Effects of Heat Treatment on the Physical Properties of Heartwood and Sapwood of *Cedrus libani*

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Effects of heat treatment on the physical properties of heartwood and sapwood of *Cedrus libani* A. Richard, such as density, equilibrium moisture content, swelling, and fiber saturation point were investigated. Heartwood and sapwood samples were treated at 140, 160, 180, 200, and 220°C for 3 h. After heat treatment, the physical properties of the samples of wood were determined according to Turkish standards. The results showed that mass loss increased and physical properties decreased as the treatment temperature increased. As the treatment temperature was increased, the mass of the heartwood decreased more than that of the sapwood, which may be due to the fact that the heartwood had greater extractives content. Conversely, even though the mass of the heartwood decreased more than the mass of the sapwood at the treatment temperature of 220°C, its physical properties, such as equilibrium moisture content, swelling, and fiber saturation point, decreased less than those of the sapwood.

Keywords: Cedrus libani; Heat treatment; Heartwood; Sapwood; Physical properties

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INTRODUCTION

Wood has been an important raw material for many centuries, and it has been evaluated for use in numerous different applications. However, it has some undesirable properties that restrict its use for some applications, and work has been conducted in an effort to develop methods for improving these undesirable properties. Hill (2011) noted that these methods include chemical, thermal, and impregnation modifications, each of which has some advantages and disadvantages. Heat treatment is regarded as a more environmentally-friendly method than others that use chemical substances (Kocaefe *et al*. 2008; Gunduz *et al*. 2010; Garcia *et al*. 2012).

The heat treatment of wood results in physical and chemical changes, including increased lignin content, increased dimensional stability, improved durability, decreased mechanical properties, lower equilibrium moisture content, and darker color (Esteves and Pereira 2009). The results that are achieved with heat treatment are affected by several factors associated with the wood, such as its density and its species, as well as the temperature and duration of heat treatment. In addition, the thermal conductivity of the wood influences the results of the heat treatment. Several factors can affect the thermal conductivity of wood. As Simpson and Tenwolde (1999) stated, "the thermal conductivity of wood is affected by a number of basic factors: density, moisture content, extractive content, grain direction, structural irregularities such as checks and knots, fibril angle, and temperature."

The *Cedrus* genus has four species, *i.e*., *Cedrus libani* A. Richard, *Cedrus atlantica* Manetti, *Cedrus brevifolia* Hen., and *Cedrus deodara* Loud. (Aiello and Dosmann, 2007; Kurt *et al*., 2008). *Cedrus libani* A. Richard occurs naturally in Lebanon, Syria, and the Cilician Taurus mountains of southern Anatolia (Boydak 2003; Aiello and Dosmann 2007; Hajar *et al*. 2010). The wood of *C. libani* (Lebanon cedar or Taurus cedar), is quite durable biologically to fungi and insect attacks (Kurt *et al*. 2008). It was reported that the extractive content of *C. libani* heartwood is significantly greater than that of its sapwood (Usta and Kara 1997; Hafızoğlu and Usta 2005).

In some previous studies, the effects of heat treatment on the physical and chemical properties of juvenile and mature wood (Severo *et al*. 2012), on the wettability of heartwood and sapwood (Kortelainen and Viitanen, 2011), on the water absorption of heartwood and sapwood (Kortelainen *et al*. 2006), and on the dimensional stability of heartwood and sapwood (Cao and Huang 2012) were investigated. But, previous studies have provided inadequate information concerning the effects of heat treatment on the physical properties of thermally-treated heartwood and sapwood. Therefore, in this study, the effects of heat treatment on the physical properties of heat-treated *C. libani* heartwood and sapwood were studied.

