THE EFFECTS OF HEAT TREATMENT ON THE PHYSICAL PROPERTIES OF JUVENILE WOOD AND MATURE WOOD OF *EUCALYPTUS GRANDIS*

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Heat treatment can be used to improve the physical properties and durability of wood. The results achieved by heat treatment can be affected significantly by various factors. Juvenile wood and mature wood from the same trunk have different properties, and the effects of heat treatment on their physical properties have not been well defined. Thus, a study to determine the differences in the physical properties of juvenile wood and mature wood of *E. grandis* after heat treatment was conducted. Samples of both types of wood were treated at temperatures of 120, 150, and 180 °C for durations of 4, 6, and 8 h. The results showed that the physical properties of juvenile and mature wood, *e.g.*, swelling, moisture content, and fiber saturation point, did not decrease to the same extent. Mass loss of mature wood was higher than that of juvenile wood. Generally, percentage decreases of volumetric swelling, moisture content, and fiber saturation point of juvenile wood were more affected than those of mature wood.

Keywords: E. grandis; Heat treatment; Juvenile wood; Mature wood; Physical properties

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INTRODUCTION

The heat treatment method for modifying wood increases dimensional stability and is more environmentally friendly than methods that use chemical treatments (Poncsak *et al*. 2006; Kocaefe *et al*. 2008; Gunduz *et al*. 2009; Garcia *et al*. 2012). Heat treatment results in significant changes in the properties of wood, but it also causes undesirable reductions in the mechanical properties of the wood. Different species of trees are affected differently by heat treatment, so it is important to determine the optimal conditions (*e.g.* duration and temperature) for heat treatment to achieve the best balance of physical and mechanical properties. To do this, tests must be conducted to determine the resulting properties of wood that has been heat treated at different durations and temperatures.

As a result of heat treatment, the chemical composition of wood is altered; the hemicelluloses are most affected, and cellulose is somewhat resistant to chemical alterations (Esteves and Pereira 2009). Other changes that result include increased lignin content, increased dimensional stability due to cross-linking in lignin, the destruction of some of the hydroxyl groups, improved durability, decreased mechanical properties *e.g.*, static and dynamic bending strength and tensile strength, lower equilibrium moisture content, and darker color (Esteves and Pereira 2009).

Numerous studies have been conducted to determine the effects of heat treatment on physical properties of different tree species using a wide range of treatment conditions. As a result of the differences in species and the different treatment schedules, calculations of the changes in physical properties, such as mass loss, anti-swelling efficiency, and equilibrium moisture content, produced a wide range of values. Mass loss is a determinative factor of the results of heat treatment, *i.e.*, the greater the mass loss, the greater the effects on the physical and mechanical properties. Gunduz *et al*. (2009) reported that a significant relationship exists between mass loss and compression strength. Esteves *et al*. (2007) and Welzbacher *et al*. (2007) noted that there is a significant relationship between mass loss and equilibrium moisture content.

Brito *et al*. (2006) determined the density and shrinkage behavior of *E. grandis* wood, and the results showed that the thermal rectification process (only when a temperature of 200 °C was used) influenced wood shrinkage significantly. Brito *et al*. (2008) studied the changes in chemical composition that occurred when *Eucalyptus* and *Pinus* woods were heat treated at 120, 140,160, and 180°C, and the results showed that the arabinose, mannose, galactose, and xylose contents of the treated wood decreased significantly at 160 and 180°C. However, the glucose content remained the same, and the lignin content increased.

Calonego *et al*. (2011) determined the physical and mechanical properties of thermally-modified *E. grandis* wood, and the results showed decreases in mass, equilibrium moisture content, and volumetric swelling of 6.7%, 21.5%, and 23.2%, respectively, at a temperature of 180 °C and a duration of 2.5 h. Garcia *et al*. (2012) studied some properties of heat-treated *E. grandis* wood and determined that the decrease of mass and the decrease in equilibrium moisture content had different values. Almeida *et al*. (2009) studied heat treatment on micro-samples of three *Eucalyptus* species, and the results showed that the mass losses of *E. grandis*, *E. saligna*, and *E. citriodora*, when treated at 180 °C for 5 h, were between 2 and 3%. In addition, it was noted for these three *Eucalyptus* species, the values of their fiber saturation points decreased as the treatment temperature increased.

