Color changes and dimensional stability in fir wood (Abies Borissi-regis Mattf.) Modified by heat treatment

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Abstract: It is widely accepted that heat treatment of wood ameliorates its dimensional stability and color evolution. This study has analyzed the effects of heat treatment in color changes and hygroscopic properties of fir wood (Abies Borissi-regis Mattf.), exposed at 200° C for 2, 3 and 4h. Dimensional stability and absorption were measured after 1 day and 3 days immersion in water of 20 ° C \pm 3 temperature. Color evolution and color changes were analyzed in three different directions using the CIE L*a*b* color space. The results showed that heat treatment resulted in a darkened color and an improvement in the dimensional stability of wood. The visual changes in color were more distinct after 4h treatment. The total color differences (ΔE) of the surface of wood substrates appear to be directly correlated with the treatment time. Swelling percentage of the samples was found to be affected by the thermal treatment intensity. The higher percentage of swelling decrease was recorded at approximately 19.61% in 4h treatment duration in a tangential direction.

Keywords: Abies Borissi-regis Mattf., Color, Dimensional stability, Heat treatment.

I. Introduction

Due to growing environmental concerns regarding the use of certain classes of preservatives, there has recently been a renewed interest in wood modification. Wood modification is a process of altering the material the aim being to overcome or ameliorate one or more of its drawbacks. Heat treatment of wood, an old and easy method of modification, has been commonly deployed since the middle of the last century and is nowadays produced industrially in several European countries. Heat treated wood is mainly used for garden furniture, windows, doors and wall or fence boarding, bathroom cabinets, floor material, musical instruments and kitchen furniture (Kartal et al 2008., Korkut and Hiziroglou., 2008).

Wood modification has many capacities, it can bring about an enhancement in decay resistance or dimensional stability, reduce water sorption, improve weathering performance and a number of other things. The application of heat to wood produces degradation associated with chemicals changes in the material. Thermal modification is invariably performed between the temperature of 180° C and 260 ° C. Thermal modification at temperatures lower than 140 ° C results in only a slight change in material properties and higher temperatures cause unexpected degradation to the substrate.

Wood darkening, and therefore color change, occurs due to both the temperature applied and time of the treatment. Treatment in produces greater color change compared to other treatments (Bekhta and Niemz., 2003). The changes in the physical properties of wood (color changes) with heat treatment is mainly the effect of a combination of the following factors: Heat degradation of the chemical constituents of wood and the removal or migration of extractives and other compounds (Korkut and Kocaefe., 2009). Li et al (2011) studied the effects of heat treatment on color change of Douglas fir and observed that the visual color changes arise drastically after heat treatment above 180 ° C. Li et al also indicate that in species with pale colored wood, usually considered less appellative, the darkening would be an important advantage of the heat treatment giving the wood a "tropical flavor" highly valued in many countries. Guller (2012) when evaluating the color change of Pinus nigra wood, noticed that a longer duration of time and/or higher temperature gives a darker color to wood.

From earlier investigations, it is known that wood exposed to high temperatures becomes more dimensionally stable. Schultz et al (2008) stated that because of the chemical alteration of the wood cell wall structural polymers, the sorption behavior of the thermally treated wood is, therefore, altered. It was also highlighted that reduced hygroscopicity was most pronounced at higher relative humidity up to 70%. Significant part of literature is devoted to the idea that the hygroscopicity of heat treated wood can be considerably diverse with varying time and temperature. The decrease in the equilibrium moisture content of wood due to heat treatment leads to an improvement of wood dimensional stability. One of the studies reporting this, stated that it was possible to reduce the deformation caused by swelling by approximately 52% in spruce and 55% in pine

just by adjusting the temperature. The increase in dimensional stability is also dependent on the wood direction and appears to be higher in tangential direction than in radial (Esteves and Pereira., 2009). Many researchers concluded that heat treatment of wood resulted in a large reduction in the hemicellulose content, and thus, improves the dimensional stability of the wood (Bekhta and Niemz., 2003). Esteves and Periera (2009) stated that in spruce, fir and poplar wood, the radial and tangential swelling has always been slighter in treated wood, and continued to decrease as the treatment become more severe. The improvement in dimensional stability depends on the wood species and the wood direction. The increase in dimensional stability for heat treated wood is mainly due to the decrease of wood hygroscopicity in view of the chemical changes at high temperatures. (this sentence does not make sense - the 'in view of' needs to change) The decrease of swelling has already been examined by other researchers such as Sahin (2010), who noticed that fir and pine wood improved as concerns swelling in all directions after heat treatment at 190 ° C and 212 ° C for 2h. The results were statistically significant compared to the untreated specimens. As for heat treated fir wood, the reduction reached 40.6%. According to Sahin (2010) swelling is a phenomenon occuring under the fiber saturation point due to moisture absorption. Guller (2012) found out that pine soft wood resulted in a significant reduction in swelling percentage, especially in long durations under water (Metsa and Kortelainen., 2010, Tuong and Li., 2010). Gunduz et al (2008) also concluded that decreases in swelling to radial and tangential directions were found to be 34.46%, 51.73%, respectively.

