



COLOUR CHARACTERISTICS OF DENSIFIED AND THERMALLY POST-TREATED BEECH AND PINE WOODS

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Abstract

Effect of thermo-mechanical densification and thermal post-treatment on the colour parameters (L^ , a^* , b^* and ΔE^*) of beech (*Fagus orientalis* L.) and pine (*Pinus sylvestris* L.) wood species were investigated. Wood specimens were densified at temperatures of 100 or 150 °C and compression ratios of 20% or 40%. Then, thermal post-treatment was applied to the specimens during 2 h at 190, 200, and 210 °C to provide dimensional stability in densified specimens. The colour change of the specimens was evaluated by CIEL*a*b* colour co-ordinate systems. The results showed that, the colour of the densified specimens at higher temperature and compression ratio was more changed. The compression temperature was more effective compared to compression ratio on the colour change of specimens. After the thermal post-treatment, colour characteristics of specimens have changed significantly depending on the increase in the treatment temperature, and specimens darkened. Densification processes has little effect on colour changes of the thermally post-treated specimens. The colour of pine specimens compared with the beech specimens more affected by the densification and thermal treatment applications. Moreover, the change in a^* value of specimens is more evident than the change in L^* or b^* values.*

Keywords: Wood material, Densification, Thermal treatment, Colour change.

YOĞUNLAŞTIRILMIŞ VE TERMAL İŞLEMLİ KAYIN VE ÇAM ODUNLARININ RENK ÖZELLİKLERİ

Özet

*Kayın (*Fagus orientalis* L.) ve çam (*Pinus sylvestris* L.) odunu örneklerinin renk özellikleri (L^* , a^* , b^* and ΔE) üzerine termo-mekanik yoğunlaştırma ve termal işlemin etkisi araştırılmıştır. Örnekler 100 °C ve 150 °C sıcaklıkta %20 ve %40 sıkıştırma oranı ile yoğunlaştırılmıştır. Daha sonra, yoğunlaştırılmış örneklerde boyutsal stabiliteyi sağlamak için 190, 200 ve 210 °C sıcaklıkta 2 saat süresince örnekler termal işlem uygulanmıştır. Örneklerin renk değişimi CIEL*a*b* renk koordinat sistemine göre değerlendirilmiştir. Araştırma sonuçlarına göre, daha yüksek sıcaklıkta ve sıkıştırma oranında yoğunlaştırılmış örneklerin rengi daha fazla değişmiştir. Örneklerin renk değişiminde, sıkıştırma oranına göre sıkıştırma sıcaklığı daha çok etkilidir. Termal işlem sonrası, örneklerin renk özellikleri işlem sıcaklığındaki artışa bağlı olarak önemli ölçüde değişmiştir ve örnekler kararmıştır. Termal işlemlerdeki renk değişimlerinde yoğunlaştırma işlemlerinin etkisi önemsiz bulunmuştur. Kayın örneklerine göre çam örneklerinin rengi yoğunlaştırma ve termal işlem uygulamalarından daha fazla etkilenmiştir. Ayrıca, örneklerin a^* değerindeki değişim L^* veya b^* değerindeki değişimden daha belirgindir.*

Anahtar Kelimeler: Ağaç malzeme, Yoğunlaştırma, Termal işlem, Renk değişimi

1 Introduction

Enhancement of properties of wood and the expansion of its lifetime have involved scientists for several years as wood material is an versatile and prominent material for an extensive variety of utilizations, for example, manufacturing of furniture and building constructions [1]. Features of wood, such as physical and mechanical, can be enhanced by some modification process. Densification of wood one of this modification process can be used to improvement of physical and mechanical properties. Thermo-mechanical (TM) densification of wood has been considered as a successful technique to improve the mechanical features of particularly low-density wood species [2]. In addition, surface features such as smoothness, brightness, color, hardness, permeability, and durability could be improved by densification modification [2-7]. A major disadvantage of mechanically densified wood is the recovery of its initial dimensions after exposure to water or

heat. The structural steadiness of mechanically densified wood can be significantly increased by thermal post-treatments [3, 8-11].