E. grandis is a preferred species for industrial plantations throughout the world due to its rapid growth. Juvenile and mature *E. grandis* woods have quite different properties. The mature wood of this species has a greater density than the juvenile wood. In addition, the mature wood has longer fibers and thicker cell walls than the juvenile wood (Malan 1995; Bao *et al*. 2001; Passialis and Kiriazakos 2004). Juvenile wood has less cellulose and more hemicelluloses and lignin than mature wood. There is a gradual increase in cellulosic content as the cells mature. Conversely, there is a gradual decrease in hemicellulosic content (Rowell *et al*. 2005). Due to these differences in chemical composition, the physical, mechanical, morphological, and chemical properties of juvenile wood and mature wood are different.

The focus of the present study was to determine the effects of heat treatment on the physical properties of juvenile wood and mature wood of *E. grandis* and assess the differences between these properties. Thus, the study included an assessment of the various physical properties of juvenile and mature wood, including mass loss, moisture content, volumetric swelling, and fiber saturation point.

MATERIALS AND METHODS

Three *E. grandis* trees (20-years-old; diameter at breast height: 40 cm and total height: about 42 m) were obtained from the Tarsus-Karabucak region in Turkey. Timbers were cut from the trees at a height from 2 to 8 m. Timbers were prepared by cutting the logs parallel to the direction of the grain. The dimensions of timbers were 35-40 x 8 x 200 cm (width x thickness x length). The timbers were stored and allowed to dry naturally for five months. After the drying period was completed, boards (2 x 2 x 100 cm-width x thickness x length) were cut from timbers, and successive samples were prepared for 10 treatment groups of juvenile wood and 10 treatment groups of mature wood from those boards. Adjacent samples were matched for homogeneity with one control and nine test groups. The remaining parts of timbers were stored for another study. The sizes of the samples were 2 x 2 x 3 cm (width x thickness x length). Juvenile wood samples were cut from parts near the pith, and mature wood samples were cut from parts near the bark. Three different temperature groups (120, 150, and 180 °C), three different duration groups (4, 6, and 8 h), and one control group were prepared. For each group, 20 samples of juvenile wood and 20 samples of mature wood were prepared from each log. Prior to the tests, all of the samples were conditioned in a test cabinet at a temperature of 20 ± 1 °C and a relative humidity of 65 ± 5% until they reached a 12% moisture content. Thereafter, the samples were dried at a temperature of 103 ± 2 °C in an oven until they reached 0% moisture content. Just after drying, the dimensions and weight of each of the samples were measured before the testing began, and heat treatment was performed in the same oven at atmospheric pressure and in the presence of air. Next, the samples were allowed to cool. After cooling, the dimensions and weight of each of the samples were measured again. The samples were stored at room conditions for one week, after which they were immersed in water for a period of four weeks. The samples were removed from the water, and their dimensions and weights were measured again.

The data were analyzed using one-way ANOVA and two-way ANOVA (P = 0.05) from the SPSS statistical software program, and significant differences were determined by the Tukey HSD (Honestly Significant Difference) multiple comparison test ($\alpha = 0.05$). In the present study, one-way ANOVA was used to determine the differences between all groups for each physical test. Two-way ANOVA was used to determine the effects of temperature and time factors.

Moisture content (MC) (after four weeks of immersion in water), oven-dried density (D_o), and volumetric swelling (VS) were determined according to Turkish standards TS 2471, TS 2472, and TS 4086, respectively. Mass loss (ML) and fiber saturation point (FSP) were determined by equations (1) and (2), respectively,

$$\text{ML} = \left(\frac{\boldsymbol{M_0} - \boldsymbol{M_1}}{\boldsymbol{M_0}}\right) \times 100(\%) \tag{1}$$

where ML is the mass loss, M_o is the mass of the sample after being dried in an oven at 103 ± 2 °C before heat treatment, and M_1 is the mass of the same sample after treatment.

$$\text{FSP} = \left(\frac{\text{VS}}{D_o}\right) \times 100 \ \ (\%) \tag{2}$$

In Eq. 2, FSP is the fiber saturation point, VS is the volumetric swelling, and D_o is the oven-dried density.

