

Pine Wood Modification *via* an Antifungal Oil – Heat Treatment and its Effect on Annual Wood Rings

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Thyme oil, which is an anti-fungicide, was used to increase the physical and mechanical properties of wood as well as improve its strength, especially in outdoor conditions. For this purpose, Stone pine (*Pinus pinea* L.) wood samples classified according to the annual number of rings were exposed to either the impregnation process or the combined process, which was comprised of impregnation followed by heat treatment. As a result of the study, it was determined that the wood had different physical and mechanical properties based upon the number of annual rings. In addition, it was specified that the thyme oil used in the impregnation process improved the physical properties of the wood and also reduced the water absorption during the combined process. As a general conclusion, the impregnation process and the combined process increased the mechanical properties of the wood in parallel with an increase in the number of annual rings. The process of impregnation of wood materials with thyme oil is promising due to its anti-fungal and antibacterial properties, its ability to be used in small amounts in on-site impregnation, and it being an environmentally friendly product for the wood protection industry.

Keywords: *Pinus pinea*; Thyme oil; Heat treatment; Mechanical properties

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INTRODUCTION

The field of wood material preservation is moving towards a new approach by avoiding toxic chemicals, developing new technologies, and avoiding producing negative effects on the environment. At the beginning of this approach, heat treatment of wood at high temperatures is an effective method of improving the properties of wood such as dimensional stability and durability (Esteves and Pereira 2009; Navi and Sandberg 2012). In addition, not using any chemicals in the heat treatment method makes it environmentally friendly (Garcia *et al.* 2012). In addition, interest in the impregnation and process development of wood materials with natural plant extracts or plant oils with anti-fungal properties has increased (Thevenon 2001; Tomak and Yıldız 2012).

In recent years, many studies have been performed to increase the useful life of wood without using any chemicals (Metsä-Kortelainen *et al.* 2006; Dubey *et al.* 2011; Korkut 2012; Gaff and Gasparik 2013; Sozbir and Bektaş 2017). In terms of impregnation processes with oils, studies have been carried out to increase the protective properties of wood *via* the addition of fungicides and thermicides to the oils (Palanti and Susco 2004; Lyon *et al.* 2007; Lyon *et al.* 2009). Thyme oil has been found to be effective against some white rot fungi and brown rot fungi (Voda *et al.* 2003; Reinprecht *et al.* 2019). In general,

it has been reported that a treatment of 10% of thyme oil provides resistance to some fungal deterioration (Yang and Clausen 2008).

In this study, the effects of impregnation and heat treatment processes on the physical and mechanical properties of pine wood samples, which were subjected to heat treatment after being impregnated *via* dipping the wood in thyme oil (an antifungal agent), were investigated. With this study, it is important that the combined process increases some of the physical and mechanical properties of the wood material and also increases the biological resistance due to the anti-fungicide property of the oil content. Easy applicability of the process before heat treatment and the ability to use thyme oil alone are important in terms of on-site impregnation processes.

EXPERIMENTAL

Materials

There are approximately 162,000 hectares of stone pine in the areal distribution of tree species in Turkey forests and it is important to expand the usage areas of wood (TOD, 2019). Stone pine (*Pinus pinea* L.) trees were obtained from the town of Kahramanmaraş-Onsen located in East Mediterranean region of Turkey. Then, the test trees were sized at an engineering workshop based on the dimensions of the test specimens required by the relevant standards mentioned below.

After the sizing process, the samples were coded and grouped. The grouping process was made with consideration to the number of annual rings. Three different groups were formed: an annual rings number less of than 10, between 10 and 25, and greater than 25.

In the study, thyme oil produced by 100% water vapor distillation was used and supplied from Mecitefendi company in Turkey.

Methods

The samples grouped based on the number of annual rings were homogeneously separated for the control, impregnation, and heat treatment processes. First, the specimens were immersed in thyme oil for 5 min and left under open air conditions for 3 weeks to condition. After the conditioning process, the impregnated samples were subjected to heat treatment in a laboratory drying oven at a temperature of 150 °C for 1 h. Because, at this temperature and duration, it has been determined as the optimum process where the oil leakage problem is the least and the temperature is the highest. The test specimens sample codes and applied processes are shown in the Table 1.