Cao et al (2011) investigated the dimensional stability of Chinese fir, finding that the heat treatment remarkably improved the dimensional stability and specifically increased as the treatment temperature and length of time increased too. Comparing the effect of the temperature and duration on dimensional stability, the temperature played a more determining role. Sahin (2010) studied the swelling and shrinkage of fir wood (*Abies bornmulleriana Mattf.*) and concluded that these physical properties improved in all directions indicating that the availability and accessibility of the free hydroxyl groups of the wood carbohydrates play an important role in the process of water absorption and desorption. The heat treatment altered the chemical structure of the wood, especially the hydroxyl groups.

Tjeerdsma et al (1998) mentioned that one of the main reasons for the increase of the dimensional stability is the loss of the methyl radials of some guaiacylic and siringic units of lignin which lead to an increase of phenolic groups and an increase of the units proportion with free ortho positions. These chemical changes lead to higher lignin reactivity, with the formation of several crosslinks, responsible for the increase of dimensional stability. Through the increase of crosslinking, the molecule becomes less elastic and the cellulose microfibrils are less likely to expand and absorb water, explaining the decrease of the equilibrium moisture and the improvement on the dimensional stability.

The present study was conducted to assess the effect of soft condition heat treatment on color change and dimensional stability of *Abies Borissi-regis* Mattf.fir wood, one of the most significant commercial wood produced in Greece.

II. Material And Methods

In this study, fir wood (*Abies Borissi-regis* Mattf.) was obtained from the Pertouli district of Trikala province in central Greece. Lumber from the logs was prepared in the wood products laboratory of the Forestry faculty, Aristotle university of Thessaloniki. Initially, lumber was cut in sawn samples of dimension 50 x 25 x 3 mm in axial section. These flitches were conditioned in the laboratory for about 1 year under $20 \pm 2^{\circ}$ C and 60 ± 5 % relative humidity. The wood samples were weighed and then dried in the oven at 103° C±2 for 24h and reweighed, according to ISO 3130:1975, in order to estimate the mean moisture content of our material. The average moisture was 11.03% (SD 0.18) while the average density (oven dry weight/volume at 11.03% (SD: 0.18) moisture) of timber used were 0.391 g/cm³ (SD 0.037). The flitches were thermally treated in an oven at 200 ° C in which the temperature could be controlled to $\pm 3^{\circ}$ C and for three different periods of time (2, 3 and 4h) in the presence of air. The flitches were placed in the unit after the desired temperature had been reached and following thermal treatment the flitches were conditioned for 15 days. The final specimens for swelling and absorption were prepared according to ISO 4859:1982. The dimensions of the specimens were 20mm x 20mm x 30mm, with the length in axial direction. For each variable, 10 specimens were prepared. The swelling (in radial and tangential directions) and the absorption percentage was conducted after the immersion of the samples in the water of $20 \pm 3^{\circ}$ C for 1 and 3 days.

With the aim of determining the heat treatment effect on color change, a colorimeter Minolta Croma-Meter CR-400 was used and the test was carried out according to EN 7224-3:2003. The colour measurements of the specimens were recorded on the surface of the wood specimens before and after heat treatment in radial, tangential and longitudinal directions. For both treated and untreated samples, three sides (radial, tangential and longitudinal) of 17 specimens were measured, 66 in total. Colour changes were studied by the CIELAB system (Figure 1). This system is characterized by three parameters, Lightness (L*) from 0% (black) to 100% (white), a* from green (-a) to red (+a), and b* from blue (-b) to yellow (+b) which are used for colour opponent dimensions and C* for the chroma or saturation. The a* and b* chromaticity coordinates range from +128 (+a) for red to -127 for green and +128 (+b) for yellow to -127 (-b) for blue, respectively. The L*, a* and b* were determined for untreated and treated specimens and their variation with regard to the treatment (Δ L*, Δ a*, Δ b*) was also calculated. From the L*a*b* values, the difference in the lightness (Δ L*) and chroma coordinates (Δ a* and Δ b*), saturation (C*) (Δ C*) and total colour difference (Δ E) meaning the distance in L*, a*, b* colour space were calculated using the following equations.

