Combined Effects of Linseed Oil and Heat Treatment on the Properties of Cypress and Maple Wood Part 1: Water Absorption, Mechanical Properties, and Sound Absorption Capacity

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The physical and mechanical properties of thermally modified wood impregnated with linseed oil were investigated to find the relationship between the treatment and the acoustic properties. The samples were impregnated with linseed oil (LO). Heat treatment was performed in an oven at four different temperatures, namely 160, 180, 210, and 240 °C. Statistically, the physical, mechanical, and acoustic properties of the treated wood were significantly intensified as compared to the control samples. The physical properties (water absorption) of the treated wood decreased by 72% as compared to the control group. However, while the increase in both MOR and MOE at 240 °C was 13%, the increase in CS at 240 °C was 7%. As a result of the heat treatment, the porosity increased by 33% as compared to the control group. At 240 °C the maximum SAC value was observed to be 0.71 at 5000 Hz and at 4500 Hz 0.78 and 0.80, respectively. The highest STL value, which was 69.9 dB, was observed at 1540 Hz and 3600 Hz. As a result of improved heat transfer, impregnating samples with LO before thermal modifications was observed to increase the efficiency of thermal modification.

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INTRODUCTION

Noise has a negative effect on the comfort of people living in urban environments (Zannin *et al.* 2002; Szeremeta and Zannin 2009; Hunashal and Patil 2012). The source of noise can be indoors or outdoors (Tang and Wang 2007; Wang and Kang 2011; Yang and Kang 2022). Building sound barriers on the walls between the interiors and near the sound source in the outer spaces are some solutions. Technically, sound barriers are sound insulation materials that absorb sound perfectly (Jang *et al.* 2020). Sound absorption is based on the principle that the sound is reduced by the friction formed on the surfaces of the pores as it passes through the porous materials and by conversion of sound energy into heat energy (Kang *et al.* 2011; Wang *et al.* 2014; Kang *et al.* 2020).

Mineral-based materials (*i.e.*, rock wool and glass wool) and synthetic materials are widely used to prevent noise. Mineral-based sound insulation materials may pose a risk to human health because they contain mineral particles that can become airborne (Berardi and Iannace 2017). Synthetic materials may cause environmental pollution during their

production and disposal (Yang *et al.* 2020; Gliscinska *et al.* 2021). Synthetic materials turn into micro plastics and pose risk especially to marine life and humans (Almroth *et al.* 2018). Therefore, research has mainly focused on natural, environmentally sensitive, sound insulation materials.

Wood can be used as a sound insulation material due to its porous structures. When a sound wave enters the pores in the wood, the energy of sound decreases, and sound is absorbed (Wang *et al.* 2014). Studies have been carried out to improve the acoustic properties of wood. Low-pressure steam application (Kang *et al.* 2010), delignification process (Kang *et al.* 2008), underwater shock application (Itoh *et al.* 1998), microwave treatment (Wang *et al.* 2014), and heat treatment (Chung *et al.* 2017) are research strategies that target at improving sound absorption capacity.

Wood is an eco-friendly traditional biomaterial that can be used in many different areas (*i.e.*, construction, furniture, and chemical industries) (Rowell *et al.* 2012). Its mechanical resistance and shock resistance are very high as compared to its specific gravity (Popescu and Popescu 2013). Its chemical structure is complex and its main components are cellulose, hemicellulose, and lignin. These main components are present in different proportions in the cell structure, which renders wood heterogeneous (Korkut and Hiziroglu 2014). The presence of many hydroxyl groups in the chemical structure of wood adversely affects its strength performance, dimensional stability, and biological structure, and this can limit the long-term use of wood.

Resistance of wood against outer atmospheric conditions is low. Wood often cannot meet the performance expected from it in the determined service period (Kasemsiri *et al.* 2012; Li *et al.* 2015; Okon *et al.* 2017). Modification methods can overcome such limitations. Researchers have developed new methods to counterbalance these disadvantages of wood. Although wood modification methods such as furfurylation, HT, condensation, and impregnation are reported to improve the elasticity, strength, shock absorption properties of wood partially, the moisture and-water resistance properties are reported to be improved significantly (Pfriem *et al.* 2007; Dietrich *et al.* 2014; Bucur 2016; Krüger *et al.* 2018).

The most common commercial treatment methods are impregnation and HT. The impregnation method, which entails injection of chemicals into the body of the wood, was observed to improve its hydrophobicity and resistance to biological pests and this also extends its lifetime. At the same time, it increases both the dimension and the number of its pores. Different impregnation materials can be used depending on whether it is intended for indoor or outdoor use (Hebisch *et al.* 2020). The CCA salts, creosote oil, coal oil, and other oily compounds are exploited for outdoor use and especially on woods in constant contact with water, roadside sound barriers, bridges, railway sleepers, power transmission poles, and construction molds (Animpong *et al.* 2017; Unnisa and Hassanpour 2017; Belchinskaya *et al.* 2020, 2021).

Since linseed oil impregnation is natural and non-toxic, it is especially preferred for impregnation of wooden materials used indoors and outdoors. LO is a hydrophobic and inexpensive product (Humar and Lesar 2013). Water absorption is decreased and biological resistance is provided by closing the pores of the impregnated wood with a thin film layer (Ulvcrona *et al.* 2006). Linseed oil creates resistance to hydrophilicity directly without chemical bonding between wood and oil (Humar and Lesar 2013).

Heat treatment is a type of wood modification that was developed *via* experimental studies and was then commercialized. Studies have focused on the effect of HT on mechanical properties, dimensional stability, and color of wood (Sailer *et al.* 2000).

For wood materials, the effects of heat treatment studies such as reduction in the emission of volatile organic compounds of wood (Manninen *et al.* 2002), increase in dimensional stability of wood material (Kamdem *et al.* 2000; Papp and Sailer 2001; Tejada *et al.* 1997), improvement in material durability (Hanger *et al.* 2002), and improvement of color uniformity (Sailer *et al.* 2000) were investigated. The data obtained in studies targeting at investigating the effect that heat treatment causes significant changes in the chemical composition of wood have shown that hemicellulose is decomposed (Tjeerdsma *et al.* 1998), the volatile components in wood decreased (Garrote *et al.* 2001; Kamdem *et al.* 2002), and the cellulose crystallinity increased (Bhuiyan *et al.* 2000), especially when the amorphous carbohydrates decompose. Wood acidity increases with heat treatment.