Test specimens were prepared on the basis of TS standard 2470 (1976). The densities were determined with samples with dimensions of 20 mm × 20 mm × 30 mm. Additionally, the static bending strength and modulus of elasticity were determined with sample dimensions of 20 mm × 20 mm × 300 mm based on TS standard 2474 (1976) and TS standard 2478 (1976), respectively. The compression strength parallel to the grain (sample dimensions of 20 mm × 20 mm × 30 mm) and Janka hardness (sample dimensions of 50 mm × 50 mm × 50 mm) were determined according to TS standard 2595 (1977) and TS standard 3459 (1980), respectively. For the physical properties, the volumetric shrinkage and swelling were calculated according to TS standard 4083 (1983), TS standard 4084 (1983), TS standard 4085 (1983), and TS standard 4086 (1983) with sample dimensions of 20 mm × 20 mm × 30 mm.

Table 1. Modification Process Parameters and Codes

Sample Code	Annual Ring Number (N)	Impregnation Time (min)	Heat Treatment (°C)
A-CS	N < 10	-	-
A-IP	N < 10	5	-
A-HT	N < 10	5	150
B-CS	10 < N < 25	-	-
B-IP	10 < N < 25	5	-
B-HT	10 < N < 25	5	150
C-CS	25 > N	-	-
C-IP	25 > N	5	-
C-HT	25 > N	5	150

Note: CS: control sample; IP: impregnation process, HT: heat treatment; A: less than 10 annual rings; B: between 10 and 25 annual rings; and C: greater than 25 annual rings

The results were analyzed using one-way ANOVA with the SPSS statistical software program and Duncan's mean separation test to populate the homogeneity groups that showed significant differences at the 95% confidence level.

Characterization of the Wood

The cell morphologies of the samples were characterized *via* scanning electron microscopy (SEM) (ZEISS-EVO LS10) at an accelerating voltage of 10 kV. The wood samples were cut into thin slices with a Leica RM2255 microtome, then coated with gold before scanning. The structures of samples were characterized using a Fourier transform infrared spectrometer (Perkin Elmer Spectrum 400, Waltham, MA, USA). Wood samples were ground into powder, mixed with potassium bromide, and made into a tablet before testing.

RESULT AND DISCUSSION

The changes in the physical properties of the wood samples with different annual ring numbers, after modification, are shown in the Table 2. It was found that as the annual number of rings in the wood sample increased its density decreased. In this study, as the annual number of rings decreased, it exhibited young wood features. Larson *et al.* (2001) found that the amount of extractive substances in young wood is greater than in mature wood. The resin contained in wood is known to increase the density of the wood (Cown 2001). With regards to this information, this study explains that the density of *Pinus pinea* wood decreases as the number of annual rings increases. Moreover, Bektas *et al.* (2003) determined that Calabrian pine wood, which has resin amounts relative to its number of annual of rings of wood, decreases in density as the annual ring number increases.

From the analysis shown in Table 2, it can be understood that the density value of wood samples impregnated *via* the dipping method increased. Due to the fact that oil absorption increases the weight of the wood, it also increases the density (Lee *et al.* 2018). However, the heat treatment applied afterwards has been found to reduce the density. Sözbir and Bektaş (2017) stated that a heat treatment modification reduced the density of wood. In addition, it was found that the weight per unit volume of wood decreases as the number of annual rings increases. As the wood matures, *i.e.*, as the number of annual rings increases, the amount of cellulose increases; accordingly, the hydrophilic cellulose

increases the volume of the material by taking more water (Şahin 2008; Arslan and Aydemir 2009). This situation causes a decrease in the weight per unit volume.

Table 2. Comparison of the Physical Properties of *Pinus pinea* Wood With Relation to the Number of Annual Rings