RESULTS AND DISCUSSION

Table 1 shows the results of the ML and the Tukey multiple comparison tests for juvenile and mature *E. grandis* wood after heat treatment. The ML values were found to differ between test groups, and the differences were significant according to one-way ANOVA (P < 0.01 for juvenile wood and P < 0.001 for mature wood).

Table 1. Physical Properties, One-Way ANOVA, and Tukey Test Results

Juvenile Wood											
		Do		ML(%)		VS (%)		MC (%)		FSP (%)	
Temp	Time	x	s	x	s	x	s	x	s	x	s
Control		554.5	67.6	-	-	13.4a	1.0	99.8a	14.2	24.4a	3.3
120	4	562.7	53.5	0.21 a	0.1	13.4a	1.0	92.3abc	6.6	24.0ab	2.4
	6	555.4	56.9	0.25 a	0.1	13.1a	0.7	91.8abc	6.1	23.9ab	2.9
	8	563.8	64.5	0.28 a	0.1	13.0a	1.0	92.6ab	8.0	23.6ab	3.3
150	4	545.3	52.9	0.57 b	0.2	13.0a	1.0	91.2abc	5.8	24.0ab	2.6
	6	560.1	62.1	0.76bc	0.1	12.7a	1.2	88.2bcd	6.5	22.8abc	2.5
	8	559.8	66.9	0.83 c	0.2	12.6a	1.0	88.6bcd	8.8	22.8abc	2.1
180	4	545.4	63.8	1.65 d	0.3	11.6b	0.9	86.4bcd	10.4	21.4bcd	2.2
	6	552.9	75.8	1.93 e	0.4	11.6b	0.9	83.0cd	14.7	21.2cd	2.8
	8	548.3	53.9	2.01 e	0.3	11.1b	0.8	81.0d	6.3	20.5d	2.2
ANOVA		F value		227.4		14.77		6.64		5.44	
		Sig.level		P<0.01		P<0.001		P<0.001		P<0.001	
Mature wood											
		Do		ML (%)		VS (%)		MC (%)		FSP(%)	
Temp	Time	x	s	x	s	x	s	x	s	x	s
Control		725.1	40.0	-	-	18.9a	3.0	81.2a	5.4	26.1a	3.6
120	4	717.0	44.7	0.39a	0.2	18.9a	3.0	80.3a	4.4	26.3a	3.3
	6	712.8	44.5	0.63ab	0.2	18.8a	3.0	80.0a	4.1	26.3a	3.3
	8	708.9	45.6	0.85 bc	0.3	18.7a	2.9	79.8a	4.5	26.4a	3.3
150	4	719.0	46.5	1.05 cd	0.2	18.6a	2.5	77.7a	3.8	25.8a	2.8
	6	708.6	35.2	1.20 de	0.3	18.3a	2.0	77.2a	4.3	26.0a	2.5
	8	701.8	36.7	1.36 e	0.3	18.0ab	1.5	76.2a	3.4	25.7a	1.9
180	4	714.0	31.0	1.83 f	0.3	17.8ab	2.3	66.8b	6.3	25.0ab	2.7
	6	709.6	33.6	2.00gf	0.4	17.0ab	2.3	66.0b	6.3	23.6ab	2.9
	8	698.9	42.7	2.24 g	0.3	15.6b	1.8	65.5b	7.2	22.4b	2.7
ANOVA		F value		103.8		3.55		31.88		4	
		Sig.level		P<0.001		P<0.001		P<0.001		P<0.001	

Means followed by the same letter are not significantly different

The ML values of mature wood were greater than those of juvenile wood for all treatment groups. The highest ML percentage was calculated for the samples that had been treated at 180 °C for 8 h. ML values ranged between 0.21% and 2.01% in juvenile wood and between 0.39% and 2.24% in mature wood. Similar results were reported for the testing of similar samples by Brito *et al*. (2006), Almeida *et al*. (2009), and Calonego *et al*. (2011). Esteves *et al*. (2007) reported similar results for *E. globulus* samples.