In studies focused on how HT affected acoustic properties (Kubojima *et al.* 1998), such as vibrational performance of wood (Schwarze *et al.* 2008), the temperature was in the range 120 to 200 °C and the process was carried out for a period between 0.5 and 16.0 h in the presence of N_2 or air. They researched acoustic performance of Norway spruce and American sycamore. In another study, effect of HT on acoustic properties of Chinese fir the study was conducted at 120 °C, 140 °C, 160 °C, 180 °C, 200 °C, and 220 °C, and the holding time of was 2 h (Zhu *et al.* 2016). The acoustic performance was studied on cellulose crystals (Ma 2005). However, a very limited number of studies were carried on sound absorption performance of wood.

Due to the heat treatment, the disadvantages related to the use of wood material (water absorption, change in size, and color, decay, *etc.*) are minimized, and the use of such heat-treated wood material has become widespread. However, combination of this modification method with other modifications has not been extensively studied. On a study performed to this end, the effect of resistance properties of heat-treated wood was investigated relying on the strong correlation between the effect of impregnation of wood with an alkaline buffering medium such as borate and its acidity and thermal degradation (Awoyemi and Westermark 2005). Salman *et al*. (2014) used boric acid in combination with HT. Irbe *et al.* (2020) applied the modification methods thermo-hydro treatment and copper impregnation processes together to increase the resistance of wood against rot fungi and found that the combined use of these two processes together against brown rot was more effective than using only one of these treatment procedures.

The present study was targeted at investigating the effect of impregnation on two different heat-treated woods, namely the Mediterranean cypress (*Cupressus sempervirens* L.) and field maple (*Acer campestre* L.) wood. The pretreatment of impregnation was performed with a natural solution LO, and the HT was performed at four different temperatures (160, 180, 210, and 240 °C). The treatment methods used in this study alone were previously reported to improve the properties of wood (Temiz *et al.* 2013; Chen *et al*. 2020; Yermán *et al.* 2021; Perdoch *et al.* 2022). The main motivation for this study was to improve the humidity and water resistance, dimensional stability, mechanical resistance, sound absorption, and reflection properties of heat-treated impregnated wood.

EXPERIMENTAL

Materials and Methods

Mediterranean cypress (*Cupressus sempervirens* L.) and field maple (*Acer campestre* L.) wood samples were harvested randomly from the natural forest in Burdur city, Turkey. The information regarding the collected samples is given in Table 1. The

samples were prepared to have regular fibers, no knots or cracks, and to be as perfect as possible. The samples were prepared by using sapwood lumber. The samples were cut into 600 ×120 × 20 mm^3 (longitudinal, tangential, radial) dimensions before HT. The samples were kept in the air conditioning room at 20 ± 2 °C with relative humidity of 65% ±5 until they attained a relative humidity of 12%. The dimensions of the samples, each of which were prepared in 30 for the experiments, were 30 × 20 ×20 mm^3 (longitudinal × radial × tangential), 300 × 20 × 20 mm^3 (longitudinal × radial × tangential), 100 × 20 mm^2 (diameter × thickness), respectively, and they had 29 × 20 mm^2 (diameter × thickness).

Table 1. Basic Information on Tree Samples Collected

Common Name	Scientific Name	Air Density (g/cm^3)	D.B.H (cm)	Location
Mediterranean Cypress	*Cupressus sempervirens* L.	0.601(0.07)	30-42	Burdur, Turkey (N 37° 11', E 29° 27')
Field Maple	*Acer campestre* L.	0.564(0.05)	30-45	

The natural LO used in the study was obtained from the BTC chemistry company (BTC Europe GmbH NY 8001-26-1, Germany) with 100% purity. Its technical properties are given in Table 2.

Table 2. Physical Properties of Linseed Oil

Indicator	Measured Value
Specific gravity (g/cm^3 @ 25°C)	0.92
Melting point (°C)	-24
Viscosity (mPas)	3900
Refractive index	1.48
Water content	<0.1

Impregnation of Wood Samples with Linseed Oil

The wood samples, which were preconditioned before the process, were immersed into containers filled with linseed oil (LO) at 130 °C and were kept in the containers for 60 min. Then they were placed and kept in another container filled with oil at 30 °C for 60 min (cooling container). The method applied for the wood samples was the hot-cold bath method (Esteves *et al.* 2014). In this method, the impregnating oil penetrated into the voids, taking advantage of vacuum that forms during the warming and cooling process without applying pressure (Lahtela and Kärki 2016). Therefore, this is an effective method by which to form a protective layer inside the internal surface as well. In the next step the wood samples were dried by keeping them at room temperature for 7 days, and then they were dried in the oven 60 ± 10 °C for 24 h. During this process, the temperature was adjusted to keep the oil leakage under control. Sorption of the impregnated oil was evaluated as per weight (WPG). Specimens that were not impregnated were kept as the control group.

Heat Treatment (HT)

Heat treatment was performed using a temperature-controlled electrical laboratory type oven (Supertherm HT16/16, Nabertherm GmbH, Lilienthal Germany). The samples from the two different tree families – namely Mediterranean cypress (*Cupressus*

sempervirens L.) and field maple (*Acer campestre* L.) – were heat-treated at atmospheric pressure under oxygen-free conditions. The heat treatment of wood samples was carried out in three main steps (Fig. 1). In the first step, water was removed from the wood starting with the ambient temperature 20 °C and rapidly heating up to 40 °C, followed by a slower increase in temperature in the range of 40 to 130 °C. This step was applied in the same way for each sample. In the second step of heat treatment, the targeted temperature (160, 180, 200, or 240 °C) was reached starting from 130 °C. When the targeted temperature was reached, the temperature was kept constant for 3 hours. In the third step, the samples were cooled gradually until the temperature of the samples reached room temperature.

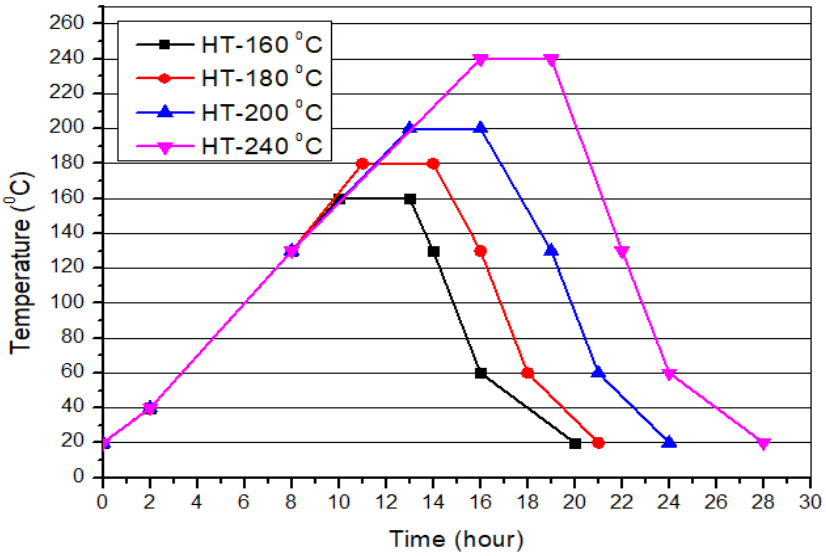

Fig. 1. Heat treatment steps

Table 3. Treatment Groups

Testing Groups	Non-Impregnated	LO-Impregnated	Temperature (°C)			
			160	180	210	240
Control	x					
Group 1	x		x			
Group 2	x			x		
Group 3	x				x	x
Group 4	x					
Reference		x	x			
Group 1		x		x		
Group 2		x			x	
Group 3		x				x
Group 4		x				
x: Specified treatment was applied on the samples.						