Sample Code	Oven Dry Density (g/cm ³)	Weight per unit Volume (g/cm ³)	Volumetric Shrinkage (%)	Volumetric Swelling (%)	Fiber Saturation Point (%)
A-CS	0.58 ± 0.07a(*)	0.52 ± 0.05a	10.89 ± 1.22bc	11.41 ± 1.37bc	20.48 ± 1.95a
A-IP	0.59 ± 0.05a	0.54 ± 0.04a	8.31 ± 1.35a	8.62 ± 1.45a	15.40 ± 2.45a
A-HT	0.57 ± 0.04a	0.53 ± 0.04a	10.79 ± 0.77bc	11.38 ± 0.86bc	19.46 ± 2.35a
B-CS	0.50 ± 0.02b	0.45 ± 0.02b	11.35 ± 1.81bc	12.15 ± 1.97bc	25.69 ± 5.33b
B-IP	0.52 ± 0.02b	0.47 ± 0.02b	8.74 ± 1.87a	9.17 ± 1.85a	18.61 ± 4.48a
B-HT	0.49 ± 0.02b	0.45 ± 0.02b	11.52 ± 0.50bc	11.93 ± 0.52bc	25.58 ± 1.75b
C-CS	0.45 ± 0.01c	0.39 ± 0.01c	12.55 ± 1.29c	13.31 ± 1.27c	31.91 ± 3.31c
C-IP	0.46 ± 0.02c	0.40 ± 0.02c	10.28 ± 1.14ab	10.76 ± 0.12ab	25.75 ± 1.22b
C-HT	0.44 ± 0.02c	0.40 ± 0.01c	11.95 ± 3.85bc	12.78 ± 4.30bc	30.26 ± 9.66bc
Sign. (p)	0.001	0.001	0.001	0.001	0.001

Note: (*) means with the same letter are not significantly different in Duncan's mean separation test

Compared to the control samples, the lowest volumetric shrinkage value was obtained from the impregnated groups. Alongside the impregnated groups, the heat treatment application also decreased the volumetric shrinkage amount. When evaluating the control samples in terms of the annual ring number, a similar tendency has been observed, *i.e.*, the lowest volumetric shrinkage was observed in the impregnated samples and then in the heat-treated samples.

As the number of annual rings in the wood samples increased, the fiber saturation point (FSP) increased, but it was found that impregnation decreased the FSP in all annual ring number groups. Impregnation with oil causes oil accumulation in the cell wall and the formation of a protective layer on the wood, which provides dimensional stability (Jalaludin *et al.* 2010). In different studies, the treatment of wood with oil has been evaluated and concluded that oil prevent between the wood and water relationship (Jämsä and Viitaniemi 2001; He *et al.* 2019).

Table 3 shows the changes in weight gain, mass loss, and water absorption according to the modification processes applied. After impregnation, the highest weight gain among the air-dried samples was obtained in group C and later in group B. The highest weight loss out in groups A-HT, B-HT, and C-HT, which were subjected to heat treatment after the impregnation process, was determined to be group C (3.11%). It was determined that as the density of the wood decreases, the amount of oil it absorbs increases.

The resin amount of *Pinus pinea* wood is quite high (De Angelis *et al.* 2018). The amount of resin in young wood is higher than the amount of resin in mature wood (Arslan and Aydemir 2009). During the oil impregnation process, it is thought that the group with fewer annual rings absorbs less oil as a result of the excess resin in the cells.

Table 3. Changes in the Weight Gain, Mass Loss, and Water Absorption Values According to Number of Annual Rings

Sample Code	Air Weight Gain (%)	Oven Dry Weight Gain (%)	Mass Loss (%)	Water Absorption (%)		
				1 h	6 h	360 h
A-CS	–	–	–	5.97 (2.01)	11.93 (4.94)	53.02(11.89)
A-IP	10.7 (1.7)(*)	1.85 (0.5)	–	5.74 (1.46)	11.31 (3.43)	52.90 (6.799)
A-HT	9.33 (1.53)	1.84 (0.53)	1.82 (0.71)	4.45 (1.14)	10.70 (2.56)	51.70 (9.51)
B-CS	–	–	–	12.48 (5.60)	20.62 (7.34)	73.56 (6.57)
B-IP	14.32 (2.53)	3.96 (1.3)	–	11.45 (4.60)	19.21 (5.87)	72.92 (4.36)
B-HT	12.84 (2.4)	3.52 (0.91)	2.77 (0.63)	9.05 (4.48)	17.19 (6.02)	70.85 (5.82)
C-CS	–	–	–	34.87 (4.55)	47.94 (5.99)	97.66 (5.48)
C-IP	15.33 (1.59)	4.95 (0.35)	–	25.63 (6.47)	40.84 (6.56)	97.51 (8.20)
C-HT	16.82 (2.61)	4.23 (0.63)	3.11 (0.47)	28.01 (3.56)	37.60 (4.51)	95.00 (5.82)