In addition, Table 1 clearly shows that the density of juvenile wood was lower than that of mature wood. The explanation for this is that the juvenile wood has more air gaps than mature wood. The coefficient of thermal conductivity for air is 0.02 W/mK and the corresponding value for air-dried wood is 0.10 W/mK (Örs and Keskin 2001). Simpson and TenWolde (1999) noted that thermal conductivity increases as density increases. Similar results were obtained and noted by Yapici *et al*. (2011). Yu *et al*. (2011) studied the thermal conductivity of some softwood and hardwood species, and stated that "…thermal conductivity of wood increases with density. This is obvious in that for a given volume, as density of wood increases, more fibril exists that is more conductive than air…". On the other hand, Suleiman *et al.* (1999) determined that voids, rays, and cell boundaries, in addition to density, affect thermal conduction. Mean oven-dried density values of all groups of juvenile wood and mature wood were 554 and 711 kg/cm^3, respectively. The calculated air gaps percentages of juvenile and mature wood were 64% and 54%, respectively. Therefore, it can be said that the low-density, juvenile wood is more resistant to heat than the high-density, mature wood. ML can be viewed as the determinative factor concerning the effects of heat treatment, because the greater it is, the greater the effects are. Obviously, ML increases as the temperature and time of heat treatment increase.

VS, MC, and FSP percentages and Tukey test results are given in Table 1. VS decreased from 13.4% to 11.1% in juvenile wood and 18.9% to 15.6% in mature wood. The differences between the groups were significant ($P < 0.001$), but only those samples treated at a temperature of 180 °C were different from the samples in the control group. Similar results were noted concerning the decrease of VS by Brito *et al*. (2006), who determined that the shrinkage of the wood was decreased significantly only at a temperature of 200 °C.

For the juvenile wood, the MC value of the control group was 99.8%. After the samples were heat treated at a temperature of 180 °C and duration of 8 h, the MC value decreased to 81.0%. For mature wood, the MC value of the control group was 81.2%. After the samples were treated at the same conditions specified above, the MC value decreased to 65.5%. In general, MC values were higher in juvenile wood than in mature wood. It can be said that MC values of mature wood were more affected in parallel with the ML percentage. In addition, Duncan test results showed that MC values of mature wood treated at a temperature of 180 °C were different from those of mature wood treated at other conditions. No differences were determined between the control group and the groups that were tested in the temperature range of 120 to 150 °C.

The FSP values of the control group were 24.4% and 26.1% in juvenile and mature wood, respectively. After the samples were heat treated at a temperature of 180 °C and duration of 8 h, the FSP values decreased to 20.5% and 22.4% in juvenile and mature wood, respectively. FSP percentages were higher in mature wood than juvenile wood. No differences were determined between the control groups and the groups tested in the temperature range of 120 to 150 °C.

Table 2 represents the percentage decreases of the VS, MC, and FSP. As indicated by the results obtained at various treatment conditions, VS, MC, and FSP were affected by heat treatment. Generally, VS, MC, and FSP of juvenile wood were affected to a greater extent than those of mature wood (except for one or two groups), and these values were affected to a greater extent at a temperature of 180 °C than any other temperature. But some percentage decreases were determined to be negative, and the reason was thought to be the lack of homogeneity among the wood samples.