MATERIALS AND METHODS

Three *Cedrus libani* trees were obtained from the Kahramanmaraş-Başkonuş region in Turkey. Logs that were 40 cm long were cut from the trees at a breast height of approximately 1.3 m. The sticks with widths, thicknesses, and lengths of 2.5, 2.5, and 40 cm, respectively, were prepared by cutting the logs parallel to the direction of the grain, as shown in Fig. 1. The sticks were stored and allowed to dry naturally for three months. After the drying period, samples with widths, thicknesses, and lengths of 2, 2, and 3 cm, respectively, were cut from sticks, and such samples were prepared for six different groups of heartwood and sapwood. To ensure the homogeneity of the samples among the six groups, two samples were collected from each stick. Heartwood samples were cut from parts near the pith, and sapwood samples were cut from parts near the bark. No samples were prepared from the transition sections. For each group, 30 samples of heartwood and 30 samples of sapwood were prepared from sticks. Then, the samples were conditioned at a temperature of 20 ± 1°C and a relative humidity of 65 ± 5% until they reached a moisture content of 12 ± 2%. Thereafter, the samples were dried at a temperature of 103 ± 2°C in an oven until they reached a moisture content of 0%. Just after drying, the dimensions and weight of each of the samples were measured. The heat treatment was performed in the same oven under atmospheric pressure and in the presence of air. After heat treatment, the samples were allowed to cool, after which the dimensions and weight of each of the samples were measured. Then, the samples were conditioned at a temperature of 20 ± 1°C and a relative humidity of 65 ± 5% until they reached a constant weight. The weight of samples was measured to determine their equilibrium moisture content, and then they were soaked in water for a period of three weeks. The samples were removed from the water and wiped with a clean cloth to remove water from the surface. Their dimensions and weights were then measured.

Some heat treatment studies that have been published in the literature indicated that the impact of temperature was more significant than the impact of treatment time

(Yildiz *et al*. 2006; Welzbacher *et al*. 2007; Bal and Bektaş 2012). Based on the earlier findings, it was decided to conduct the experiments at five different temperatures, *i.e.*, 140, 160, 180, 200, and 220°C, and a heat treatment time of 3 h was used in all cases.

Oven-dried density (D_o), equilibrium moisture content (EMC), volumetric swelling (VS), and fiber saturation point (FSP) were determined according to Turkish standards TS 2472, TS 2471, TS 4086, and TS 2471, respectively. The obtained data was analyzed using one-way ANOVA (P = 0.05) from the SPSS statistical software program, and significant differences were determined by the Tukey comparison test (α = 0.05).

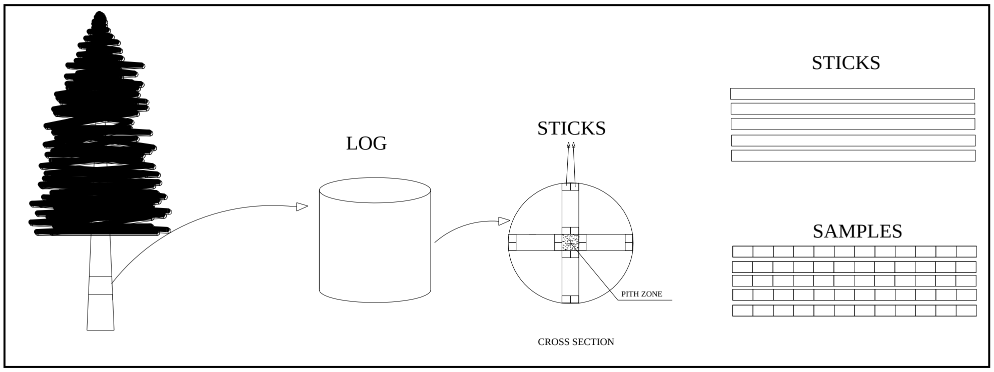

Fig. 1. Preparation of samples from sticks obtained from the logs

RESULTS AND DISCUSSION

The results of physical properties, one-way ANOVA, and Tukey test of heat treated *C. libani* heartwood and sapwood are given in Table 1. When the physical properties of the control groups of heartwood and sapwood are compared, it can be clearly seen that all of the physical properties of heartwood were lower than those of sapwood. The reason for this was that heartwood contains juvenile wood and sapwood contains mature wood in the *C. libani* wood that was tested. It was noted that density of the juvenile wood was lower than that of the mature wood (Simpson and TenWolde, 1999; Bao *et al*. 2001; Bal *et al.* 2011). The densities of the control samples of heartwood and sapwood were 468 and 512 kg/m^3, respectively. Depending on the density, the other physical properties of the control samples were also low in the heartwood. An analysis of the data presented in Table 1 shows that all of the physical properties of the test groups were affected significantly by heat treatment. The lowest physical properties of both heartwood and sapwood were found in the test groups that were treated at 220°C. It is well known that wood is affected to a great extent by heat treatment, especially at temperatures of 200°C and above.