Note: (*) the values in parentheses indicate standard deviation

The extractive substances show hydrophobic properties, and since the A-CS, A-IP, and A-HT groups contain more extractive substances, there is less free and bound water (Singh *et al.* 2011). This situation caused less mass loss during the heat treatment, as it made heat transfer more difficult. In addition, during the heat treatment, as there is a high rate of hemicellulose in the mature wood, the total mass loss is greater (Rowel 2005). Hemicellulose is the first wood component to be affected by heat treatment and causes an increase in mass loss (Esteves and Pereira 2009). Likewise, the water intake value decreases as the annual number of rings decreases. The least amount of water absorption was realized in impregnated wood treated with heat in A-HT group, which had the least number of annual rings. In this study, the most effective method to reduce water absorption is the impregnated heat treatment method (HT process).

The effects of the modification process on the mechanical properties according to the number of rings per year are shown in Table 4.

Table 4. Effect of the Modification Process on the Mechanical Properties According to Number of Annual Rings

Sample Code	Compression Strength (N/mm ²)	Bending Strength (N/mm ²)	Modulus of Elasticity (N/mm ²)	Janka Hardness (N/mm ²)
A-CS	46.87 ± 3.72a	69.92 ± 13.22a	5708 ± 1427ab	50.16 ± 10.22a
A-IP	48.76 ± 4.63cd	73.10 ± 12.26ab	6081 ± 876ab	50.80 ± 8.69a
A-HT	52.44 ± 4.05d	77.68 ± 8.74ab	5509 ± 1477a	51.24 ± 6.31a
B-CS	42.13 ± 2.59ab	83.96 ± 9.76ab	6334 ± 605ab	44.41 ± 11.08a
B-IP	45.73 ± 2.85bc	84.64 ± 9.25ab	6434 ± 589ab	46.47 ± 10.79a
B-HT	46.80 ± 2.29c	89.92 ± 19.13b	6195 ± 959ab	47.77 ± 11.88a
C-CS	39.76 ± 4.21a	83.63 ± 4.74ab	6788 ± 750ab	43.43 ± 9.62a
C-IP	41.76 ± 1.24ab	84.33 ± 16.85ab	7034 ± 573b	45.42 ± 11.42a
C-HT	45.70 ± 5.03bc	88.70 ± 6.12ab	6715 ± 800ab	46.10 ± 13.25a
Sign. (p)	< 0.050	< 0.050	< 0.050	> 0.050

The highest compression strength value was obtained in group A, which had the lowest number of annual rings. Among the group A samples, the highest value was observed in the AHT group, which was subjected to combined modification. Tomak *et al.* (2011) and Bak and Nemeth (2012a) reported that an oil treatment increased the compression strength and the reason for this increase was that the oil filled the wood cell lumen and thickened the cell wall. Heat treatment performed after impregnation caused the greatest increase in compression strength of the tested methods. The reason for this is that the increase in the amount of lignins during the heat treatment caused an increase in strength (Yıldız *et al.* 2006; Bektaş *et al.* 2017).

The combined process (impregnation + heat treatment) yielded the highest bending strength values when the samples were evaluated according to their modification process. The bending strength values of the A-CS, A-IP, and A-HT groups, which have fewer rings per year, were significantly lower. Bao *et al.* (2001) compared the mechanical properties of multiple mature wood and young wood species, and as a result, determined that the strength values of young wood were lower than the strength values of mature wood.

As the number of rings increased annually, the modulus of elasticity increased. Impregnation with oil increased the modulus of elasticity, but heat treatment decreased it. Other studies support the result that the modulus of elasticity increases when the samples are subjected to oil impregnation (Kocafe *et al.* 2008; Bak and Nemetz 2012b). It is stated in many studies that the heat treatment of wood decreases the modulus of elasticity (Bekhta and Niemz 2003; Korkut *et al.* 2008; Düzkale Sözbir *et al.* 2017).

A higher hardness value was obtained in group A, which had a lower number of rings per year. Both oil impregnation and the combined process (impregnation + heat treatment) increased the hardness values. Since the number of rings per year was less and the amount of extractive material was greater in group A, a higher hardness value was found compared to other the groups. Brémaud *et al.* (2011) stated that the extractive substance in the wood moderately affects the hardness value. The reason for the highest increase in hardness due to the combined process is thought to be the increase in the amount of lignins.