$\Delta L^* = L^*t - L^*ut$	Equation (1)
$\Delta a^* = a^*t - a^*ut$	Equation (2)
$\Delta b^* = b^* t - b^* c$	Equation (3)
$C^* = (a^*2 + b^*2)^{1/2}$	Equation (4)
$\Delta \mathbf{E} = (\Delta \mathbf{L}^* 2 + \Delta \mathbf{a} * 2 + \Delta \mathbf{b}^* 2)^{1/2}$	Equation (5)
$\Delta C^* = C^* t$ -Cut	Equation (6)

where, L*t, a*t, b*t are L*, a* and b* of the heated specimens L*ut, a*ut, b*ut are L*, a* and b* of the control specimens, respectively. One way analysis of variance (ANOVA), comparing the differences of values at 0.05 level was examined in order to determine the significant differences among various heat treatment combinations on color change and hygroscopic properties.

III. Results And Discussion

Mean values, standard deviation and coefficient of variation of the obtained color changes of treated wood for three different directions are presented in Table1. According to the results, the duration heat treatment has influenced color values. Fir wood showed a darker tone for all heat treatment conditions. The wood darkening was clearly visible and increased with treatment duration. As expected, the treatment decreased the Lightness (Δ L) and yellowness (Δ b) of fir wood in contrast to redness (Δ a) which increased, but the changes are not particularly important except for Lightness after 3h and 4h treatment. The highest Lightness reduction, was observed at 3 h treatment duration where it was noticed the values to be double compared to the untreated samples. Moreover, the coordinates a* and b* changed by the treatment conditions but not significantly. The most remarkable finding of these results is that the values of longitudinal direction are lower especially for a* coordinate meaning that longitudinal direction affected deeper than others direction due to parenchymal cells direction. Akgul and Korkut (2012) found out that the color coordinates of yellow (+b) and blue (-b) for Uludag fir wood treated for 120 ° C, 150 ° C and 180 ° C for 6h and 10h presented a high variation in the heat treated samples, with increasing yellowness initially and decreasing yellowness later under more severe conditions.

Regarding L* coordinate, as shown in figure 2, tends to decrease after heat treatment as the duration increases in a gradual and uniform way. Radial direction gave the highest values and longitudinal the lowest. More specifically, average lightness (L*) of the control specimens was about 76.96, 76.83 69.95 for radial, tangential and longitudinal direction, respectively, and decreased slightly in the first treatment to 69.39, 68.37 and 62.62, respectively (Fig 2). Between 3h and 4h treatment, there was a reduction of about 1.3%, not considered significant. As stated above, as much as L* coordinate approaches 0, this results in black becoming the colour which, in turn, darkens the wood.

Chromaticity coordinates a* and b* did not follow the same trend as lightness. In the case of a* values (Fig.3) for the untreated samples, they appear to be 4.36, 4.38 and 4.82 for radial, tangential and longitudinal direction. Whereas for 2h, treated samples measurement they were found to be slightly increased without significant differences. The higher percentage of improvement was recorded between 2h and 3h treatment about 1.4%. The lower increase was observed in longitudinal direction. According to a* coordinate features, the fir wood after heat treatment tends to change to the colour red.

A drift to the yellow colour (b*) was observed, according to the results and b* values (Fig. 4) which means that the appearance of the wooden surface approaches the yellow colour more than the blue one, as showed the corresponding axis showed (Fig 1). In particular, b* values referring untreated specimens reached 20.57, 20.1 and 19.5 for radial, tangential and longitudinal direction. After the first treatment for 2h, the values appeared to improve more than others about 24% abruptly. A gradual reduction of the values of b* coordinate was recorded after 3h and 4h treatment, not significantly important and naturally without approaching the untreated specimens values.

The same progress with L^* coordinate between the two last treatments was observed for a^* and b^* values meaning that between 3 and 4 hours of treatment the wood discoloration was not as intense as in the beginning.