Table 2. Percentage Decreases of VS, MC, and FSP

		Juvenile Wood				Mature Wood		
Temp. (^{o}C)	Time(h)	VS (%)	MC (%)	FSP (%)		VS (%)	MC (%)	FSP (%)
120	4	-0.32	7.49	1.71		0.18	1.12	-0.83
	6	1.97	7.95	2.11		0.67	1.39	-0.94
	8	2.76	7.22	3.54		1.05	1.62	-1.17
150	4	2.77	8.63	1.65		1.64	4.22	0.81
	6	5.06	11.61	6.52		3.42	4.85	1.04
	8	5.51	11.19	6.85		4.94	6.10	1.56
180	4	13.30	13.38	12.24		5.66	17.63	4.19
	6	13.45	16.82	13.07		11.27	18.71	9.26
	8	16.68	18.79	16.17		17.34	19.31	13.96

Table 3 shows the significance level (P) values of the physical properties of heat-treated *E. grandis* wood based on ANOVA results. As can be seen, the temperature of heat treatment had an effect on all of the physical properties. The time of heat treatment only affected the ML values of the juvenile and mature wood. The reason for this may be that the durations were not sufficiently different in the present study. But similarly, in some other studies, it has been reported that the impact of temperature is greater than the impact of time (Welzbacher *et al*. 2007). Yildiz *et al*. (2006) noted that heat treatment at lower temperatures for longer time does not result in corresponding changes in properties.

Table 3. Significance Levels of Two-way ANOVA Results of Physical Properties

	Source of Variance	P values			
		ML	VS	MC	FSP
Juvenile Wood	Temperature	0.000	0.000	0.000	0.000
	Time	0.000	0.800	0.253	0.183
	Temperature * Time	0.030	0.840	0.740	0.870
Mature Wood	Temperature	0.000	0.000	0.000	0.000
	Time	0.000	0.090	0.498	0.258
	Temperature * Time	0.784	0.400	0.987	0.303

Tukey multiple comparisons of the mean values of ML, VS, MC, and FSP associated with the various treatment conditions are given in Table 4. The results showed that temperature had a greater effect on VS, MC, and FSP values than did the duration of the heat treatment. The ML and MC values for sample groups treated at different temperatures were different for both juvenile and mature wood. VS and FSP values were different only for the groups of samples that were treated at 180 °C. The duration of the heat treatment affected only the ML of the mature wood.

Table 4. Tukey Multiple Comparisons of Treatment Means for ML, VS, MC, and FSP According to Temperature and Time Factors

		ML (%)				VS (%)			
	N	Temp.(°C)	means	Time(h)	means	Temp(°C)	means	Time (h)	means
Juvenile wood	20	Control	-	Control	-	Control	13.37a	Control	13.37a
	60	120	0.24a	4	0.81a	120	13.18a	4	12.69a
	60	150	0.72b	6	0.98b	150	12.83a	6	12.46a
	60	180	1.86c	8	1.04b	180	11.44b	8	12.30a
Mature wood	20	Control	-	Control	-	Control	18.92a	Control	18.92a
	60	120	0.62a	4	1.09a	120	18.80a	4	18.45ab
	60	150	1.20b	6	1.27b	150	18.29a	6	18.00ab
	60	180	2.02c	8	1.48c	180	16.76b	8	17.45b
		MC (%)				FSP (%)			
	N	Temp(°C)	means	Time (h)	means	Temp(°C)	means	Time (h)	means
Juvenile wood	20	Control	99.79a	Control	99.79a	Control	24.43a	Control	24.43a
	60	120	92.26b	4	89.99b	120	23.83a	4	23.16ab
	60	150	89.34b	6	87.69b	150	23.20a	6	22.66b
	60	180	83.50c	8	87.42b	180	21.05b	8	22.26b
Mature wood	20	Control	81.15a	Control	81.15a	Control	26.06a	Control	26.06a
	60	120	80.04a	4	74.94b	120	26.31a	4	25.70a
	60	150	77.05b	6	74.40b	150	25.76a	6	25.24a
	60	180	66.09c	8	73.84b	180	23.68b	8	24.81a

Figures 1A and 1B show the relationship between ML and VS of juvenile and mature *E. grandis* wood.

