

Effects of heat treatment on some macroscopic and physical properties of Scots pine sapwood and heartwood

Isıl işlemin sarıçam diri odunu ve öz odununun makroskobik ve fiziksel özellikleri üzerindeki etkileri

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ABSTRACT

Impact of heat treatment (ThermoWood) on the macro structure and physical properties of Scots pine sapwood and heartwood were studied by visual examinations, using the following relevant standard test methods: ASTM D2244 and TS 2472, respectively. In the study, two processes-Thermo-S (190°C) and Thermo-D (212°C)-were employed for heat treatment. To compare the effect of different types of heat treatment, kiln dried wood samples were used for reference. Macroscopic investigation showed that superficial cracks occurred in all samples, and as the temperature increased, the severity and number of cracks increased. In the Thermo-D process, internal cracks and cupping were seen only in heartwood samples. Physical examination showed that as the temperature increased, color of the samples darkened, the density of the samples decreased, dimensional stability was enhanced. The Anti Swelling-Efficiency (ASE) in the Thermo-S and Thermo-D processes evaluated in sapwood samples was 17.04% and 24.77%, respectively; however, values in the heartwood samples were 11.97% and 30.45%, respectively. The highest reduction ratio of air dried density was 14.04% in the Thermo-D process applied to the heartwood samples. Thus, it can be concluded that this reduction due to the increased temperature is related to the formation of internal cracks.

Keywords: ThermoWood, macro structure, physical properties, scots pine, crack formation

ÖZ

Isıl işlemin (ThermoWood) sarıçam diri odunu ve öz odununun makro yapısı ve fiziksel özellikleri üzerindeki etkileri görsel olarak ve ilgili standartlar (ASTM D2244, TS 2472) aracılığıyla incelenmiştir. Thermo-S için 190°C ve Thermo-D için 212°C olmak üzere iki farklı işlem kullanılmıştır. Isıl işlemin etkilerini karşılaştırmak amacıyla fırın kurusu örnekleri referans alınmıştır. Makroskobik incelemeler sonucunda tüm örneklerde yüzeysel çatlakların olduğu ve sıcaklık arttıkça çatlakların sayısının ve şiddetinin arttığı görülmüştür. Sadece Thermo-D uygulanmış öz odun örneklerinde iç çatlak oluşumu ve oluklaşma tespit edilmiştir. Fiziksel incelemeler göstermektedir ki sıcaklık arttıkça örneklerin rengi koyulaşmakta, yoğunluk değerleri düşmekte ve boyutsal stabilizasyon iyileşmektedir. Anti Genişleme Etkisi sırasıyla Thermo-S ve Thermo-D diri odun örnekleri için %17,04 ve %24,77, öz odun örnekleri için %11,97 ve %30,45 olarak belirlenmiştir. Hava kurusu yoğunluk değerlerindeki en yüksek azalma oranı Thermo-D uygulanmış öz odun örneklerinde %14,04 olarak tespit edilmiştir. Sıcaklığın artması sonucu oluşan bu azalma eğiliminin çatlak oluşumu ile ilişkili olduğu düşünülmektedir.

Anahtar Kelimeler: ThermoWood, makro yapı, fiziksel özellikler, sarıçam, çatlak oluşumu

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INTRODUCTION

Heat treatment is a thermal modification method that enhances the dimensional stability and increases the durability of wood, yet lowers the strength properties thereof, and increases the tendency for it to crack, while also causing it to acquire a darker color. Different methods for thermal modification of wood have been developed, ThermoWood process being one of the modification methods used in commercial production (Hill, 2006; Metsa-Kortelainen, 2011). The ThermoWood process is mainly based on heating the wood for a few hours at high temperature (190°C and 212°C for softwoods) without pressure and under a protective water vapor environment (Anonymous, 2003).