At the end of this process, the lid of the device was opened after the samples were cooled down to room temperature. After heat treatment was applied, the test specimens were kept in an air-conditioned cabinet until they reached constant weight under the conditions of 65±5% relative humidity at 20±2 °C. In this case, heat treatment was applied to the untreated (Untreated) and the reference (impregnated pre-treated) groups. Nine different treatment groups were used for comparisons (Table 3).

Measuring Weight Gain in Percentage (WPG)

Weight Gain in Percentage (WPG) was determined after impregnation process. WPG was determined according to Eq. 1,

$$WPG = ([m_1\text{-}m_2]/m_2) \times 100\ (\%) \quad (1)$$

where m_1 and m_2 are the dry weight (g) after and before impregnation, respectively.

Density Determination (DD) and Mechanical Properties (MP)

Density was determined according to ISO13061-2 standard (2014) *via* the stereometric method, which relies on measuring the densities, volumes, and weight of the samples. Weight of each sample was determined by using analytical balance (with 0.001 g accuracy). To find the volume, all dimensions were determined with a digital caliper with 0.01 mm precision. The densities were calculated *via* Eq. 2,

$$d_o = m_o/v_o \quad (2)$$

where d_o is the density of the oven-dried sample (g/cm³); m_o is the weight of the oven dried sample (g) and v_o is the volume of the oven dried sample (cm³).

The mechanical tests were performed with a Shimadzu AGS-X (10 kN) universal test device. The bending elasticity modulus test (MPa) was used to determine the stiffness of the wood by using a four point system according to the ISO 3349 (1975) standard. The distance between the support points was 320 mm, and the loading points were at an 80 mm distance. The static bending resistance test (MPa) represents the maximum weight that wood can momentarily support before breaking when the ends of the test sample are placed on a support and when the sample is gradually loaded from two points according to the ISO 3133 (1975) standard. The compression strength (||) (MPa) ISO 3787 (1976) standard represents the maximum weight applied before the sample breaks.

The changes in the mechanical properties and density of the wood samples before treatment (control and reference groups) and after treatment at different temperatures were calculated according to Eq. 3,

$$\Delta_{d,MOR,MOE,CS} = ([S_{C,R}\text{-}S_1]/S_{C,R}) \times 100\ (\%) \quad (3)$$

where $S_{C,R}$ indicates the values before the treatment and S_1 indicates the values after the treatment, respectively.

Water Absorption (WA)

The samples were conditioned until they attained constant weight. The samples were immersed in a container filled with water at 20 °C and then they were removed out of the water in periodic time intervals of 2 h, 24 h (1 d), 48 h (2 d), 72 h (3 d), 96 h (4 d), 120 h (5 d), 168 h (7 d), 336 h (14d), 504 h (21 d), and 720 h (30 d), and they were dried with a dry paper before the measurements (Poncsak *et al.* 2011). The weight measurements were performed. Water absorption was determined according to Eq. 4,

$$WA = ([w_1-w_2]/w_2) \times 100\ (\%) \quad (4)$$

where w_1 and w_2 are the weight (g) after and before immersion, respectively.

Porosimetry (P)

Mercury inlet porosimetry (MIP)

Mercury intrusion porosimetry (MIP, AutoporeTM IV 9500 Automated Mercury Porosimeter, Micrometrics Instrument Corp., USA) was used to characterize the porosity, pore dimensions, and pore size distributions of the samples. The samples with the dimensions 8×6×6 mm (L×T×R) were dried in an oven at 103 ± 2°C before the tests. The pressure capacity of the equipment used was 200 MPa. The process parameters were as follows: mercury with surface tension of 0.485 g/cm^2 and the density lies between 13.5325 and 13.5379 g.mL^{-1}. The contact angle between the mercury and the wood (forward-backward) was 130°, and the balance period between the high and low pressure was 10 s.

Scanning Electron Microscope Imaging (SEM)

The changes in the microstructures of the samples used in this study were investigated *via* a Hitachi S-4800 Surface Emission Scanning Electron Microscopy (SEM, Japan). The samples were cut in the tangential and cross-section directions with 5x5 mm^2 dimension. The acceleration voltage in this study was 10 kV. The samples were mounted onto the aluminum studs *via* double sided tape and were gold plated *via* spraying. Then the microstructures of the tangential and cross-sections were analyzed.

Sound Absorption Coefficient (SAC)

The sound absorption capacity (α) of the wood samples was tested according to ISO standard 10534-2 (1998) method by using impedance tubes of Type SW 422 and Type SW 477 (BSWA Technology Co., Ltd., China). The air void between the sample and the steel support in the sound absorption measurements was 10 mm. The sound absorption coefficients of the 1/3-octave frequencies were measured by using a big tube (SW 422, Φ 100 mm) in the frequency range between 63 and 1600 Hz. The sound frequency absorption coefficients in the frequency range between 1600 and 6300 Hz were measured by using a small tube (SW 477, Φ29 mm).

Sound Transmission Loss (STL)

Sound transmission loss in the frequency range of 50 to 6400 Hz was measured by using ASTM standard E2611-Type SW 422 and Type SW 477 impedance tubes (BSWA Technology Co., Ltd., China). An impedances tube of 29 mm diameter was used for measuring sound. The transmission loss was measured in the frequency range of 50 to 6400 Hz at ambient conditions of 20 °C and 1031 hPa.

Statistical Analysis

Statistical differences in the mean values of the parameters investigated in this work were estimated by using two-way analysis of variance (ANOVA) to investigate the effects of different types of impregnating oil and HT (temperature) on physical-mechanical and acoustic properties. When significant differences were detected by ANOVA, Duncan's test was used to evaluate the relationship between applied temperature and impregnation. All statistical analyses were performed *via* SPSS® 20.0 for Windows®. The mean values were accepted to be considerably important when $p \leq 0.05$.