Thermal modification of wood is an industrially practical technology. It utilizes heat as a means to modify the physical and chemical of wood to obtain desired features [12]. Moreover, thermal modification is a operation that gives the chance to increase of some properties of wooden material without charging an additional load on the environment, as preservatives clearly do. Particularly, thermal modification used to improve dimensional stableness and biological resistance of wood. This improvement is primarily triggered by thermal deterioration of hemicelluloses and, for the most part, the changes of properties continue with the increment of temperature value. Depend on reduction of the amount of humidity content, swelling and shrinkage arise, color darkens, some of the extractives substances leak out, acidity increases

and thermal insulation features are developed [1, 13]. But, the mechanical resistance of thermally modified wooden material may decrease due to mass loss and thermal deterioration, which is the primary disadvantage of thermal processing [14-17]. Thermal treatment gaining importance day by day, and there are a developing number of industrial enterprises in most of countries in the world. Plato Wood in the Netherlands, Thermo Wood in Finland, Rectification in France, and Le Bois Perdure in Québec, are some of the centers specializing in thermal treatment. A portion of the timber types are processed beneath specific conditions, relying on the species and the purpose of the final product. AL of those methods utilizes cut timber and process temperatures between 160 °C and 260 °C [18]. Thermal treatment gives new physical features to wooden material including decreased hygroscopy, enhanced dimensional stability, higher resistance to deterioration by biotics, and most significantly, appealing darker color [19]. Pending thermal process at excessive temperature values, the color of wood material has a tendency to darken because of the noteworthy modification it's chemical composition, which includes the deterioration of the amorphous carbohydrates [20, 21]. Sometimes, color changes arose as a result of thermal treatment may be more preferred for customers. This case especially can be seen on the wood species which have light colors such as pine, beech, poplar [22, 23].

Color is an important factor both in terms of aesthetic properties and economic indicators affecting the pricing of the finished product. By changing the parameters of thermal modifications, conditions can be customized when less decorative wood, could get the color and texture similar to exotic wood species [2]. Otherwise, the amount of colorant penetrated into the plasma membrane, in addition to density, moisture content, texture type of wood, and similar factors may influence color tone of wooden material [24, 25]. The point of this study was to evaluate the effect of thermo-mechanical densification and thermal post-treatment on the colour properties (L^* , a^* and b^*) and the total color change (ΔE^*) of beech (*Fagus orientalis* L.) and pine (*Pinus sylvestris* L.) wood specimens.

2 Material and Method

2.1 Wood material

In this research, Scots pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus Orientalis* L.) woods, which has been extensively utilized in the furniture industry, were used. Specimens were prepared from timbers, which have fresh state moisture content, using a band saw machine. Cuts were performed taking into account sample dimensions as annual rings parallel to the surface (tangent section). The specimens were firstly dried to 12% moisture in an conventional drying furnace which temperature was automatically controlled, and then were cut to the dimensions of 450 × 95 mm (longitudinal direction × tangential direction) and two different thicknesses 12.5 and 16.7 mm (radial direction). Before the densification process, the specimens were held in a conditioning cabin (RH 65 ± 3% and 20 ± 2 °C) until they reached a stable weight [26].

2.2 Densification and thermal post-treatment

The thermo-mechanical densification process was done with a hydraulic press machine at temperatures of 110 °C and 150 °C, with compression ratios of 20% and 40%. After thermo-mechanical densification, thermal post-treatment was carried out on the wood specimens to ensure dimensional steadiness. The thermal treatment was conducted under the protection of water vapor at the temperatures 190, 200, and 210 °C for 2 h. The thermo-mechanical densification and thermal post-treatment processes have been described in detail in a previous

study [7]. After thermal post-treatment, specimens remained in a conditioning cabin (RH 65 ± 3% and 20 ± 2 °C) until they reached a constant weight [26]. The densified and thermal treated specimens were then cut into smaller specimens in the dimension of 80 × 80 × 10 mm³ (longitudinal × tangential × radial direction) and as to be repetitive for 6 times (n=6) for every test variable.