When subjected to higher temperature, wood undergoes degradation in wood substrate and changes in chemical composition. As a result of this degradation, the physical and biological properties of wood change. These changes are induced by the treatment method, and vary according to the time, temperature and wood species. (Fengel and Wegener, 1989; Hill, 2006; Esteves et al., 2008). Changes related to wood species could be explained by different anatomical structures. As is well-known, the structural characteristics of wood vary among different tree species (Kollmann and Cote, 1968; Zobel and Buijtenen, 1989; Schweingruber, 2007). Previous studies revealed that there was a strong interaction between the effects of process conditions and anatomical properties of wood (Ward and Simpson, 1991; Anonymous, 2003; Suttie and Thompson, 2004; Boonstra et al., 2006; Dogu et al., 2015; Dogu et al., 2016). Even though several studies have investigated the effects of heat treatment on anatomical aspects, they are nonetheless quite restricted (Fengel and Wegener, 1989; Hietala et al., 2002; Andersson et al., 2004; Gosselink et al., 2004; Suttie and Thompson, 2004; Yildiz et al., 2004; Abe and Yamamoto, 2005; Sehlstedt-Persson et al., 2006; Boonstra, 2008; Dogu et al., 2010; Dubey, 2010; Awoyemi, 2011; Dogu et al., 2015; Dogu et al., 2016). Moreover, the differences between thermally modified sapwood and heartwood have been studied even less frequently (Boonstra, 2008; Metsa-Kortelainen, 2011; Metsa-Kortelainen and Viitanen, 2012; Esteves et al., 2013).

Chemical and physical properties of sapwood and heartwood are different (Hillis, 1987; Rowell, 2005). Therefore, it is expected that their reaction to heat treatment will also be different. The aim of this study was to investigate some macroscopic and physical differences between the sapwood and heartwood of thermally modified Scots pine wood. The effects of the heat treatment process on the macrostructure and physical properties of Scots pine wood were also examined.

MATERIALS AND METHODS

This study was performed on Scots pine wood samples obtained from the trees by controlled cutting from the Western Black Sea region. The trunks were sawn into pieces of timber having dimensions of 5x12.5x200 cm (Figure 1). The pieces of timber were divided into two parts, one part to serve as the control and the other to be used for the heat treatment sample. Kiln dried samples were employed as references for comparing the effect of heat treatment.

Heat treatment was carried out according to VTT (Technical Research Center of Finland)'s ThermoWood schedule with two different final temperatures (Thermo-S: 190°C, Thermo-D: 212°C) (Anonymous, 2003).

After the heat treatment, the pieces of timber were cut into 3-cm-high strips from internal and external parts to compare the effect of the treatment in the macro structure and the remaining parts were used to determine the physical properties (Figure 2). The changes generated in the macro structure as a result of the heat treatment were investigated using a SZX16 Stereomicroscope (Olympus, Tokyo, Japan) and a DP72 digital camera.

Color analysis, air-dried density, kiln-dried density, and ASE (Anti swelling efficiency) were performed to investigate the changes in the physical properties after the heat treatment. For the color analysis, the color measurements of the kiln dried samples were referenced and then the color differences were calculated by color measurements of the heat-treated samples using CR-200 Chroma Meter (Minolta, Osaka, Japan). The color measurements were performed according to the ASTM D 2244 standard test method using the CIELAB color system. The total color difference ΔE^*ab is calculated as follows:

$$\Delta E^*ab = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (1)$$

where: L^* , a^* , b^* are chromaticity scales;

ΔL^* : lightness-darkness, Δa^* : redness-greenness, Δb^* : yellowness- blueness.