Mass loss (ML) and percentage decrease of physical properties are shown in Table 2. It is inferred from Table 2 that ML values of heartwood at 140, 160, 180, 200, and 220°C were greater than those of sapwood. It is evident that the greater extractive content of heartwood affected the ML values. However, it is not evident why there should be such differences between percentage decreases of EMC, TS, RS, VS, and FSP of heartwood and sapwood after heat treatment at 140, 160, 180, and 200°C. In addition, at a heat treatment temperature of 220°C, the percentage decreases of EMC, TS, RS, VS, and FSP were greater for sapwood than for heartwood. The highest percentage decrease measured was the 79.7% decrease in the TS of the sapwood that was treated at 220°C.

Similar findings were obtained by Cao and Huang (2012), and they noted that the dimensional stability of steam-heat-treated Chinese fir wood was increased by 73% for heartwood and 71% for sapwood at 230°C for 5 hours. It is thought that thermal conductivity may be responsible for the differences between heartwood and sapwood. In previous studies, it was noted that the thermal conductivity of wood is affected by the species of the wood, density, moisture content, extractive content, grain direction, and treatment temperature (Simpson and TenWolde 1999; Şahinkol 2009; Yapıcı *et al*. 2011). In addition, Suleiman *et al.* (1999) noted that voids, rays, and cell boundaries affect thermal conduction. Yu *et al*. (2011) studied the thermal conductivity of some species and made the following statements: "The thermal conductivity of wood increases with density. This is obvious in that, for a given volume, as the density of wood increases; more fibril exists that is more conductive than air."

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

	Tem. °C	**D_o**		**EMC**		**TS**		**RS**		**VS**		**FSP**	
		x	**s**	**x**	**S**	**x**	**s**	**x**	**s**	**x**	**s**	**x**	**s**
Heartwood	Con.	**468.4**	18.4	**10.4b**	0.4	**6.22a**	0.8	**3.73a**	0.6	**9.95a**	1.1	**21.33a**	2.6
	140	**461.9**	17.9	**11.2a**	0.3	**6.12a**	0.6	**3.53ab**	0.4	**9.65a**	0.8	**20.42ab**	2.2
	160	**460.9**	16.4	**11.3a**	1.0	**5.99a**	0.8	**3.54ab**	0.4	**9.53a**	0.9	**19.96b**	2.0
	180	**454.9**	19.2	**10.0c**	0.3	**5.44b**	0.7	**3.30b**	0.5	**8.74b**	1.0	**18.40c**	2.0
	200	**441.8**	19.4	**8.3d**	0.4	**3.74c**	0.5	**2.47c**	0.3	**6.21c**	0.6	**13.13d**	1.4
	220	**418.8**	20.4	**6.7e**	0.3	**1.64d**	0.3	**1.32d**	0.5	**2.96d**	0.6	**6.18e**	1.1
One-way ANOVA		F value		353		240		127		303		271	
		P value		P<0.001		P<0.001		P<0.001		P<0.001		P<0.001	
Sapwood	Con.	**512.0**	48.8	**11.7c**	0.3	**8.68a**	0.4	**4.91a**	0.9	**13.59a**	1.1	**26.63a**	1.2
	140	**511.8**	51.0	**12.2a**	0.2	**8.51a**	0.6	**4.78a**	0.8	**13.29a**	1.1	**25.87ab**	1.4
	160	**510.3**	52.5	**11.9b**	0.2	**8.37a**	0.5	**4.59a**	0.8	**13.9ab**	1.1	**25.25b**	1.4
	180	**505.1**	54.5	**11.2d**	0.3	**7.75b**	0.7	**4.43a**	0.8	**12.17b**	1.4	**23.72c**	1.2
	200	**489.0**	51.2	**8.5e**	0.3	**5.31c**	0.5	**3.20b**	0.8	**8.51c**	1.1	**16.65d**	1.1
	220	**450.6**	49.5	**6.9f**	0.1	**1.77d**	0.5	**1.07c**	0.4	**2.84d**	0.7	**5.53e**	1.1
One-way ANOVA		F value		2248		787		114		427		1297	
		P value		P<0.001		P<0.001		P<0.001		P<0.001		P<0.001	

