07)}$ c	$28.85^{(0.02)}$ a	$67.78^{(0.10)}$ b
		21	$2.96^{(0.03)}$ b	$29.10^{(0,70)}$ a	$0.25^{(0.07)}$ c	$29.35^{(0.63)}$ a	$67.78^{(0.66)}$ b
	150	7	$3.02^{(0.04)}$ b	$29.05^{(0.63)}$ a	$0.26^{(0.01)}$ c	$29.31^{(0.62)}$ a	$67.67^{(0.66)}$ b
		21	$3.02^{(0.02)}$ b	$29.96^{(0.11)}$ a	$0.20^{(0.02)}$ b	$30.16^{(0.14)}$ a	$66.82^{(0.11)}$ b
	170	7	$4.83^{(0.05)}$ d	$31.68^{(0.23)}$ b	$0.18^{(0.05)}$ b	$31.86^{(0.28)}$ b	$63.31^{(0.33)}$ a
		21	$4.11^{(0.20)}$ c	$32.09^{(0.13)}$ b	$0.16^{(0.07)}$ a	$32.25^{(0.20)}$ b	$63.65^{(0.00)}$ a
Eucalyptus	-	-	$2.22^{(0.27)}$ b	$27.73^{(0.43)}$ a	$2.17^{(0.01)}$ c	$29.9^{(0.42)}$ a	$67.88^{(0.69)}$ b
	130^{*}	7	$1.61^{(0.01)}$ a	$27.83^{(0.44)}$ a	$1.97^{(0.01)}$ c	$29.8^{(0.45)}$ a	$68.59^{(0.44)}$ b
		21	$1.77^{(0.05)}$ a	$27.27^{(0.46)}$ a	$1.98^{(0.01)}$ c	$29.25^{(0.46)}$ a	$68.98^{(0.52)}$ b
	150	7	$4.38^{(0.06)}$ d	$26.93^{(0.15)}$ a	$1.68^{(0.01)}$ c	$28.61^{(0.16)}$ a	$67.01^{(0.22)}$ b
		21	$3.13^{(0.01)}$ c	$26.66^{(1.05)}$ a	$1.90^{(0.02)}$ c	$28.56^{(1.03)}$ a	$68.30^{(1.04)}$ b
	170	7	$8.97^{(0.26)}$ e	$28.49^{(0.54)}$ a	$1.25^{(0.01)}$ b	$29.74^{(0.53)}$ a	$61.28^{(0.79)}$ a
		21	$8.77^{(0.18)}$ e	$27.90^{(0.15)}$ a	$1.08^{(0.00)}$ a	$28.98^{(0.14)}$ a	$62.25^{(0.32)}$ a

* Mean values followed by the same letter, for each species; in each column, do not differ by the Scott-Knott test at 5% significance. **The standard deviation are in parentheses.

The color change of pine and eucalyptus wood occurred in all treatments. For both species a lighter color became darker, which is aesthetically advantageous for some applications (Table 4).

Table 4. Colorimetric Parameters

Species	Temp (°C)	Time (min)	ΔL	Δa	Δb	ΔC	ΔH
Pine	-	-	$74.9^{(1.5)**}$ a	$10.2^{(1.6)}$ c	$26.1^{(2.2)}$ a	$28^{(2.4)}$ a	$68.7^{(2.7)}$ a
	130^{*}	7	$-6.0^{(2.2)}$ b	$-0.7^{(1.6)}$ c	$-0.8^{(1.5)}$ a	$-1.0^{(1.9)}$ a	$1.0^{(2.5)}$ a
		21	$-4.5^{(2.3)}$ b	$1.1^{(0.9)}$ b	$-1.7^{(2.2)}$ a	$-1.2^{(2.1)}$ a	$-3.6^{(2.2)}$ b
	150	7	$-6.5^{(2.1)}$ b	$-0.8^{(1.2)}$ c	$-1.2^{(1.4)}$ a	$-1.4^{(1.5)}$ a	$0.7^{(1.9)}$ a
		21	$-10.5^{(1.9)}$ c	$1.7^{(1.7)}$ b	$-1.3^{(1.3)}$ a	$-0.5^{(1.7)}$ a	$-4.2^{(2.6)}$ b
	170	7	$-21.0^{(3.1)}$ d	$4.5^{(1.0)}$ a	$-3.4^{(1.6)}$ b	$-1.0^{(1.5)}$ a	$-11.5^{(2.3)}$ d
		21	$-18.0^{(1.9)}$ e	$3.2^{(3.1)}$ a	$-1.6^{(1.5)}$ a	$0.0^{(2.4)}$ a	$-6.9^{(5.2)}$ c
Eucalyptus	-	-	$74.3^{(2.4)}$ a	$14.4^{(1.8)}$ a	$20.4^{(1.4)}$ a	$25.1^{(1.9)}$ a	$54.8^{(3.1)}$ a
	130^{*}	7	$-7.5^{(2.1)}$ b	$1.6^{(1.8)}$ b	$-2.7^{(0.5)}$ b	$-1.1^{(1.4)}$ a	$-6.7^{(3.0)}$ b
		21	$-8.7^{(1.4)}$ b	$2.0^{(1.0)}$ b	$-2.3^{(1.0)}$ b	$-0.5^{(0.9)}$ a	$-7.0^{(2.4)}$ b
	150	7	$-22.6^{(3.7)}$ c	$4.5^{(2.0)}$ c	$-3.7^{(0.9)}$ c	$0.3^{(1.6)}$ a	$-13.2^{(3.6)}$ c
		21	$-25.1^{(3.2)}$ d	$5.8^{(2.3)}$ c	$-4.9^{(1.0)}$ b	$0.5^{(2.3)}$ a	$-17.1^{(2.4)}$ d
	170	7	$-32.5^{(3.7)}$ e	$2.8^{(1.0)}$ b	$-6.0^{(1.7)}$ e	$-2.6^{(1.6)}$ b	$-15.0^{(3.2)}$ c
		21	$-36.8^{(2.3)}$ f	$0.7^{(0.9)}$ a	$-7.0^{(1.1)}$ f	$-4.8^{(1.3)}$ c	$-13.3^{(2.0)}$ c

*Mean values followed by the same letter, for each species, in each column, do not differ by the Scott-Knott test at 5 % significance. **The standard deviation are in parentheses.