These three chromaticity coordinates were used in order to compute the color difference (ΔE) and saturation index (ΔC) of total color change. The highest color deference (ΔE) values were 18.32 after heat treatment for 4h compared to untreated samples and they appeared to be correlated with treatment duration. The

improvement appears to be particularly noticeable between the last two treatments in relation to the first one (2h). Observing the last two treatments, they seem to have no significant differences. Gonzales-Pena and Hale (2009) mentioned that ΔE could be a better predictor and a more reliable indicator than ΔL for most properties of wood. On the other hand, saturation index (ΔC) seems to decrease with the duration but the reduction is not considered significant.

The results of this study is consistent with other findings in literature on different tree species. Gunduz et al (2011) mentioned according to their results in Uludag fir that the samples treated at 210 ° C were found to be darker than the other ones treated at lower temperatures. In general, total color difference values seem to be higher at high temperatures due to total color change. L* values usually decreased after heat treatment, but a* and b* values generally increased. The data of this study agree with those of previous studies except for Δb which decreased but this is not significantly important.

Li et al (2011) investigating the color change of Douglas fir, mentioned that the most interesting finding is that the darkening of the treated samples occurred evenly throughout the wood and not only on the surface when duration is over 1 hour. Guller (2012) studied the color change of softwood (Pinus nigra) at 200 ° C for 1, 2 and 3h and found out that the color values decreased after treatments and the highest decreases in L* were recorded in severe and mild conditions, respectively. Guller also mentioned that the negative values of (Δ L), (Δ b) and (Δ a) indicated that the color became darker while increasing temperature and duration.

Moreover Akgul and Korkut (2012) found out that Lightness of Uludag fir wood was significantly influenced and showed a darker tone under all treatment conditions but the maximum lightness reduction was obtained under the most intense treatment. Uludag fir wood presented the highest color difference (ΔE) value of about 12.81 after 10h treatment at 180 ° C in comparison with Scots pine softwood which appeared similar values, about 12.02.

Table 2 shows the average values of water absorption and swelling of the treated and untreated specimens after 1 and 3 days immersion in water.

Swelling percentage of all thermal treatment was lower than this of the untreated specimens and the lowest percentage of about 2.58%, was recorded in 4h treated specimens, after 1 day immersion. Compared to three days immersion, the swelling rate for 1 day was higher found to be affected by the thermal treatment intensity (Tab. 2). Additionally, the effects were found to be greater when the higher duration treatment was applied and higher the duration, the lower the swelling percentage was after the immersion of the samples in water. A marginally higher swelling percentage was recorded in tangential direction, as expected.

The results depicted here indicate that lower water absorption (34.86%) was observed for 4h treated specimens after 1 day immersion in contrast to untreated specimens where the percentage of absorption was 44.11%. The differences among the control samples and those treated for 2 and 3h were relatively minor and the wood samples were not altered significantly. The reduction of absorption after 3 days immersion in water was not considerable although the specimens of every treatment conditions presented about 2% reduction as the duration of treatment was increasing.

The effect of heat treatment on EMC and density of fir wood at $20^{\circ}C\pm 2$ and $60\pm 5\%$ conditions after four months is presented in table 3. Regarding moisture content, it was observed that the samples subjected to treatment conditions got lower moisture content in contrast to moisture of samples before treatment even though they were conditioned to get the EMC. The reduction in moisture content at treated samples compared with untreated was 48,86%, 51,19% and 53,49% after 2h, 3h and 4h respectively. The higher the treatment temperature is and the longer the treatment time, the lower the EMC. Previously published reports clearly indicate that heat treatment can significantly decrease moisture absorption in wood, higher temperature with longer duration cause the lowest hygroscopic property of wood. Based on the combined effect of the above factors, the OH groups available for moisture adsorption are significantly reduced by the heat treatment, which, in turn, decreases the hygroscopicity and EMC of wood (Li et al., 2001).

As a result of this study, we determined that heat treatment causes mass losses in the wood, which has a negative effect on density. Thus, the greatest density loss occurred for treatment conditions of 200 °C and 4h, estimated at about 2.78%, whereas the minimum density loss about 0.76%, occurred at treatment conditions of 200 °C and 2h (Table 3). The result is similar to previously published reports for different species (Guller., 2012; Gunduz et al., 2009). Generally, the degradation of hemicelluloses into volatile products and the evaporation of water and extractives of wood were considered to be the main reason why density decreases.

IV. Conclusions

In conclusion, the results showed that heat treatment at 200 $^{\circ}$ C for three different durations (2h, 3h and 4h) resulted in a darkened color and decreased dimensional stability of fir wood. Specifically, longer treatment duration affected the discoloration but not significantly, due to big dimensions of treated flitches. The highest color deference (ΔE) values were recorded after heat treatment for 4h and appeared to be correlated with

treatment duration. Saturation index (ΔC) seemed to decrease with the duration, but the reduction is not considered significant.