The variation in color was calculated as the difference of L^* , a^* and b^* between treated and untreated wood in percentage of the initial value, as follows:

$$\Delta L^* (\%) = (L^* \text{ treated} - L^* \text{ untreated}) / L^* \text{ untreated} * 100 \quad (2)$$

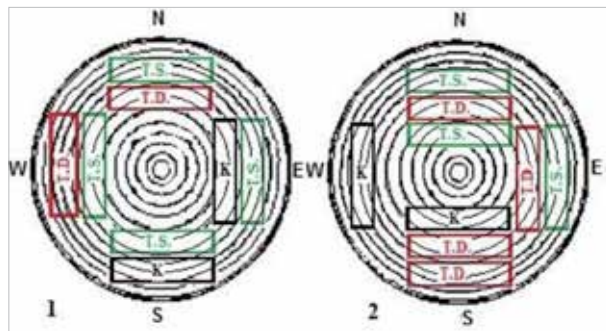


Figure 1. Cutting plan of trunks
 K: reference; T.S.: Thermo-S; T.D.: Thermo-D; W: west; E: East; S: south; N: north

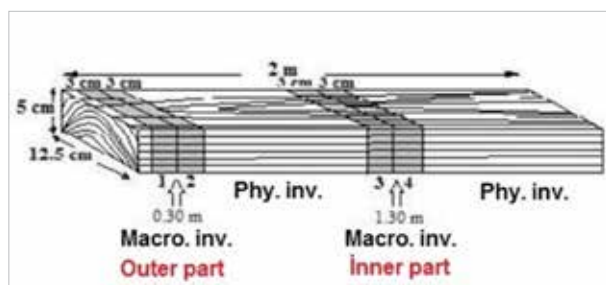


Figure 2. Cutting plan of timbers
 Macro. inv.: macroscopic investigation; Phy. inv.: physical investigation

In order to determine the air-dried and kiln-dried density values, samples having dimensions of 20 x 20 x 30 mm were prepared according to the TS 2472 (1976) Standard. The specimens were conditioned at 20°C and 65% relative humidity (RH) to 12% moisture content in order to determine the air-dried density. After that, the specimens were placed in a drying kiln at 103 °C until they reached a constant weight suitable for the determination of the kiln-dried density. The air-dried D_{12} and kiln-dried density D_0 were calculated as follows:

$$D_{0,12} = \frac{W_{0,12}}{V_{0,12}} \text{ (g/cm}^3\text{)} \quad (3)$$

where: D_0 : Kiln-dried density (g/cm³), W_0 : kiln-dried weight (g), V_0 : kiln-dried volume (cm³), D_{12} : air-dried density (g/cm³), W_{12} : air-dried weight (g), V_{12} : air-dried volume (cm³)

Dimensional stability of the heat-treated samples was calculated with ASE (Anti swelling efficiency). The kiln-dried samples were sized measured and then samples were immersed in water at a temperature of 20°C, until a constant weight was reached (fiber saturation point), and the sample sizes were then measured again. Volumetric swelling coefficients (S) were calculated as follows:

$$S \text{ (\%)} = \frac{(V_2 - V_1)}{V_1} \times 100 \quad (4)$$

where: V_2 : Water-saturated volume, V_1 : oven-dried volume

ASE gives the difference between swelling coefficients of treated and untreated samples and was calculated as follows:

$$ASE \text{ (\%)} = \frac{(S_c - S_t)}{S_c} \times 100 \quad (5)$$

where: S_c : The volumetric swelling coefficient of reference, S_t : the volumetric swelling coefficient of treated samples

The statistical evaluation of the data in the study was analyzed using a SPSS statistical package program having a 95% confidence level.

RESULTS AND DISCUSSION

This study revealed that crack formation was an important macroscopic change during heat treatment and the formation of cracks in all samples varied in the inner and outer parts of timber and inner/outer parts of timber were differently affected by the crack formation (Figures 3-5). Our research showed that the cracks which formed after drying in the outer parts of the timber did not extend to the inner parts. Therefore, it was determined that these cracks were superficial cracks which could be removed by various types of surface treatment (Figures 3, 4). The examinations made in the reference samples showed that the beginning of the crack formation was based on the technical drying, and the heat treatment applications led to both the formation of new cracks and the aggravation of existing cracks (Figure 4).