However, the effect was different among species and was affected by the experimental conditions. The color parameters *L* (lightness), *a** (green-red), *b** (blue-yellow), color saturation (*C*), and tonality angle (*H*) of the pine and eucalyptus control, as well as the variation of each parameter compared to control for all pre-hydrolysis treatment can be observed in Table 4.

The particles without treatment for both species showed similar values of lightness index (*L*). However, after pre-hydrolysis, the particles of pine acquired a lighter color than the eucalyptus particles. For both species the value of *L* was reduced, *i.e.* the particles acquired a darker color. Reduction in the rate of luminosity was observed in pre-hydrolysis at 130 °C and 7 min; however, it became more pronounced when the particles were heated at 170 °C for 21 min. The pine wood was more resistant to darkening than eucalyptus.

Similar results were observed for Pincelli *et al*. (2012), who concluded that the conifers woods thermal treated below 160 °C are more resistant to color change than the hardwood. These authors also observed that wood treated at 180 °C had indices with similar brightness. According to Bourgois *et al*. (1991) darkening for heat treatments between 240 °C and 310 °C wood is caused by the reduction of the content of hemicellulose, especially pentosans. According Mitsui *et al*. (2001), heat in the presence of water accelerates the change of color of wood. This may be caused by phenolic components coming from depolymerization of lignin, and also the sugars generated by depolymerization of the cellulose and hemicelluloses. The phenolic compounds coming from the degradation of lignin and sugars coming from the breakdown of cellulose or hemicellulose molecules can produce colored compounds by oxidation processes, which can at least partly explain the phenomenon of darkening.

Generally a change was observed in red-green coordinate (*a**) for both species. Eucalyptus particles became redder at 150 °C while that in pine particles this change was observed only for heating at 170 °C. Moura and Brito (2011) carried out various heating treatments on eucalyptus and pine woods and observed that generally an average higher red hue in eucalyptus (13.06) in the pine (10.60) wood. They also observed that the pine wood showed no significant changes in the level of red depending on the maximum temperature of the heat treatment. However, for the *Eucalyptus* wood the level of red level decreased with increasing temperature.

The *b** values were higher for the flakes of pine than eucalyptus in all treatments. The particles of pine showed significant reduction in this ratio only at 170 °C. On the other hand, eucalyptus particles showed a tendency to decrease in yellow color with increasing temperature.

The work by Moura and Brito (2011) corroborates the reduction in *b** for pine for pine wood subjected to heating at temperatures exceeding 160 °C (Moura and Brito 2011). Also, Bekhta and Niemz (2003) found that wood of *Picea abies* suffered a considerable decrease in yellow after treatments above 150 °C. Pincelli (1999) was another author who observed a reduction in yellow hues in the wood of *Pinus caribaea* var. *hondurensis* when exposed to temperatures exceeding 160 °C. The yellowing is associated with the chromophores in the lignin, extractives, and organometallic complexes found in some extractives. The heat treatment affects the structure and / or quantitate of these compounds, resulting in a color change (Moura and Brito 2011).

The color saturation (*C*) was higher in pine particles than the particles of eucalyptus under all conditions. The saturation of pine wood remained constant in all treatments, while heating at 170 °C reduced this parameter in the case of the eucalyptus wood. The color saturation is calculated as a function of the coordinates *a** and *b**. The *b** value is typically higher than the a* value as measured on wood. Thus it is expected that *b** has a greater influence on the actual values of *C*. Thus, the pattern of variation of *C* is generally similar and dependent to the color yellow.

The tonality angle (*H*) of pine particles was higher than that observed in particles of eucalyptus in all treatments. For eucalyptus all *H* values decreased compared to the control, while pine values didn't show a pattern. In other words, for pine some values decreased and some increased. Pincelli *et al*. (2012) also found in pine wood that *H* values were higher than for eucalyptus wood. However, the tonality angle of pine was reduced by heating while eucalyptus was increased. This reduces the variation pattern of tone difference between species.

Further work will be developed in order to verify the effect of pre-hydrolyzed strand particles on the properties of resulting OSB panels.

CONCLUSIONS

1. The temperature had a significant effect on the chemical composition of wood. The pre-hydrolysis at 170 °C resulted in higher chemical modifications in pine and eucalyptus wood.
2. Pre-hydrolysis reduced the equilibrium moisture content of pine and eucalyptus wood, especially at 170 °C.
3. The species of pine and eucalyptus showed distinct color changes due to the treatment, with greater darkening in eucalyptus.
4. For the eucalyptus wood, the green-red coordinate (*a**) increased in all treatments, reaching a maximum value at 150 °C for 21 min. For pine, the maximum value was found in the 170 °C treatments.
5. The values of the blue-yellow coordinate (*b**), color saturation (*C*), and tonality angle (*H*) had their values reduced after all treatments. For the three parameters, the reduction was greater for eucalyptus than pine.

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