RESULTS AND DISCUSSION

Measuring Weight Gain as a Percentage (WPG)

Table 4 presents the results of the Mediterranean cypress and field maple wood samples impregnated with linseed oil (WPG). It can be seen that after impregnation, 39.5 % of the linseed oil was retained inside the wood. This is considered to be a high value for treatment. Temiz *et al.* (2013) reported similar results in their study in which LO was used as impregnating agent and following impregnation WPG was found to be 40.4%. This indicates that LO highly penetrated the wooden structure. Lahtela *et al.* (2016) treated Scots pine (*Pinus sylvestris*) wood with melamine, and the average WPG was found to be 38.71%, which also indicates strong penetration into the wood structure.

Table 4. WPG Values in Various Linseed Oil Impregnation Treatments

	Before Impregnation (g)	After Impregnation (g)	WPG (%)
Mediterranean Cypress	10.33 ± 0.11	14.41 ± 0.08	39.50 (2.85)
Field Maple	9.87 ± 0.16	13.44 ± 0.14	36.20 (4,21)

Density Determination and Mechanical Properties

Sample densities before and after modification are shown in Table 5. As presented in Table 5 and Fig. 2, when the Mediterranean cypress and field maple wood samples are compared with the control group (Un-treated) for the non-impregnated samples, as the temperature increased, the mechanical properties such as the modulus of rupture (MOR-bending strength) and modulus of elasticity (MOE), as well as the modulus of rupture (MOR-bending strength) and modulus of elasticity (MOE) values, decreased, while the compression strength (CS) increased, as it is generally accepted (Korkut *et al.* 2008; Ateş *et al.* 2009; Militz and Altgen 2014).

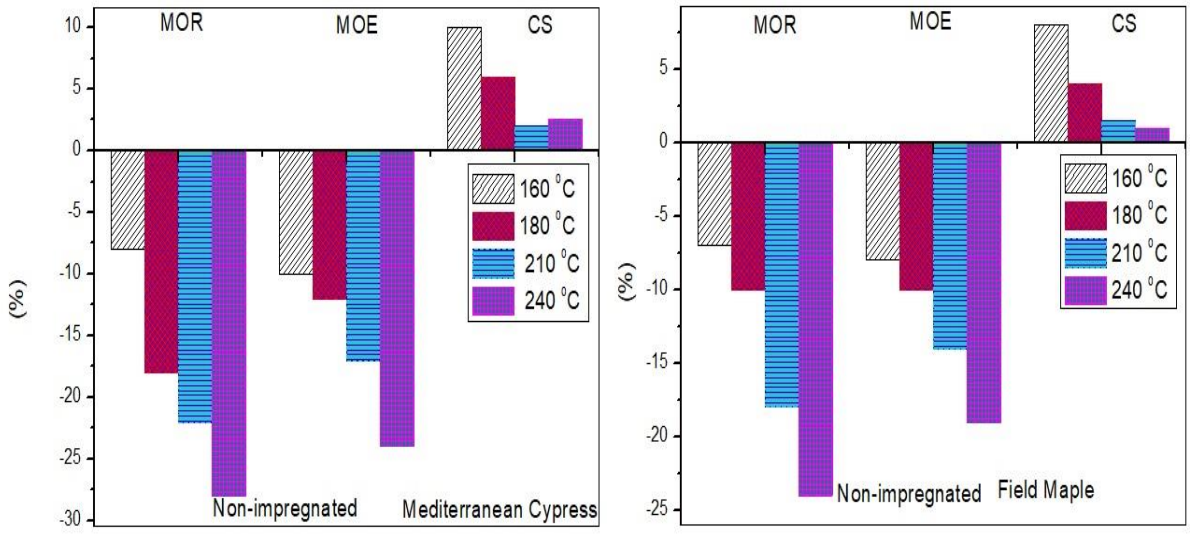

Fig. 2. Percentage of change in the mechanical properties of the non-impregnated samples as a function of HT

Table 5. Properties Before and After Impregnation and HT

Sample	Impreg.	HT	Density (g/cm³)	MOR (N/mm²)	MOE (N/mm²)	CS (N/mm²)
Mediterranean Cypress	Non Impregnation	Control	0.574 (0.015)*A**	111.80 (2.89)A	10930.5 (157.5)A	78.56 (3.15)BC
		160 °C	0.554 (0.007)B	102.85 (3.07)C	9387.2 (179.3)BC	86.41 (3.12)AB
		180 °C	0.539 (0.008)BC	91.67 (2.60)H	9618.4 (202.1)B	89.27 (4.16)A
		210 °C	0.513 (0.014)C	87.20 (2.84)HI	9071.9(303.5)C	80.13 (2.89)B
		240 °C	0.471 (0.012)D	80.49 (2.01)DE	8306.8 (256,8)CD	80.52 (3.09)B
Field Maple	Non Impregnation	Control	0.601 (0,058)A	108.14 (2.29)B	8892 (136.30)C	74.30 (2.29)BC
		160 °C	0.588 (0,032)AB	100.57 (3.54)C	8180.64 (251.23)E	80.24 (3.18)A
		180 °C	0.535 (0,054)BC	97.32 (4.15)CD	8002.80 (200.12)EFG	77.27 (4.22)AB
		210 °C	0.513 (0,080)CD	88.67 (3,11)D	7647.12 (198.25)G	75.41 (3.01)B
		240 °C	0.423 (0,056)D	82.18 (2,88)E	7202.52 (191.10)GH	75.04 (2.22)B
Mediterranean Cypress	LO Impregnated	Reference	0.756 (0,088)A	101.12 (4,25))E	10336 (222,90)DEF	74.36 (3.25)CD
		160 °C	0.744 (0,054)B	102.36 (2,56)CD	10542 (265,54)DEF	74.38 (4,25)C
		180 °C	0.739 (0,048)BC	103.12 (2,10BC)	10668 (306,98)DE	74.38 (3,65)C
		210 °C	0.734 (0,095)CC	103.95 (5,23)BC	10852 (333,99)D	74.37 (2,25)C
		240 °C	0.735 (0,010)CC	108.25 (2,98)B	10911 (205,28)BD	74.35 (4,99)CD
Field Maple	LO Impregnated	Reference	0.729 (0,071)A	98.8 (2,19)CD	8460 (105,23)DE	70.22 (3,54)DE
		160 °C	0.719 (0,092)AB	100.8 (2,65)C	8730 (159,35)CD	71.01 (3,12)D
		180 °C	0.716 (0,041)B	105.6 (4,98)BC	8550 (258,32)D	72.14 (5,89)D
		210 °C	0.712 (0,021)BC	107.4 (5,12)B	9306 (365,54)B	74.42 (2,15)BC
		240 °C	0.710 (0,035)C	112.3 (3,54)A	9512 (412,5)A	76.15 (2,99)AB

* Standard deviation(SD),** Homogeneity groups(HG)

For the Mediterranean cypress wood samples, the maximum decrease in MOR was observed at 240 °C and was 27%. The decrease in MOE at the same temperature was 23%. The increase in the CS value at 160 °C was 9%. The increase in the CS tended to decrease as the temperature increased, and it was not more than 2% at 210 °C. The decrease in MOR for the field maple wood samples at 240 °C was 23% and the MOE was 18%. The increase in the CS at 160 °C was 6%. The reaction of the Mediterranean cypress wood to heat was more pronounced than that of the field maple wood.