In previous studies on heat treatments, superficial cracks based on conventional drying have also been reported. Since these cracks could easily be removed by various types of surface treatment, they were not considered to be a serious defect (Johansson, 2005; Johansson, 2006; Sehlstedt- Person et al., 2006; Kallender and Landel, 2007; Boonstra, 2008; Altgen et al., 2012; Altgen et al., 2015). The wood material exposed to high temperatures shows different reactions in different directions due to its anisotropic nature. Thus many defects occurred because the stresses in the different directions of wood were nonequal. Thus, stress discrepancies in the different direction emerged as the cause of many defects (Ward and Simpson, 1991). Fengel and Wegener (1989) concluded that the shrinkage in the structure of heat-treated wood due to thermal degradation induced mass loss and volumetric shrinkage. They indicate that this may cause crack formation by creating stress on the cellular basis in the weakest areas of the cell. Heat treatment also caused changes in the chemical structure of wood, like the degradation of polymers due to the breakage of molecular bands. Furthermore, the degradation of polymers led to mass loss and volumetric shrinkage of wood (Fengel and Wegener, 1989; Boonstra, 2008). Many researchers have acknowledged that the basis of the changes in

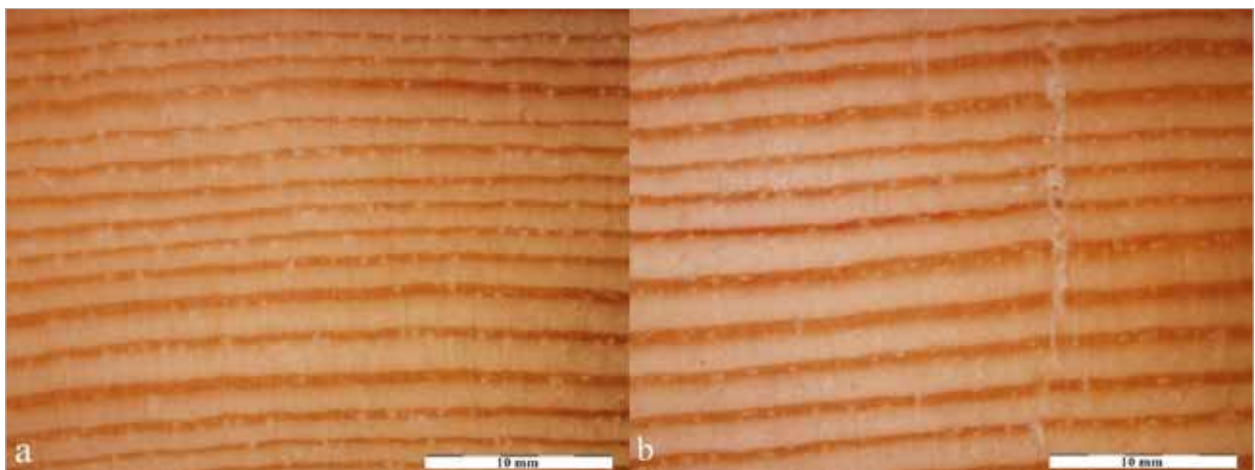


Figure 3. a, b. Kiln dried reference samples (a) inner part, (b) outer part

wood structure during heat treatment is related to the chemical and anatomical nature of the wood which is responsible for all the physical and mechanical changes (Fengel and Wegener, 1989; Ward and Simpson, 1991; Viitanen et al., 1994; Viitaniemi and Jamsa, 1996; Hietala et al., 2002, Anonymous, 2003; Suttie and Thompson, 2004; Johansson, 2005; Boonstra et al., 2006; Johansson, 2006; Kallander and Landel, 2007; Dogu et al., 2015; Dogu et al., 2016). It was thought that the formation of cracks was the result of changes in the chemical and anatomical structure of the wood during the heat treatment.