2.3 Colour measurement

The colours of the pine and beech specimens were measured by a colorimeter (BYK-Gardner Spectrophotometer) before and after the treatments. In the three-dimensional CIE $L^*a^*b^*$ colour space, every colour is explained as a point in the Euclidean space defined by three coordinates correlating with the subjective color sensation (Figure 1) [27, 28]. In this space, L^* (lightness) is positioned on the black-white axis ($L^* = 0$ for black, $L^* = 100$ for white), a^* on the red-green axis (red for positive values and green for negative values), and b^* for the yellow-blue axis (yellow for positive values and blue for negative values) [29, 30]. The colour red (+a) and the colour yellow (+b) was separately researched for detected which colour value was affected in each colour throughout treatments and besides, the total change in colour values (ΔE^*) was determined according to ASTM D2244 (2015) using the following Equation (1) [31].

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

Where ΔL^* is the difference in lightness, Δa^* is the difference in a^* coordinate, Δb^* is the difference in b^* coordinate. The lower ΔE^* value found from the computations would demonstrate very little or no difference in color values [32]. Color values were determined before and after the treatment at three different locations for each set of specimens and mean value recorded.

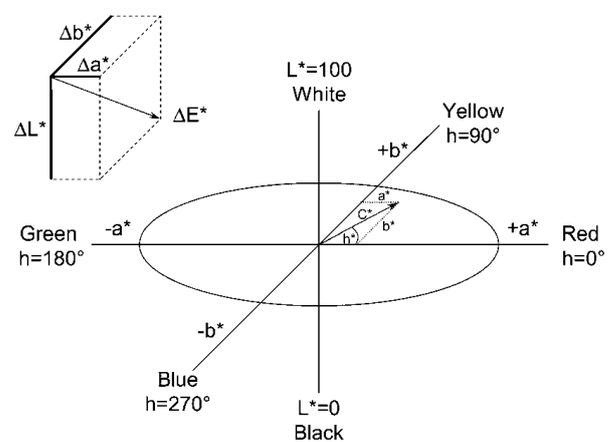


Figure 1. The CIE $L^*a^*b^*$ colour space.

2.4 Statistical analysis

The MSTAT-C software program was used for evaluations of data. ANOVA (analysis of variance) was made to determine the effects of thermo-mechanical densification and thermal post-treatment on the colour values (L^* , a^* , b^* and ΔE) of the pine and beech wood specimens at the 95% confidence level. Significant differences among the groups were compared using the Duncan test.

3 Results and Discussion

After densification and thermal treatment processes, L^* , a^* , and b^* color values of pine and beech wood specimens were changed. Analysis of variance was performed to determine the

influential factors on change in color values of specimens. Analysis of variance results of L*, a*, and b* values of pine and beech wood specimens thermo-mechanical densified and thermal post-treated are shown in Table 1.

Table 1. Analysis of variance (ANOVA) results for L*, a* and b* values.