In a similar way, when decrease in density was observed for the Mediterranean cypress wood, it decreased from 0.574 to 0.471 g/cm^3 at 240 °C, and the loss in density was 18%. The loss in density for the field maple wood was 30%. The main reason for this decrease was the low moisture content due to removal of water in the structure and the degradation that starts in the wood and especially in the hemicellulose components (Ateş *et al.* 2009; Boonstra *et al.* 2007). In Table 5 and Fig.3, Mediterranean cypress and field maple wood samples were compared with the reference group impregnated with linseed oil (LO) (impregnated with LO but not heat treated). It was seen that as the temperature increased the mechanical properties such as MOR and MOE increased but CS did not change. For the Mediterranean cypress wood samples, the maximum increase in MOR was observed at 240 °C, and it was 8%. The increase in MOE was 4% at 240 °C. For the field maple wood samples, the increase in both MOR and MOE was 13% at 240 °C, while the increase in CS at 240 °C was 7%.

Monitoring the loss in density, it was observed that for the Mediterranean cypress wood the loss was 3%. While HT caused a decrease in the impregnated samples, it was observed that the MOR, MOE, and the CS values of the samples that were thermally processed with linseed oil treatment increased by ~10%. This can be considered as evidence of the positive effect of natural linseed oil. Other studies reported a similar increase in MOR and MOE upon impregnation with wax (Humar *et al.* 2017).

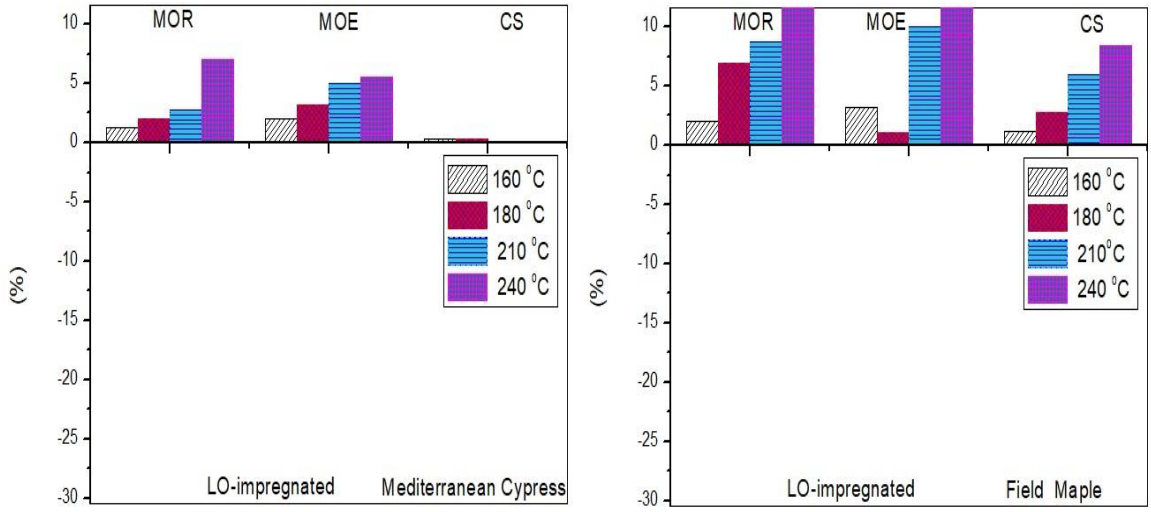

Fig. 3. Percentage of change in the mechanical properties of the samples impregnated with LO as a function of HT

Water Absorption of Wood (WA)

Absorption of water by wood is important in buildings and industrial applications. Depending on the area of use (*i.e.*, indoor or outdoor), wood material is generally subject to water, water vapor, and water leakage. This affects dimensional stability and durability of wood. Wood is hygroscopic in nature and therefore it is rendered more hydrophobic through treatment. Impregnation and HT are widely used for this purpose.

Water absorption properties of the non-impregnated but heat-treated Mediterranean cypress and the field maple wood samples are presented in Fig. 4. In the first 300 hours of the water absorption test, it was observed that water absorption ratio of the non-impregnated samples was similar. As the treatment temperature increased, water absorption decreased. It was observed that at 240 °C the lowest absorption was 39% for

the field maple wood. This data became stable after 400 hours. In a similar way after 350 h stability was achieved for the Mediterranean cypress wood. This situation can be explained by the degradation and decomposition of hemicellulose, which is hydrophilic component of wood, in both tree species, and by the fact that the extractive agents become mobile at high temperatures and evaporate to a certain extent and the remaining part migrates to the surface, covers the surface, and decomposes. Metsä-Kortelainen and Viitanen (2012) have achieved similar results in their study. In another study, Nuopponen *et al.* (2003) achieved this effect through softening lignin due to the mobility of the extractive agents. The water absorption properties of the wood, which were obtained from the Mediterranean cypress and the field maple trees, impregnated with linseed oil and the reference (Ref. LO) wood samples after HT are presented in Fig. 5.

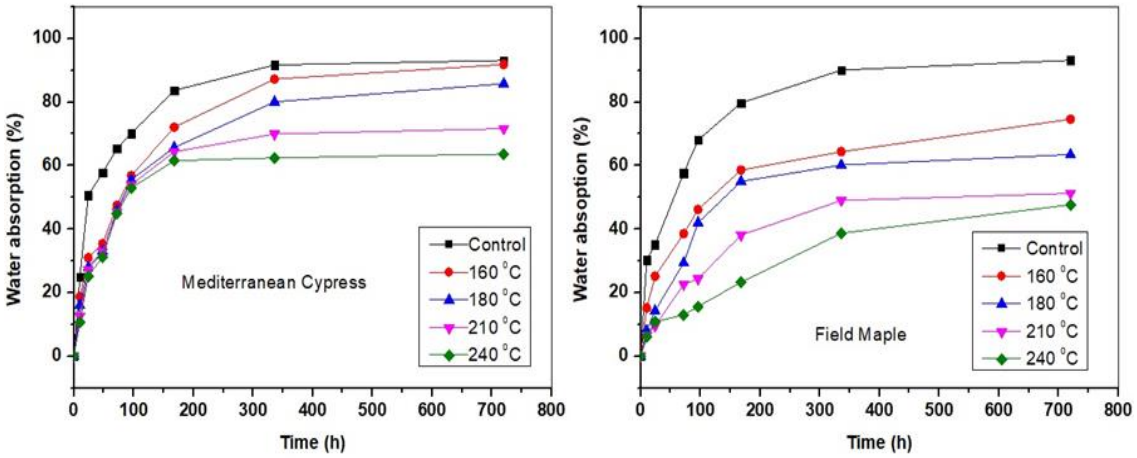

Fig. 4. Water sorption data of the Mediterranean cypress and field maple wood after HT (heat treatment)