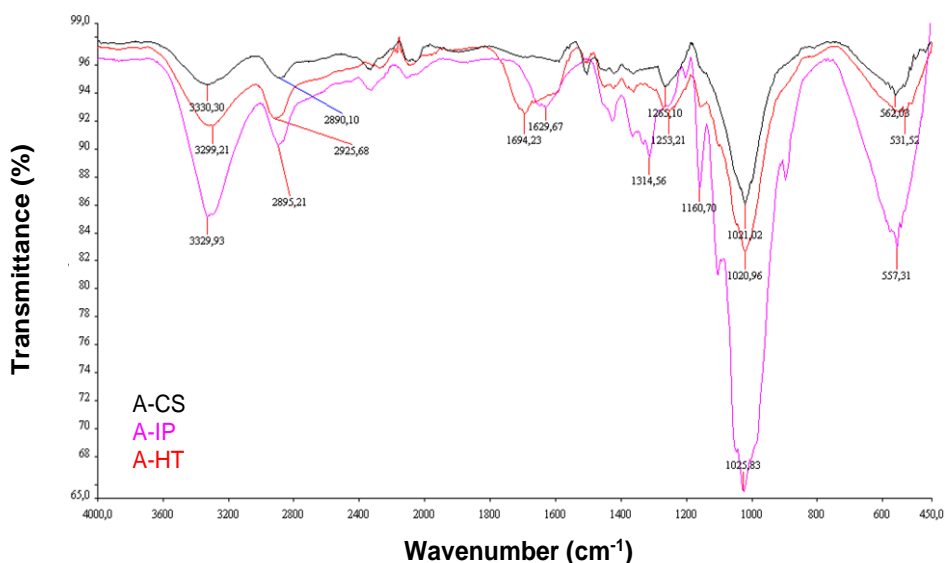


Fig. 1. FTIR spectra of the A-CS, A-IP, and A-HT samples

Figure 1 shows the FTIR spectroscopy results of the A-CS, A-IP, and A-HT groups. The wavelengths ranging from 3330 to 3400 cm^{-1} show the OH stretching of alcohols, phenols, and acids (Gönültaş and Candan 2018). The -OH stretching of A-CS at a wavelength of 3330 cm^{-1} length decreased to a wavelength of 3299 cm^{-1} in the A-HT group. This is an indication that the heat treatment reduces the absorbance associated with -OH stretching. Additionally, the impregnation process had a minor effect on OH stretching. The wavelengths ranging from 2850 to 2970 cm^{-1} correspond to the C-H stretching (Esteves *et al.* 2013).

The C-H bond was found at a wavelength of 2890 cm^{-1} in the A-CS samples, a wavelength of 2895 cm^{-1} in the A-IP samples, and a wavelength of 2926 cm^{-1} in the A-HT samples. For the C-H stretching, the apparent change in frequency is due to changes in the structural and relative wood components, *i.e.*, the degree of crystallinity of cellulose and the change in lignin methoxyl groups (Coates 2000; Moharram and Mahmoud 2008; Spiridon *et al.* 2011).

There was an absorbance band for C = C stretching at a wavelength of 1600 cm^{-1} in thyme oil. While this band was observed at a wavelength of 1629 cm^{-1} in A-IP, it was seen at higher wavelengths (1694 cm^{-1}) in group A-HT, which underwent both the thyme oil and heat treatments. González-Peña *et al.* (2009) attributed the heat treatment to the increase of conjugated carbonyl groups in the lignins.