When the effect of heat treatment on timbers obtained from sapwood and heartwood was examined, only Thermo-D applied heartwood samples showed internal crack formation and cupping (Figures 5, 6). Similar to these findings, internal crack formation was detected in heat treated wood materials in previous studies (Johansson, 2005; Johansson, 2006; Kallander and Landel, 2007). Johansson (2006) also found that internal cracks were formed in boards thicker than 50 mm and since these cracks could not be identified from the outside, they should be considered more serious defects than surface cracks.

Similar to surface cracks, internal cracks are also associated with the changes in the chemical and anatomical properties of wood. The stresses generated in the internal and external part of the timber during drying are different from each other. Studies about the drying of wood (Ward and Simpson, 1991; Johansson, 2005) showed that when the stress level exceeds the recyclable elastic regime of wood, crack formation occurs. It is a known fact that in many wood species, heartwood has different chemical and physical properties from sapwood. In particular, the high content of extractives in heartwood has an effect on many properties, from physical properties to its durability (Kollmann and Cote, 1968). Some extractives can penetrate the secondary wall of cells affecting the shrinkage of wood during drying (Wangaard and Granados, 1967). These various facts about heartwood could reveal the differences between heartwood and sapwood samples undergoing the same process conditions (Thermo-D).

It has also been observed that heat treatment applications lead to the formation of resin flow, especially in the heartwood samples, and they appear like darker spots on the surface of the samples, which is in keeping with the literature (Sehlstedt-Persson et al., 2006; Boonstra, 2008; Esteves et al., 2008).