Means followed by the same letter are not significantly different, x: mean values, s: standard deviation, TS: tangential swelling, RS: radial swelling.

Many other researchers have stated that ML is the indicator value of the heat treatment, and as ML increases, physical properties decrease. But, in the present study, there was an important difference. The increase of ML of heartwood was greater than that of sapwood, but the percentage decrease of physical properties of heartwood treated at 220°C was less than that of sapwood. On this topic, Simpson and TenWolde (1999) stated that thermal conductivity of wood increases as density, moisture content, temperature, and extractive content of the wood increase. In the present study, the moisture content of the wood samples was 0%, which precluded any effect of moisture content on thermal conductivity, but other factors, such as density, treatment temperature, and extractive content could have had a significant effect on the thermal conductivity of the wood samples. In previous studies, the soluble part in alcohol-benzene of heartwood and sapwood was at 9.0% and 6.8% (Usta and Kara 1997), and at 7.6% and 3.1% (Hafızoğlu and Usta 2005), respectively. No significant differences were determined between heartwood and sapwood in percentage amount of cellulose and lignin (Hafızoğlu and Usta 2005), and cellulose, hemi-cellulose, and lignin (Usta and Kara 1997). According to those previous findings, the amount of extractives was significantly higher in heartwood than in sapwood compared to other wood components.

Bal and Bektaş (2012) studied the effects of heat treatment on the physical properties of juvenile wood and mature wood of *E. grandis* and stated that the low-density, juvenile wood is more resistant to heat than the high-density, mature wood. But *E. grandis* is a broadleaf tree species that does not contain as much extractive content as *C. libani*, which is a conifer that contains essential oils. That is why the ML of *E. grandis* was lower than that of *C. libani* when treated at the same temperatures (140 and 180°C). A similar study was conducted by Severo *et al*. (2012), and they determined that physical and chemical changes occur in both juvenile and mature woods of *Pinus elliottii* by thermal modification. The results showed that the influence of thermal modification in juvenile wood was lower than that in mature wood at 200°C and a period of 4 h. In the present study, similar results were obtained at 220°C and a treatment period of 3 h. Esteves and Pereira (2009) stated "most of the extractives disappear or degrade during the heat treatment, especially the most volatile, but new compounds that can be extracted from wood appear, resulting from the degradation of cell wall structural components." In addition, Gonzalez-Pena *et al*. (2004) studied the effect of extractives on heat-treated wood, but they did not determine that there was a significant relationship. Kortelainen and Viitanen (2011) studied wettability of sapwood and heartwood of heat treated Norway spruce and Scots pine, and they reported that changes in the wettability of wood was related to the degradation of the hemicelluloses and the migration of the extractives to the wood surface.

Table 2. ML and Percentage Decreases of EMC, TS, RS, VS, and FSP

	Temp. °C	ML (%)		EMC	TS	RS	VS	FSP
		x	s	%	%	%	%	%
Heartwood	Control	-	-	0.0	0.0	0.0	0.0	0.0
	140	**2.59**	0.4	-7.6	1.6	5.3	3.0	4.3
	160	**3.55**	0.6	-8.1	3.8	5.0	4.2	6.4
	180	**4.25**	0.6	4.5	12.6	11.6	12.2	13.7
	200	**6.81**	0.6	20.9	39.9	33.8	37.6	38.5
	220	**12.39**	0.9	35.6	73.6	64.6	70.2	71.0
Sapwood	Control	-	-	0.0	0.0	0.0	0.0	0.0
	140	**0.67**	0.1	-4.7	1.9	2.6	2.2	2.8
	160	**0.86**	0.1	-2.1	3.6	6.5	4.7	5.2
	180	**1.64**	0.2	4.3	10.8	9.8	10.4	10.9
	200	**4.23**	0.4	27.1	38.8	34.8	37.4	37.5
	220	**11.72**	0.8	41.0	79.7	78.1	79.1	79.2