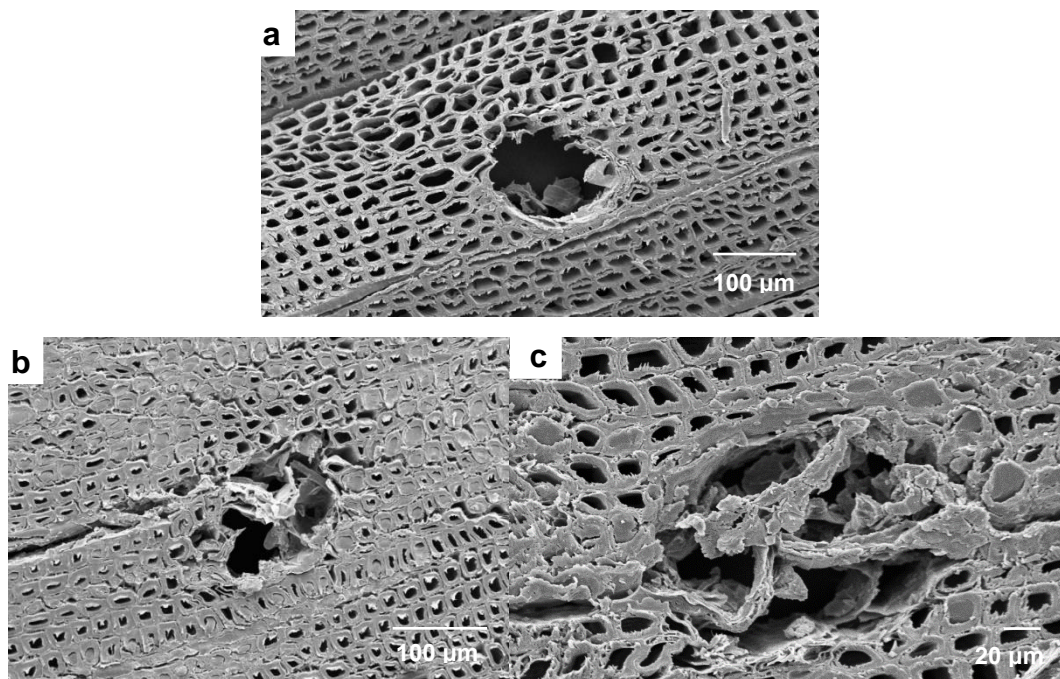


Fig. 2. SEM images of the control group (a) and the thyme oil treatment group (b) images taken at approximately 500x and the thyme oil treatment + heat treatment group images taken at 1.00 Kx

Lignin S units and G units are seen at wavelengths ranging from 1240 cm^{-1} to 1320 cm^{-1} (Esteves *et al.* 2013). The increase in the lignin unit wavelengths (1314 cm^{-1}) in the A-HT group is related to the increase in lignin density. The same phenomenon was reported by Windeisen *et al.* (2007). The reason for the lignin unit wavelength increase in the A-IP group (1265 cm^{-1}) is that the thymol in thyme oil (seen at a wavelength of 1289 cm^{-1}) enters the wood structure and increases the density (Valderrama and De 2017). Valderrama and

De (2017) found that the presence of carvacrol in the thyme yielded a wavelength of 1173 cm^{-1} . The presence of carvacrol attached to the structure in the samples of the A-HT group was seen at a wavelength of 1161 cm^{-1} . The C-O-C deformation peak was higher in the A-HT group. This situation is thought to be caused by the heat treatment.

The SEM micrographs of the group A samples treated in different ways are shown in Fig. 2. The A-CS group image (Fig. 2a) shows the resin channels and the cell walls of the wood samples. Looking at the SEM image of the A-IP group samples (Fig. 2b), it can be seen that thyme oil fills the cell lumens. This can explain the increased weight gain in the samples. In the A-HT group samples, the resin and oil leakage as well as the deterioration of the cell structure are clearly seen. In addition, the cell deformation has been found to be caused by heat treatment. This is the reason for the weight loss, decrease in density, and the low mechanical properties of the samples. In addition, this is the reason for the weight loss and the decrease in density in the samples.

CONCLUSIONS

1. The density and the weight per unit of volume decreased as the number of annual rings increased, and its relationship with extractives has been reported.
2. Since the number of annual rings is evaluated within itself, the lowest volumetric shrinkage, volumetric swelling, and FSP were determined in the group impregnated with thyme oil. The impregnation process provided water repellent properties to lignocellulosic material, and this could be beneficial in the products used in outdoor conditions.
3. In the study, weight gain of the samples under air-dry conditions was determined to be approximately 9% to 17% compared to the annual rings, and this ratio is considered sufficient to provide resistance against fungus.
4. It was concluded that the combined process (impregnation + heat treatment) is more effective in samples exposed to water for a long time.
5. It was determined that just the impregnation process with thyme oil or in combination with heat treatment increased the strength values, but the combined process decreased the modulus of elasticity in groups A-HT, B-HT, C-HT. It was clearly seen that the heat treatment reduced the elasticity of the wood.
6. According to the results of the FTIR analysis, the combined process provided the greatest reduction in hydroxyl groups. With the procedures applied, it was concluded that the thymol and carvacrol found in thyme oil were bonded.

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