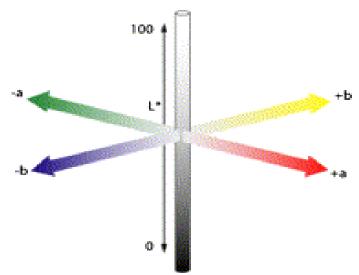
The lowest percentage of swelling after 1 day immersion was recorded in 4h treated specimens whereas the highest swelling rate was measured after 1 day immersion without being significantly important. Furthermore, larger decreases were found to occur in the tangential direction rather than in the radial. Heat treatment also reduced wood density and EMC depending on time and temperature of treatment conditions

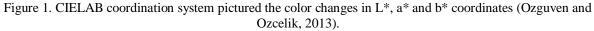
Heat treatment is considered an environmentally friendly process producing more stable products with alluring appearance because of the color. Due to its "new" appearance in color and stability, heat treatment is a viable method and the resulting products can be applied outdoors such as garden furniture, window frames e.t.c. From this research, it can be ascertained that under relatively soft conditions of heat treatment, we can be led to improved color and hygroscopic properties of this important commercial wood that is produced in Greece.

Further studies are in progress to determine the influence of heat treatment in more intense conditions (Temperature-time) and its effectiveness in other properties of fir wood.

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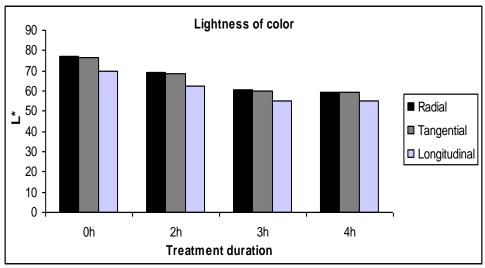