Test	Wood species	Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance ($P \leq 0.05$)
L*	Scots pine	Densification (A)	4	43.973	10.993	8.8953	0.0000
		Thermal treatment (B)	3	39460.503	13153.501	10643.4099	0.0000
		Interaction (AB)	12	189.625	15.802	12.7866	0.0000
		Error	100	123.584	1.236		
		Total	119	39817.684			
	Eastern beech	Densification (A)	4	20.048	5.012	6.1875	0.0002
		Thermal treatment (B)	3	30534.589	10178.196	12565.4204	0.0000
		Interaction (AB)	12	87.755	7.313	9.0281	0.0000
		Error	100	81.002	0.810		
		Total	119	30723.393			
a*	Scots pine	Densification (A)	4	9.830	2.457	14.0318	0.0000
		Thermal treatment (B)	3	516.472	172.157	982.9961	0.0000
		Interaction (AB)	12	2.827	0.236	1.3453	0.2056**
		Error	100	17.514	0.175		
		Total	119	546.642			
	Eastern beech	Densification (A)	4	1.799	0.450	2.0523	0.0928**
		Thermal treatment (B)	3	794.320	264.773	1207.9097	0.0000
		Interaction (AB)	12	8.447	0.704	3.2114	0.0006
		Error	100	21.920	0.219		
		Total	119	826.487			
b*	Scots pine	Densification (A)	4	45.080	11.270	13.4145	0.0000
		Thermal treatment (B)	3	1169.976	389.992	464.1969	0.0000
		Interaction (AB)	12	34.562	2.880	3.4282	0.0003
		Error	100	84.014	0.840		
		Total	119	1333.633			
	Eastern beech	Densification (A)	4	12.809	3.202	5.1290	0.0008
		Thermal treatment (B)	3	4134.146	1378.049	2207.2241	0.0000
		Interaction (AB)	12	29.913	2.493	3.9927	0.0000
		Error	100	62.434	0.624		
		Total	119	4239.302			

** : Not significant

According to Table 1; densification and thermal treatment factors on L*, a*, b* values of pine and beech wood specimens were significant ($P \leq 0.05$). However, densification factor on a* value of beech wood specimens was found to be insignificant. Comparison results of Duncan test for L*, a* and b* values of

densified and thermal-treated pine and beech specimens are given in Table 2.

Table 2. Comparison results of Duncan test for L*, a* and b* values of pine and beech wood specimens.

Densification	Heat treatment	Scots pine			Eastern beech		
		L*	a*	b*	L*	a*	b*
Undensified	Untreated	82.61 a ^{SG}	4.71 l	22.66 def	67.99 a	11.15 b	22.10 b
	190 °C	43.73 g	10.06 de	22.05 fgh	35.92 e	9.00 c	15.64 d
	200 °C	37.92 k	9.85 ef	18.79 j	29.80 f	7.06 d	11.22 e
	210 °C	31.71 n	8.69 i	14.78 l	26.08 h	4.98 f	7.74 g
110 °C / 20%	Untreated	81.39 ab	5.45 jk	24.04 abc	67.65 a	11.39 ab	22.44 ab
	190 °C	46.51 e	10.38 bcd	23.39 bcde	37.71 d	9.40 c	16.83 c
	200 °C	40.33 ij	10.30 bcde	21.03 hi	28.55 g	6.30 e	9.65 f
	210 °C	32.49 mn	8.66 i	14.89 l	26.64 h	4.49 fg	7.16 gh
110 °C / 40%	Untreated	81.01 b	5.20 k	24.36 ab	67.01 ab	11.89 a	23.18 a
	190 °C	44.64 fg	10.45 abcd	21.92 fgh	36.51 e	9.05 c	15.69 d
	200 °C	41.96 h	10.71 ab	21.81 fgh	28.83 fg	5.90 e	9.11 f
	210 °C	35.30 l	9.33 gh	16.51 k	26.68 h	4.31 g	6.79 h
150 °C / 20%	Untreated	79.37 c	5.54 jk	23.57 abcd	66.04 b	11.07 b	21.92 b
	190 °C	45.42 ef	10.59 abc	23.27 cde	38.14 d	9.32 c	16.82 c
	200 °C	39.44 j	10.22 cde	20.24 i	28.48 g	6.17 e	9.47 f
	210 °C	33.38 m	8.86 hi	15.42 l	25.90 h	4.65 fg	7.02 gh
150 °C / 40%	Untreated	77.71 d	5.80 j	24.61 a	63.89 c	10.94 b	21.60 b
	190 °C	44.77 fg	10.70 abc	22.45 efg	35.54 e	9.03 c	15.22 d
	200 °C	40.83 hi	10.86 a	21.48 gh	29.49 fg	6.24 e	9.39 f
	210 °C	35.38 l	9.44 fg	16.71 k	26.84 h	4.50 fg	6.68 h