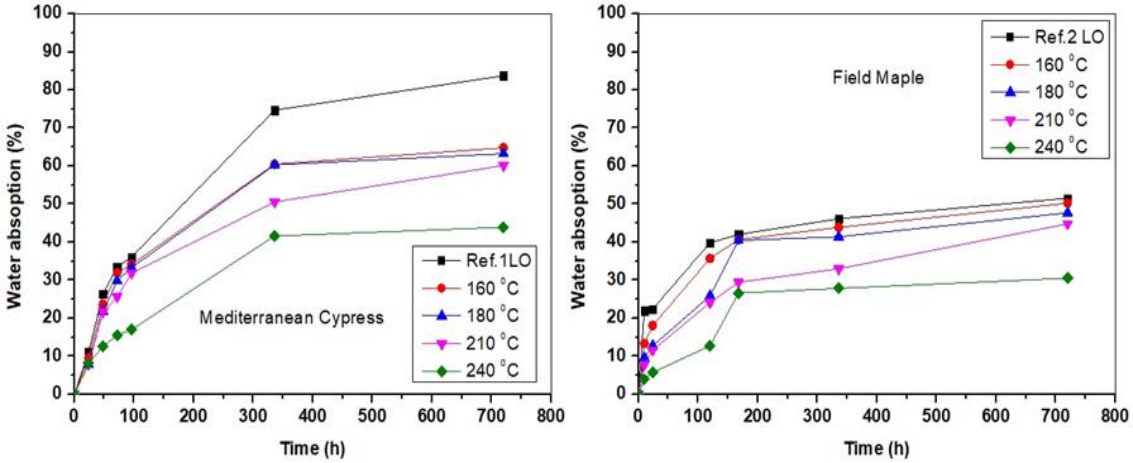

Fig. 5. Water sorption data of the Mediterranean cypress and field maple wood impregnated with LO and after HT (heat treated)

The data in Fig. 5 show that after 240 °C treatment, water absorption from the Mediterranean cypress wood at the end of Day 30 (720 h) was 36%. At the same temperature and in the same time interval water absorption was 80% for the reference group. This result shows that a 55% improvement in water absorption was found to be 65%

for the control group. The finding of a similar result for field maple wood shows that a 50% improvement in water absorption was found to be 72% for the control group. According to the results due to the effect of HT, LO – an impregnating agent – provided considerable input, and thus high hydroscopic property was attained. This finding shows the importance of impregnation with linseed oil. Meyer *et al*. (2016), Humar *et al.* (2017), and Fojutowski *et al.* (2009) have achieved similar results.

Porosimetry

Wood material is a porous polymer in nature. The wood pore structure directly affects its density, permeability, dimensional stability, durability, impregnability, absorption, and acoustic properties, *etc*. (Hill and Papadopoulos 2001). The pore size, amount, and pore type of wood change with the type of the wood, the height of the tree, the meteorological conditions of the place where the tree is located, and the anatomical structure of the wood (Ona *et al.* 1999; Almeida and Hernández 2007).

The porosimetry results of the wood samples presented in Table 6 indicate that the porosity was 62.2% for the non-impregnated Mediterranean cypress wood at 240 °C before impregnation and 65.3% after HT. It was observed that the porosity increased by 4.9% for the Mediterranean cypress wood with temperature. The change in porosity was 58.1% and 64.8%, respectively, for the non-impregnated field maple wood. Porosity was observed to increase with the temperature systematically with the field maple wood, and the ratio of change at 240 °C was 11.5%. Porosity increased as a function of temperature in HT, and porosity and dimensional stability increased due to the degradation of hemicellulose followed by decrease in the number of the hydroxyl groups.

Table 6 shows 59.3% and 78.7% increase in LO impregnated Mediterranean cypress wood as compared to the pre-treated wood. After the treatment with LO impregnating agent, the porosity increased by 32.6%. The porosity before and after impregnation of the field maple wood with LO were 55.2% and 74.8%, respectively. Impregnation of the field maple wood with LO caused a 35.3% increase in porosity. When HT was applied on LO-impregnated Mediterranean cypress at 240 °C, the porosity increased for both of the tree types. The wood samples with the highest porosity were filled with the impregnating oils the most. Porosity was observed to increase after impregnation because, in the course of impregnation, LO fills the air voids in the cell walls in the wood and this has adverse effect on porosity.

Table 6. Porosity of the Treated and Untreated Trees

Untreated (Control)	Cypress(%)	62.21(5.70)*	**Impregnated LO (Reference)**	Cypress(%)	59.33(7.77)
	Maple(%)	58.12(4.12)		Maple(%)	55.25(7.54)
Porosity heat to 160 °C	Cypress(%)	63.28(6.25)	**Porosity LO 160 °C**	Cypress(%)	61.99(8.56)
	Maple(%)	60.51(3.58)		Maple(%)	57.45(10.15)
Porosity heat to 180 °C	Cypress(%)	64.25(6.87)	**Porosity LO 180 °C**	Cypress(%)	70.50(3.20)
	Maple(%)	62.32(5.74)		Maple(%)	64.47(4.20)
Porosity heat to 210 °C	Cypress(%)	64.41(6.69)	**Porosity LO 210 °C**	Cypress(%)	74.84(2.20)
	Maple(%)	63.54(4.87)		Maple(%)	69.67(7.40)
Porosity heat to 240 °C	Cypress(%)	65.28(3.42)	**Porosity LO 240 °C**	Cypress(%)	78.68(4.44)
	Maple(%)	64.80(8.47)		Maple(%)	74.75(5.89)
* Standard deviation(SD)					

Scanning Electron Microscope Imaging (SEM) Studies

In Figs. 6 and 7, the SEM micrographs of the Mediterranean cypress and the field maple wood, which depict the effect of thermal process after impregnation and HT, are presented. When the internal surface images of the cell walls of the wood samples given in Fig. 6 are investigated. As can be seen in Fig. 6 (a to d), no deformation was observed in the cell structure.