In many earlier studies, it was determined that the relationship between oven-dried density and volumetric swelling was strongly positive ($R^2 \geq 0.50$) (Kord *et al*. 2010; Kiaei 2011). As density increases, volumetric swelling increases. Similar results were obtained in the present study, as shown in Fig. 2B. The relationship between density and volumetric swelling of the control samples of sapwood was positive, and the correlation was very strong ($R^2 = 0.75$). On the contrary, the respective relationship of heartwood was insignificant (R^2 = 0.03) (Fig. 2A). This difference can be explained in that the extractive content of the samples enhanced the density of samples but not their swelling. Similar findings were noted by Bal *et al*. (2011).

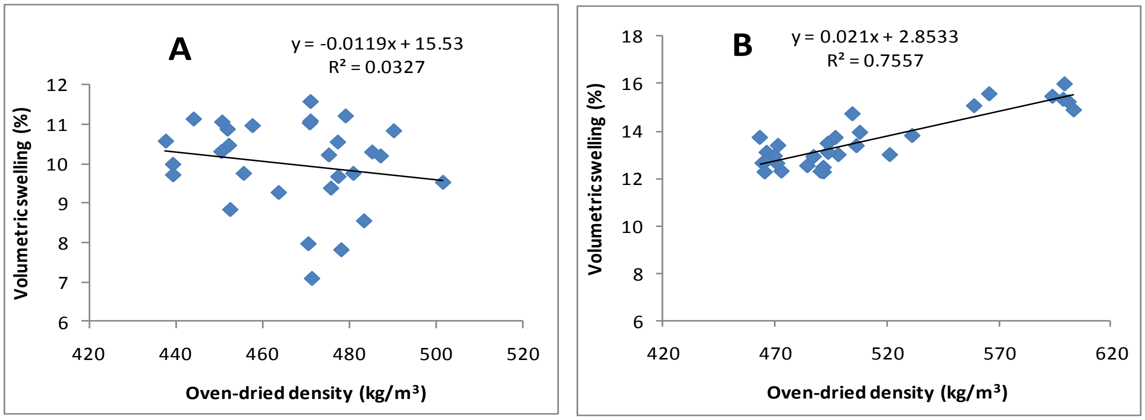

Fig. 2. Relationship between D_0 and VS at control samples (A: heartwood; B: sapwood)

As a result of heat treatment at 220°C, in contrast to the control samples, the relationships between oven-dried density and volumetric swelling in both heartwood (Fig. 3A) and sapwood (Fig. 3B) were positive, and R^2 of heartwood increased and R^2 of sapwood decreased compared to the control groups. It is surmised that the removal of the extractive content from the wood as a result of heat treatment caused the correlations to change.