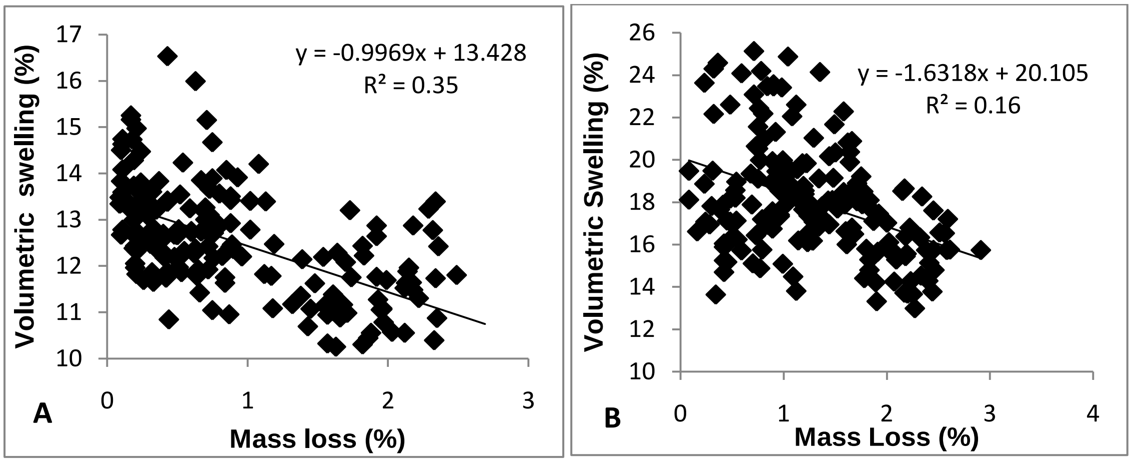

Fig. 1. Relationship between ML and VS of juvenile wood (A) and mature wood (B)

Regression analysis showed that the relationships were negative, and coefficients of determination were higher in juvenile wood ($R^2 = 0.35$) than mature wood ($R^2 = 0.16$). It has been reported that VS decreases when ML increases. The reason is that heat treatment changes the chemical properties of wood (Brito *et al*. 2008), and as a result of heat treatment, decomposition of hemicelluloses, ramification of lignin, and crystallization of cellulose occur (Kocaefe *et al*. 2008). Due to these changes, the wood does not swell as much as it would have without heat treatment (Welzbacher *et al*. 2007; Calonego *et al*. 2011).

Figures 2A and 2B represent the relationships between ML and FSP of juvenile and mature wood. The relationships were negative both in juvenile ($R^2 = 0.23$) and mature wood ($R^2 = 0.22$). When ML increases, FSP simultaneously decreases because there are fewer free hydroxyl (OH) groups. Similar results were obtained by Almeida *et al*. (2009) concerning FSP reduction in heat-treated *E. grandis* wood.

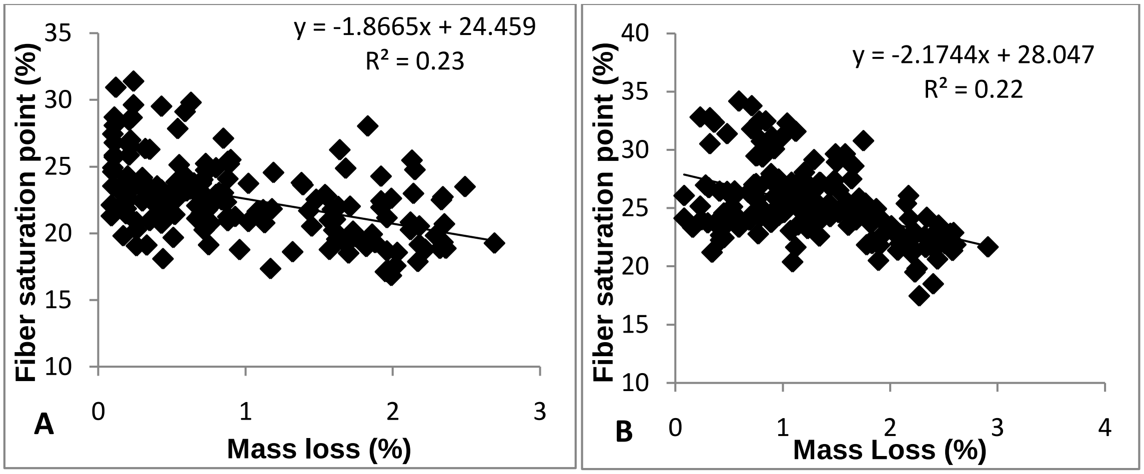

Fig. 2. Relationship between ML and FSP of juvenile wood (A) and mature wood (B)