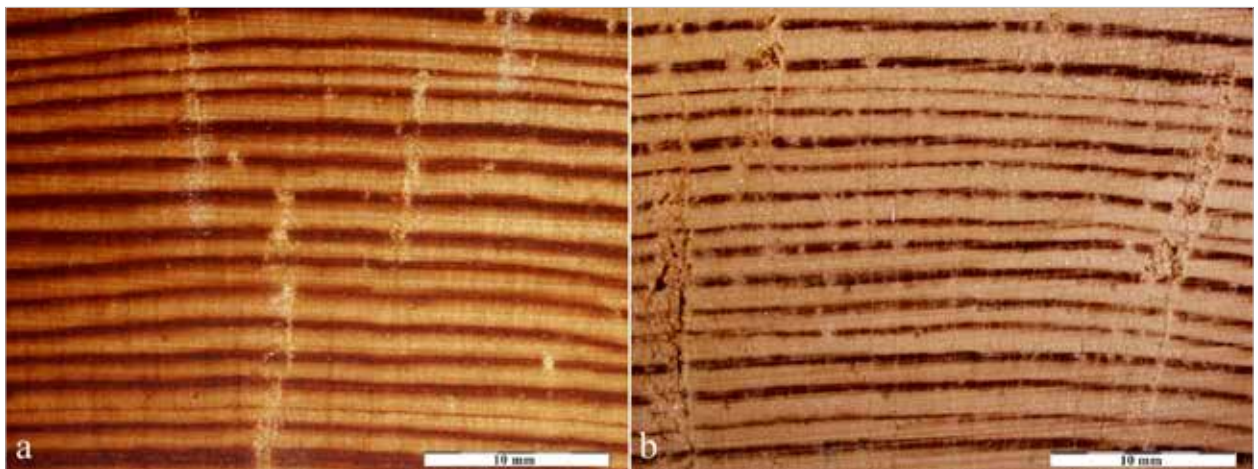


Figure 4. a, b. The outer part of sapwood samples (a) Thermo-S, (b) Thermo-D

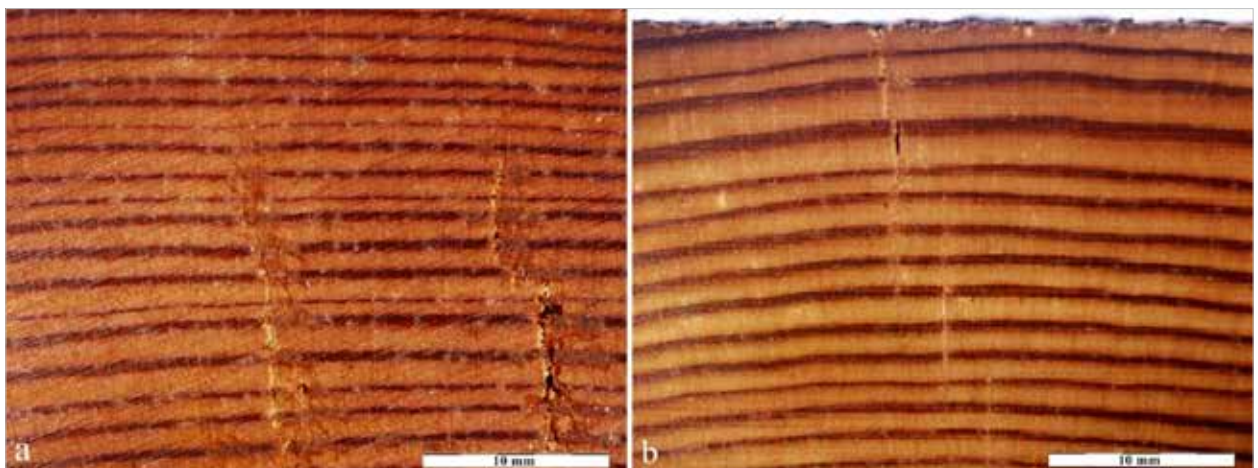


Figure 5. a, b. Thermo-D applied heartwood samples (a) inner part, (b) outer part

Macroscopic studies have shown that as the temperature increases (Thermo-S, 190°C; Thermo-D, 212°C), the darkening of the color of the specimens also increases. The most significant change after heat treatment was observed in the L* value which indicated that the color of the samples became darker, and similar results were found in previous studies (Militz, 2002; Bekhta and Niemz, 2003; Esteves et al., 2008; Gonzalez-Pena and Hale, 2009; Akgul and Korkut, 2012; Guller, 2012; Todorovic et al., 2012).

Color measurements of untreated and treated samples and their comparison with reference samples (%) are shown in Table 1. After heat treatment applications, the color of sapwood samples became redder (+Δa*) and yellow (+Δb*) while the color of heartwood samples became less red (-Δa*) and yellow (-Δb*). It is thought that the removal of volatile extractives such as outflow of resin could be the reason for the color changes in the heartwood samples. After the heat treatment, the color of the samples also became darker, and the changes in color were greater in the Thermo-D process than those in the Thermo-S process. The higher degree of darkening after heat treatment was observed in sapwood samples compared to heartwood

samples. The total color changes of samples were higher in the sapwood samples. Todorovic et al. (2012) also found that sapwood parts of beech wood had a higher mean value of total color difference than that of red heartwood. The changes in wood color after heat treatment are mainly associated with the degradation of chemical constituents and the removal or migration of extractives and other compounds (Sundqvist, 2004; Sehlstedt-Persson, 2008; Johansson, 2008; Dubey, 2010). The differences between the chemical components of sapwood and heartwood parts of Scots pine wood could explain the differences in color changes after treatment.

Density values of the treated and untreated samples (a) and their rate of changes (b) are shown in Figure 7. As the heat treatment temperature increased, the density values decreased and the highest decrease in density values was seen in the



Figure 6. a, b. Thermo-D applied heartwood samples (a) internal crack formation, (b) cupping

Table 1. Color changes

		C	TS	TD	C-TS (%)	C-TD (%)
Sapwood	L*	78.08	54.06	50.25	-24.02	-27.83
	a*	7.04	14.33	14.65	7.29	7.61
	b*	30.64	34.01	32.49	3.37	1.85
	ΔE				25.33	28.91
Heartwood	L*	56.82	57.01	46.53	0.19	-10.29
	a*	16.99	14.46	13.42	-2.53	-3.57
	b*	33.52	32.1	26.67	-1.42	-6.85
	ΔE				2.91	12.87