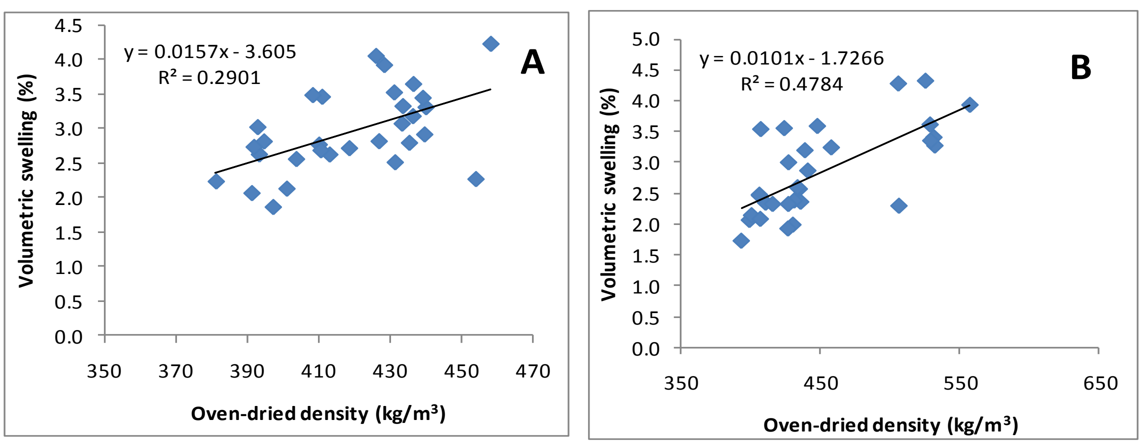

Fig. 3. Relationship between D_0 and VS at samples treated 220°C (A: heartwood; B: sapwood)

The relationship between ML and VS for the whole test samples is shown in Fig. 4. It can be seen that VS decreased as ML increased, and the correlation was very strongly negative (R^2 > 0.70). The coefficients of determination (R^2) of heartwood and sapwood were 0.87 and 0.91, respectively. Similar results were obtained in many other previous studies concerning the relationship between ML and VS (Esteves and Pereira 2009; Almeida *et al*. 2009; Bal and Bektaş 2012).

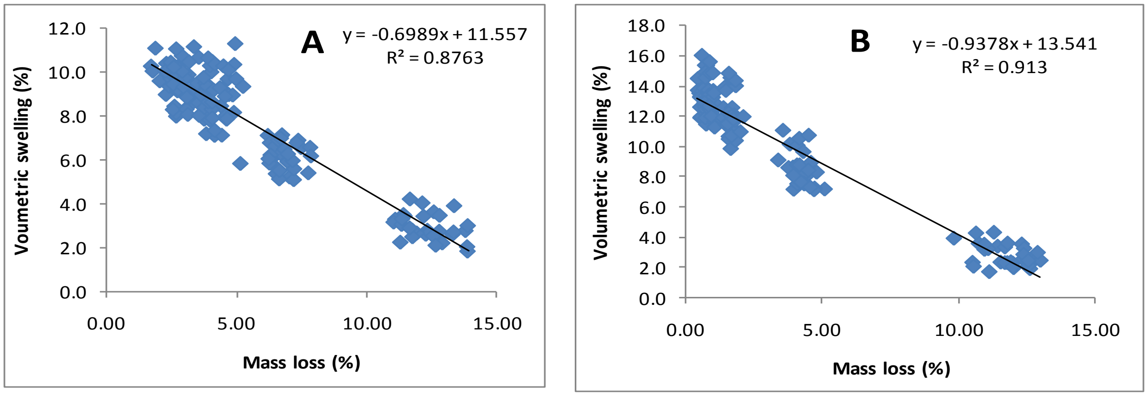

Fig. 4. Relationship between ML and VS at whole test samples (A: heartwood; B: sapwood)

CONCLUSIONS

In this study, the effects of heat treatment on some of the physical properties of *C. libani* wood were investigated. The results showed that:

1. At all treatment conditions, the mass loss (ML) of the heartwood was greater than that of sapwood. This was tentatively attributed to its greater extractive content.
2. ML is known as an indicator value of the thermal treatment of wood. Percentage decrease of physical properties increase as ML increases. Although the ML of the heartwood was greater than that of the sapwood, the percentage decrease of the physical properties of the heartwood was lower than that of the sapwood after treatment at 220°C. This difference was attributed to the higher density of the sapwood and the effect of the elevated treatment temperature on the thermal conductivity of the sapwood.
3. The differences between the physical properties of heartwood and sapwood were not clear at 140, 160, 180, and 200°C, but there were clear differences when the samples were treated at 220°C.
4. The relationship between D_o and VS for the control samples of heartwood and sapwood were quite different due to the effect of extractive content, but the relationship was similar together with decreased extractive content in the 220°C groups.

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