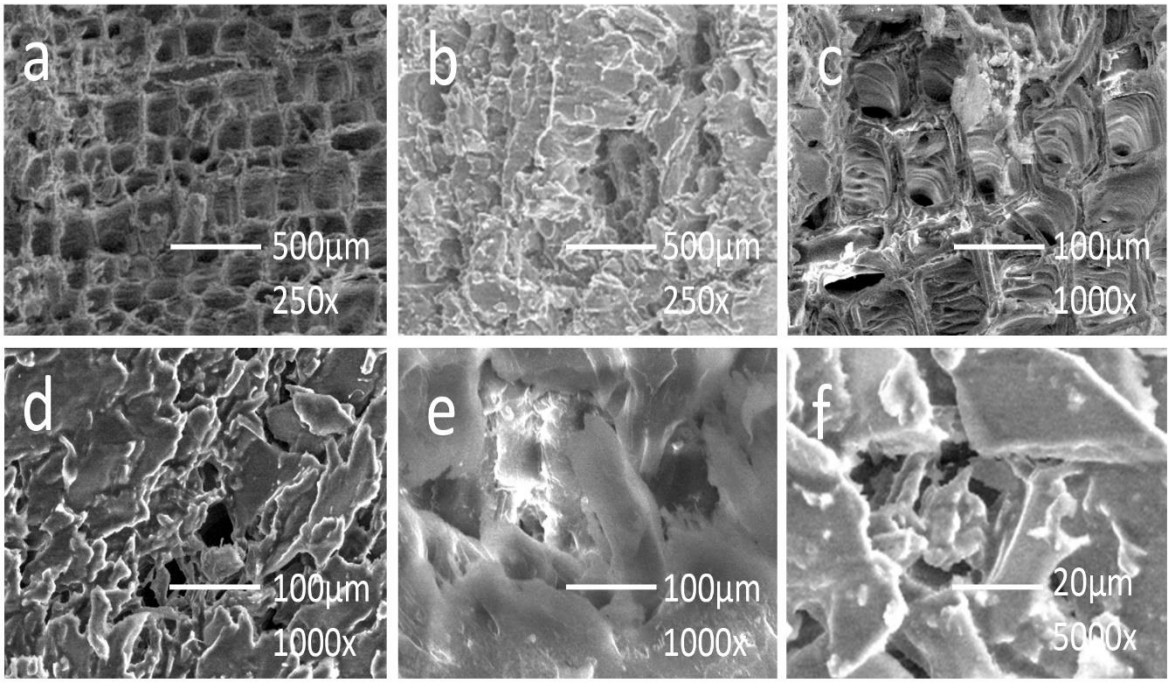

Fig. 6. SEM micrographs of Internal surface of the cell wall in Mediterranean cypress and field maple woods untreated (control) (a and d), impregnated (LO) (b and e), impregnated and heat treated at 240 °C (c and f)

In Fig. 6 (b-e) it can be seen in the images that LO filled the cell voids after impregnation. The extent of absorption in this image is in conformity with the result of the WPG results. The average WPG of the impregnating agents LO was found to be 37.8%. The micro-cracks that form on the surface after the HT are indicative of the deformation caused by the heat. Under the influence of heat treatment, the shape of the cell changed and the cell wall became rough. The intercellular bonds were broken by the effect of heat. Both the micro-cracks and the bubbles caused by boiling of LO can be seen in Fig. 6 (c-f).

In a similar way, in Fig. 7 different images obtained due to the properties of the impregnating agent can be seen. The empty cell lumen observed in Fig. 7 (a-d) indicates that no treatment was given. Figure 7 (b-e) shows the diffusion of the impregnating agent, which has expanded upon heating, into the internal pores of the wood. The voids in the cells are partially filled with the impregnating oils and they act as a barrier against water and humidity. Due to the differences in pressure, partial damage and collapses in the cell walls were observed after impregnation. In Fig. 7 (c-f), the damage, which was caused by the migration of the impregnating agent from inside out and its entrapment inside the voids caused by the mobility due to HT, can be seen. This supports the finding that porosity increases with increase in the temperature of wood. It is observed that hemicelluloses

decomposed and lignin was plasticized, and thus structural changes took place in the cell and the voids increased due to the physical and chemical change caused.

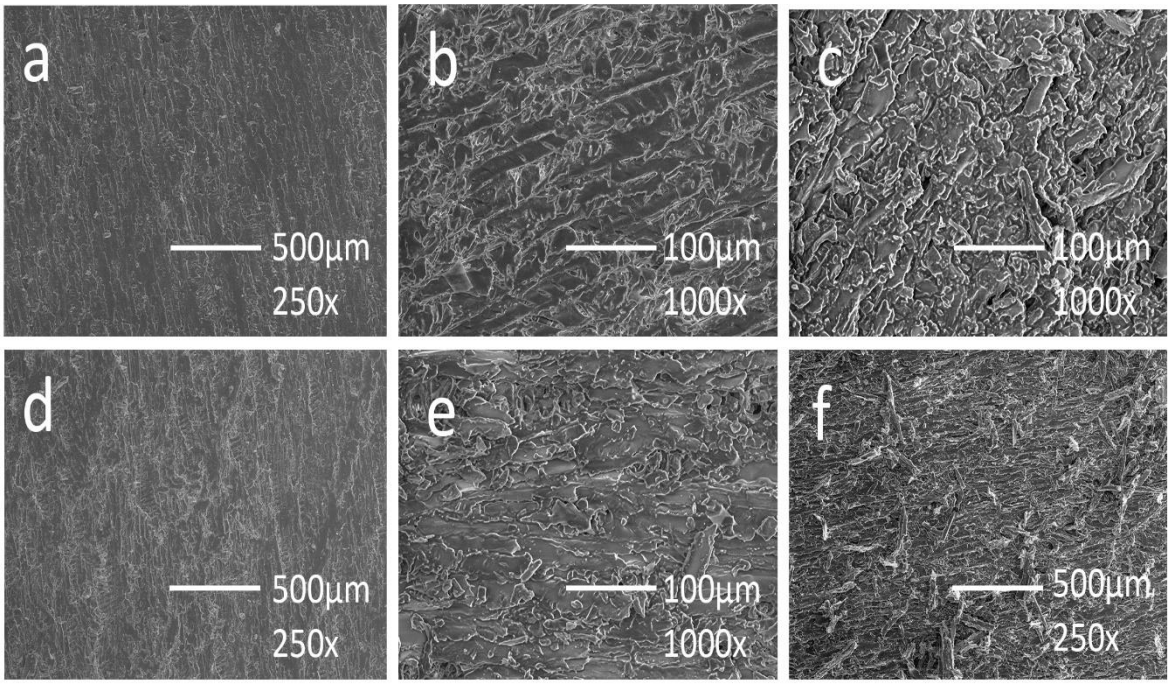

Fig. 7. SEM micrographs of cross-sectional surface of the cell wall in Mediterranean cypress and field maple woods untreated (control) (a and d), impregnated (LO) (b and e), impregnated and heat treated at 240 °C (c and f)

Sound Absorption Coefficient (SAC)

In Fig. 8 a-d are given the SAC values measured in the 100 to 6300 Hz frequency range before (control) and after impregnation of the Mediterranean cypress wood and the field maple wood samples with LO. In general an increase was observed in sound absorption coefficient due to the effect of impregnation. In Fig. 8a it can be seen that for the Mediterranean cypress wood samples the average sound absorption coefficients were 0.19 and 0.28 before impregnation (control) and after impregnation with LO and in the tangential direction the SAC were 0.12 and 0.17 (Fig. 8c).