SG: Statistical group

With respect to Table 2 results, the highest L* value was obtained in control (undensified and untreated) specimens. For both densified wood species, L* values decreased slightly depending on increase at compression temperature and ratio. Can be effective on results is more of the total press time in the specimens densified at a high compression ratio (40%). Compression ratio has little effect compared to compression temperature on the discolouration of specimens. Similar results were reported by Atik et al. [23] and Bekhta et al. [2]. L* value of pine and beech wood specimens decreased significantly depending on increase of thermal post-treatment temperature and specimens darkened. The lowest L* values were measured in the specimens which thermal treatment is applied at 210 °C. L* value of this specimens was decreased up to 62% compared with control specimens. In previous papers, it has been explained that wood color more darkened with increasing thermal treatment times and temperatures [14, 21, 33-38]. Hemicelluloses degrade in thermally treated wood and thus the lignin content of treated wood increases in parallel. Therefore, changes in wood lightness throughout thermal process are observed primarily because of the degradation of hemicelluloses and wood colour gets to be darker from the starting point of thermal treatment. Degradation of hemicelluloses is enhanced by increase of thermal treatment temperature [19, 2]. In addition, the reduce of the lightness (L*) of thermal treated woods due to changes in the principal structure of cell wall components. However, the reduces in the colour values based on the thermal treatment terms and on the structural components of woods, which is associated with the depolymerization reactions in wood components throughout

the thermal treatment. In particular, extractives and hemicelluloses, which are lesser durable to heat than cellulose, are the basic factors for change natural colour in high-heat processes [39]. On the other hand, it was observed that L* value of densified pine specimens compared with undensified specimens less affected by the thermal post-treatment application.

After densification, a* values of pine wood specimens have increased up to 23%. The a* values was higher in pine specimens compressed 40% at 150 °C. The a* values of densified beech wood specimens have increased up to 7%. Higher a* values was found in beech specimens compressed 40% at 110 °C. According to Bekhta et al. [2], beech and pine wood veneers tend to become redder during the thermo-mechanical densification process. Beech and pine veneers have red colour due to the reduction of a tonality component in colour and slightly increasing colour saturation. After thermal post-treatment, a* value of pine wood specimens increased significantly. The highest a* values were obtained in pine specimens heat-treated at 190 and 200 °C. The a* values of these specimens increased up to 114% compared to un-treated pine specimens. After thermal post-treatment, increases in a* value were lower in densified pine specimens. The a* values showed a decreasing trend in pine specimens thermal post-treated at 210 °C compared with 190 and 200 °C. After thermal post-treatment, a* values of beech wood specimens decreased significantly depending on temperature increase. The highest a* value was measured in the beech specimens without thermal post-treatment, while the lowest a* value was measured in the beech specimens subjected to thermal post-treatment at 210

°C. The a^* values of these specimens decreased up to 64% compared with un-treated beech specimens.

The b^* values of densified pine wood specimens have increased up to 9%. The b^* values was found to be higher in pine specimens compressed 40% at 150 °C. Additionally, the b^* values of densified beech wood specimens have increased up to 5%. Higher b^* values was obtained in beech specimens compressed 40% at 110 °C. The b^* values decreased slightly in beech specimens densified at 150 °C compared to undensified specimens. However this situation not statistically significant. In the literature it was stated that the b^* values of beech and pine veneers increased with rising densification temperature and

pressure, describing the trend to become yellower due to increasing colour saturation. The tones of yellow color of wood are firstly determined by the photochemistry of the principal wood components, especially lignin [2]. After thermal post-treatment, the b^* values of pine and beech specimens decreased based on temperature increase. The highest b^* value was measured in the specimens without thermal post-treatment, while the lowest b^* value was found in the specimens thermal post-treated at 210 °C. The b^* value decreased up to 71% in beech specimens and 38% in pine specimens after thermal post-treatment at 210 °C.