Figure 2. Effect of heat treatment duration in three different directions on L*

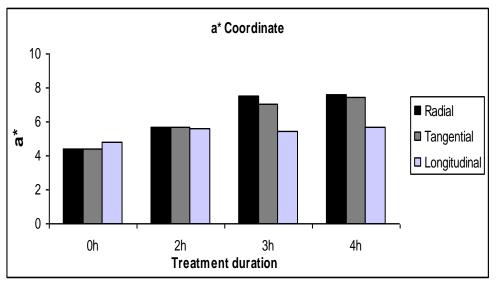


Figure 3. Effect of heat treatment duration in three different directions on a*

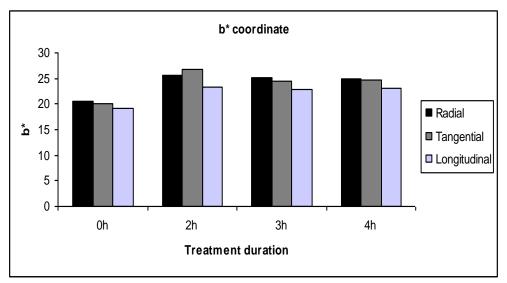


Figure 4. Effect of heat treatment duration in three different directions on b*

Heat			Fir wood				
treatment	Direction	Unit	Color change values				
Time (h)			$\Delta \square$	$\Delta \mathbf{L}$	$\Delta \mathbf{a}$	$\Delta \mathbf{b}$	$\Delta \mathbf{C}$
	Radial	Avg.	9.27	-7.59	1.36	4.95	5.2
		±s	2.38	2.66	0.41	0.77	0.7
		S ²	5.67	7.09	0.17	0.59	0.5
		CV	0.25	0.35	0.30	0.15	0.1
	Tangential	Avg.	9.27	-7.59	1.36	4.95	5.1
2h		$\pm s$	0.55	0.64	0.31	0.35	0.3
		8 ²	0.30	0.41	0.09	0.12	0.1
		CV	0.05	0.07	0.24	0.05	0.0
	Longitudinal	Avg.	8.65	-7.33	0.76	4.27	4.3
	-	±s	1.28	1.66	1.04	0.75	0.5
		S ²	1.64	2.75	1.09	0.57	0.3
		CV	0.14	0.22	1.36	0.17	0.1
	Radial	Avg.	17.46	-16.50	3.15	4.54	5.1
		±s	4.09	4.26	0.8	0.68	0.6
		S ²	16.77	18.19	0.64	0.47	0.3
		CV	0.23	0.2	0.25	0.15	0.1
	Tangential	Avg.	15.54	-14.59	2.63	4.42	4.9
3h	-	±s	2.67	2.92	0.64	0.75	0.6
		S ²	7.13	8.55	0.41	0.56	0.4
		CV	0.17	0.2	0.24	0.17	0.1
	Longitudinal	Avg.	15.43	-14.65	0.59	3.86	3.9
		±s	4.75	5.38	0.54	1.72	1.5
		S ²	22.62	29.02	0.30	2.98	2.4
		CV	0.30	0.36	0.92	0.44	0.4
	Radial	Avg.	18.32	-17.32	3.26	4.42	5.1
		$\pm s$	3.97	4.32	0.77	1.61	1.5
		S ²	15.82	18.73	0.59	2.6	2.3
		CV	0.21	0.24	0.23	0.36	0.3
	Tangential	Avg.	18.24	-17.21	3.07	4.66	5.2
		±s	4.29	4.57	0.60	1.84	1.8
4h		S ²	18.43	20.9	0.36	3.39	3.2
		CV	0.23	0.26	0.19	0.39	0.3
	Longitudinal	Avg.	15.92	-15.11	0.89	4.08	4.1
	-	±s	5.44	5.83	0.66	2.04	1.9
		S ²	29.65	34.01	0.44	4.16	3.9
		CV	0.34	0.38	0.74	0.5	0.4

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Treatment		Tangentia	1	Radia	1	Duration of i
			Swelling	g %		Absorpti
e II. Swellin	g and absorption	presentence	of treated a	and untreated	l fir wood	
		CV	0.34	0.38	0.74	0.5
		S ²	29.65	34.01	0.44	4.16
		$\pm s$	5.44	5.83	0.66	2.04
	Longitudinal	Avg.	15.92	-15.11	0.89	4.08
		CV	0.23	0.26	0.19	0.39
4h		S ²	18.43	20.9	0.36	3.39
		±s	4.29	4.57	0.60	1.84
	Tangential	Avg.	18.24	-17.21	3.07	4.66
		CV	0.21	0.24	0.23	0.36
		S ²	15.82	18.73	0.59	2.6
		±s	3.97	4.32	0.77	1.61
	Radial	Avg.	18.32	-17.32	3.26	4.42
		CV	0.30	0.36	0.92	0.44
		8 ²	22.62	29.02	0.30	2.98
		$\pm s$	4.75	5.50	0.54	1./2

Table I. The effect of heat treatment for three different durations on color changes in fir wood.

			Absorption %					
Treatment duration		Tar	Swelling % Tangential Radial			Duration of immersion		
	Unit	Duration of immersion		Duration of immersion		_		
		1 day	3 days	1 day	3 days	1 day	3 days	
	Avg.	3.20	3.34	4.85	5.05	44.11	62.94	
	$\pm s$	0.45	0.34	0.58	0.61	8.07	13.10	
Oh	S ²	0.20	0.11	0.34	0.37	65.14	171.81	
	CV	0.14	0.10	0.12	0.12	0.18	0.20	
	Avg.	3.03	3.12	5.10	5.40	43.27	60.29	
	±s	0.50	0.56	0.80	0.87	5.79	8.60	
2h	S ²	0.25	0.32	0.64	0.76	33.54	74.09	
	CV	0.16	0.18	0.15	0.16	0.13	0,14	
	Avg.	2.84	2.95	5.18	5.33	42.91	58.48	
	±s	0.19	0.24	0.93	0.92	5.12	8.59	
3h	S ²	0.03	0.06	0.87	0.85	26.29	73.90	
	CV	0.06	0.08	0.18	0.17	0.11	0.14	
	Avg.	2.58	2.77	4.96	5.14	34.86	55.68	
	±s	0.80	0.56	0.64	0.69	13.87	10.71	
4h	S ²	0.64	0.32	0.41	0.48	192.55	114.70	
	CV	0.31	0.20	0.13	0.13	0.39	0.19	

Table III. Equilibrium moisture content attained after heat treatment and conditioning at 20°C and 65% relative	e
humidity.	

Treatment Duration	EMC%	Percentage of decrease %	Density g/cm ³	Percentage of decrease %
Untreated	11.03	_	0.39	-
2h	5.64	48.86	0.387	0.76
3h	5.38	51.19	0.385	1.46
4h	5.13	53.49	0.379	2.78