C: reference; TS: Thermo-S; TD: Thermo-D

Table 2. Anti-Swelling Efficiency (ASE)

	Sapwood		Heartwood		Total	
	TS	TD	TS	TD	TS	TD
ASE (%)	17.04	24.77	11.97	30.45	14.88	24.86

TS: Thermo-S; TD: Thermo-D

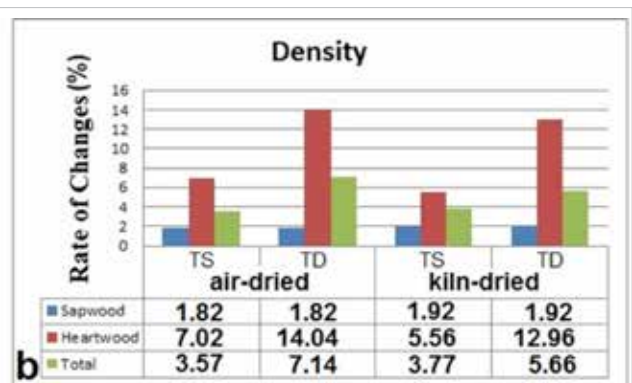
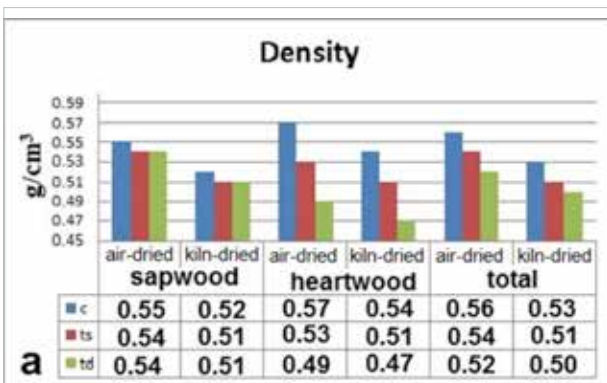


Figure 7. a, b. (a) Density values of the samples, (b) Rate of changes in densities according to reference samples
 C: reference; TS: Thermo-S; TD: Thermo-D

Thermo-D applied heartwood samples. Air-dried density values were higher than kiln-dried density in all samples. A higher rate of change in density values after the heat treatment was seen in the heartwood samples. Previously conducted studies have confirmed that the density decreases after the application of heat treatment for various wood species (Boonstra, 2008; Guller, 2012; Percin et al., 2016). The degradation of wood components (mainly the hemicelluloses) into volatile compounds and the evaporation of extractives are considered as the main parameters responsible for the density reduction of wood after heat treatment (Fengel and Wegener, 1989; Boonstra, 2008; Esteves and Pereira, 2009). According to Sehlstedt-Persson (2008), extracted pine heartwood acts in the same way as pine sapwood in moisture diffusion experiments which indicate the extractive content in softwood has a great influence on drying. Moisture diffusivity is one of the main factors that affect the success of drying applications. However, Sehlstedt-Persson (2008) has found that density has greater influence than extractive content on diffusivity. It is a well-known fact that the main difference between heartwood and sapwood is extractive content which is considerably higher in heartwood (Fengel and Wegener, 1989). In light of this information higher density reduction in heartwood samples could be explained with their higher extractive content. It could be concluded that higher density reduction could be the reason for internal crack formation in heartwood samples.