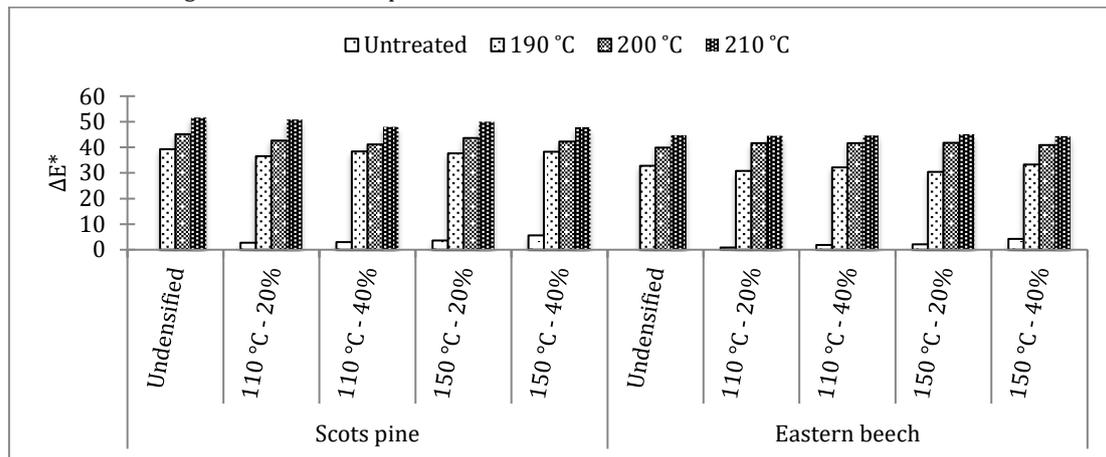


Figure 2. ΔE^* values of the densified and thermal post-treated pine and beech wood specimens.

According to the comparison results taken at reference of control (undensified and untreated) pine and beech specimens (Figure 2), the higher ΔE^* values was observed in pine wood compared with beech wood. The findings of the present study were consistent with previous study results [21]. Pine wood specimens more affected by the densification and thermal treatment applications. Compression temperature according to compression ratio more effective on ΔE^* values of densified specimens. The ΔE^* value was higher in the specimens compressed at a high temperature (150 °C). However, the main factor affecting ΔE^* values is the thermal post-treatment application. The ΔE^* values of specimens increased significantly based on temperature increase after thermal post-treatment. The highest ΔE^* values were obtained in the specimens thermal post-treated at 210 °C. In the literature, it was reported that the total colour change of wood well associated with temperature and duration of thermal treatment for Eastern beech and Scots pine. The total colour change (ΔE^*) of thermal-treated Eastern beech and Scots pine were changed from 5.69 to 47.55 and 6.26 to 58.90, respectively. The total colour changes of thermal-treated Eastern beech and Scots pine woods increased with increasing thermal treatment temperature and duration [21].

4 Conclusion

In the densification process, the compression temperature has more effect in comparison with compression ratio on the colour change of specimens. The colour of the densified specimens at higher temperature (150 °C) was more changed. The thermal post-treatment application has a significantly effect on L^* , a^* , b^* , and ΔE values of specimens. The L^* values of pine and beech specimens decreased up to 62% depending on increasing temperature of thermal post-treatment and specimens darkened. Similarly, The b^* values decreased up to 71% in beech specimens and 38% in pine specimens depending on increase of treatment temperature. In addition, the a^* value decreased up to 64% in the beech wood

specimens. However, the a^* value increased up to 114% in the thermal post-treated pine wood specimens. In general, pine wood specimens more affected by the densification and thermal treatment applications.

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