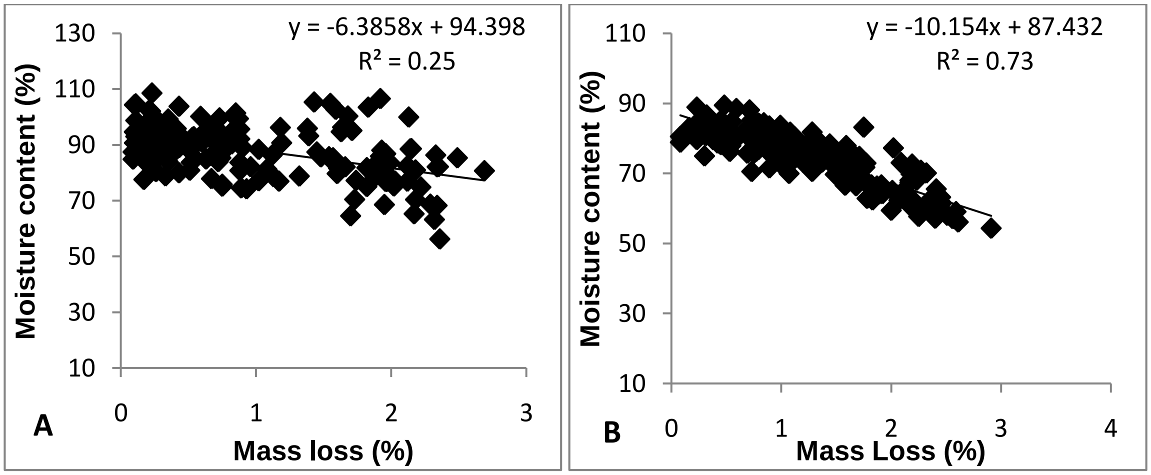

Fig. 3. Relationship between ML and MC of juvenile wood (A) and mature wood (B)

Figures 3A and 3B show the relationship between ML and MC of juvenile wood (A) and mature wood (B). The relationships were negative in both cases. The coefficients of determination were higher in mature wood ($R^2 = 0.73$) than in juvenile wood ($R^2 = 0.25$). This was tentatively attributed to the lower permeability of juvenile wood.

It can be seen in the graphs that the MC of the wood decreased as ML increased. There are two important reasons for this: 1) heat treatment reduces the FSP, and the amount of bound water in the cell wall decreases, and 2) heat treatment decreases the VS, and the amount of free water in the cell gaps decreases. As a result of these two reductions, the MC of the heat-treated wood decreases.

CONCLUSIONS

This study focused on the differences between heat-treated juvenile and mature wood of *E. grandis*. The results obtained in the study led to the following conclusions:

1. As a result of the severity of heat treatment, ML varies, and ML is higher in mature wood than in juvenile wood. It is thought that juvenile wood has more air gaps than mature wood and these air gaps serve as isolators, reducing the effect of temperature.
2. In contrast to the ML, in general, VS, MC, and FSP values of juvenile wood were more affected by heat treatment than those of mature wood. It can be said that this occurs because of the different chemical composition of juvenile wood.
3. The two-way ANOVA results indicated that the impact of the temperature of heat treatment was much more significant than the impact of the duration of heat treatment, a finding that was similar to that of similar previous studies.
4. Regression analysis showed that MC, VS, and FSP decrease as ML increases. The decrease in the MC of the samples was attributable to the decrease of FSP (due to the decrease in the number of OH groups) and the decrease of VS (due to decrease of cell gaps).

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