Anti-Swelling Efficiency (ASE) of heat-treated Scots pine wood is shown below in Table 2. The heat treatment improved the dimensional stability of the Scots pine wood in direct proportion to process temperature. The most significant improvement in dimensional stability was seen in the Thermo-D applied heartwood samples (%30.45). Similar to this study, Tjeerdsma et al. (1998) reported that the heat treatment allowed the reduction of swelling for Scots pine from 22% to 40%, respectively. Yıldız (2002) reported that beech wood's ASE increased with the increase of the temperature and time of treatment, reaching 50% at 200°C. Militz (2002) showed that the improvement of dimensional stability depends on the species. Under the same conditions, radial and tangential ASE values were 10% and 13% for beech wood, 13% and 23% for Douglas fir, 11% and 40% for spruce, 35% and 40%, for Radiata pine, and 33% and 41% for Scots pine, respectively. Esteves et al. (2013) reported that after heat treatment *Pinus pinaster* radial ASE reached 52% for sapwood and 50% for heartwood, while tangential ASE reached 50% and 40%, respectively. Several studies have shown that, generally, heat-treated wood loses hygroscopicity leading to an increase in dimensional stability with low shrinkage and swelling values.

The degradation of the hemicelluloses, removal of the volatile extractives, breaking of hydroxyl groups of amorphous cellulose, plasticization of lignin and the reorganization of the lignocellulosic polymeric components of wood were proposed as explanations for the increased dimensional stability of heat treated wood (Bekhta and Niemz, 2003; Weiland and Guyonnet, 2003; Esteves and Pereira, 2009; Korkut and Kocaefe, 2009). These chemical changes may have also some effect on other

properties. In this study, these properties were density reduction, color changes and crack formations. Alterations in the chemical structure cause mass losses and breakage of bonding sites for waters, leading to a decrease in density, shrinkage and swelling. While these changes occur in the molecular bonds in the wood structure, stresses emerged in the anatomical structure on the cellular basis. These stresses induced the formation of cracks when they exceeded the recyclable regime. When wood is subjected to heat treatment, the color of the samples darkened, depending on the chemical changes. Several studies found that these color changes also appeared to correlate with the wood density. Density of wood is one of the most important characteristics of heat treatment and is commonly referred to as an indication of quality. Although it was not studied in this work, many researchers have confirmed that mechanical properties are also induced by these physical changes. Each effect created has interrelated reactions on the material. In this study, our main research emphasis has been the effects of heat treatment on certain properties of Scots pine wood. This study revealed that heat treatment affects sapwood and heartwood to different degrees. It was thought that these differences were induced by their different chemical structures.

CONCLUSION

Based on conventional drying, superficial cracks occurred in all heat-treated samples. As the temperature increased the heat treatment led to both the formation of new cracks and the aggravation of existing cracks. Since these defects could easily be removed by surface treatments, they were not considered to be serious defects. However internal crack formation and cupping were determined in the Thermo-D applied heartwood samples. Since internal cracks cannot be detected from outside the material, they have to be considered as a significant defect that reduces the quality of the material.

The color of heat-treated samples was darkened. A higher degree of darkening was observed in the sapwood samples. Limited content of volatile extractives in sapwood leads to less removal of extractives from wood. This slight outflow ends up with higher color change. Therefore, if the darker color is asked for, Thermo-D sapwood samples would be preferable.

With increased temperature, the density of the samples was reduced. Higher reductions were found in air-dried heartwood samples (for the Thermo-S 7.02%, for the Thermo-D 14.04%). Density is the main physical property of wood which affects most of the strength properties. Thus, if the strength properties are required in the usage area, utilization of Thermo-D heartwood samples should be avoided.

The heat treatment improved the dimensional stability of the Scots pine wood, increasing with temperature of treatment. The most significant improvement in dimensional stability was seen in the Thermo-D applied heartwood samples (ASE %30.45). Thus, if there is a need for high stability on the place of use, utilization of Thermo-D heartwood samples would be more suitable.

The highest reduction in density and improvement in dimensional stability was achieved in the Thermo-D applied heartwood samples. These findings indicate the extractives are very effective on the quality of heat treatment.

Ethics Committee Approval: There is no need for ethics committee approval for this subject. But the legal permission for taking the samples from forest was obtained from General Directorate of Forestry.

Peer-review: Externally peer-reviewed.

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