In Fig. 8b, the field maple wood samples the average sound absorption coefficients in the longitudinal direction before (control) and after impregnation with LO were found to be 0.23 and 0.31, and in the tangential direction after impregnation with LO the average sound absorption coefficients were found to be 0.14 and 0.17, respectively (Fig. 8d).

Figure 8b shows that the impregnating agents (LO) decreased the SACs to the 100 to 1000 Hz range. While the effect of impregnating agent could not be observed in the frequency range of 1000 to 2000 Hz, they were found to increase sound absorption by 10% in the 2000 to 6300 Hz frequency range.

When the data given in Figs. 8a and 8b were evaluated at low frequency bands, the impregnating agents were observed to stop the sound waves from entering the wood. Since the cell wall was full of the impregnating agent after the impregnation process, the sound waves could not reach this distance. Most of the low frequency sound waves were reflected

back. However, as the frequency increased, the situation was reversed. When the frequency of the sound waves increased, they moved toward the cell wall but could not exit from there and they were trapped inside, because after the impregnation with the collapse of the cell plates inside and the contraction of the cell wall, the sound energy was rapidly converted into heat energy.

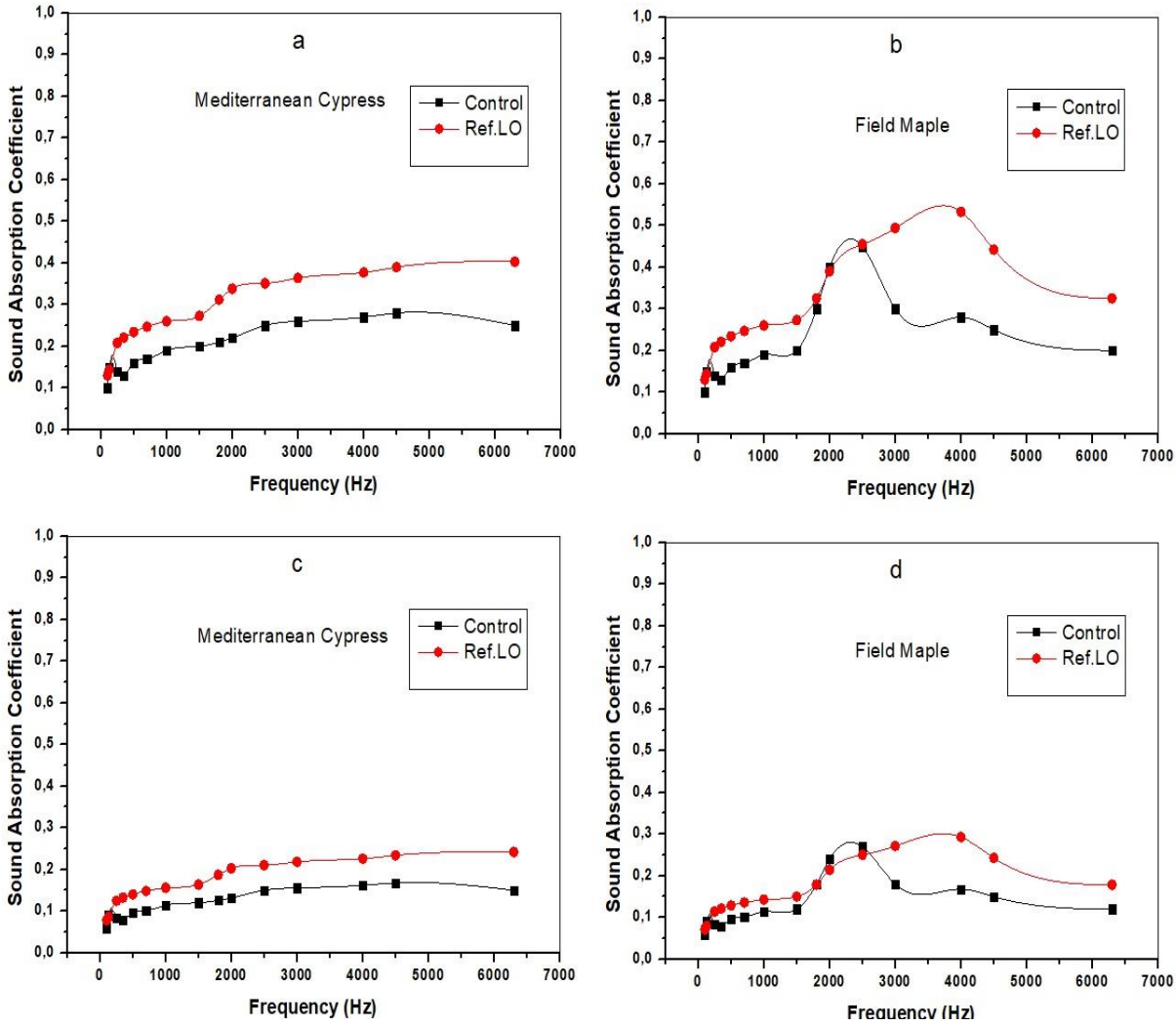

Fig. 8. The sound absorption coefficient (SAC) of the Mediterranean cypress and the field maple wood samples before impregnation (control) and after impregnation with LO, longitudinal direction (a-b), tangential direction (c-d)

The natural LO impregnating agents vibrate with high frequency sound waves and form resonance and synergistically contribute to absorption of sound inside the wood. Liu *et al*. (2008) achieved similar results in their work. About half of the effect of this situation in the longitudinal direction occurred in the tangential direction.

In Fig. 9a, the HT results of the LO impregnated Mediterranean cypress wood are given, and the SAC value, which peaked at 5000 Hz at 240 °C, was 0.71. At 240 °C, the average SAC was found to be 0.30.

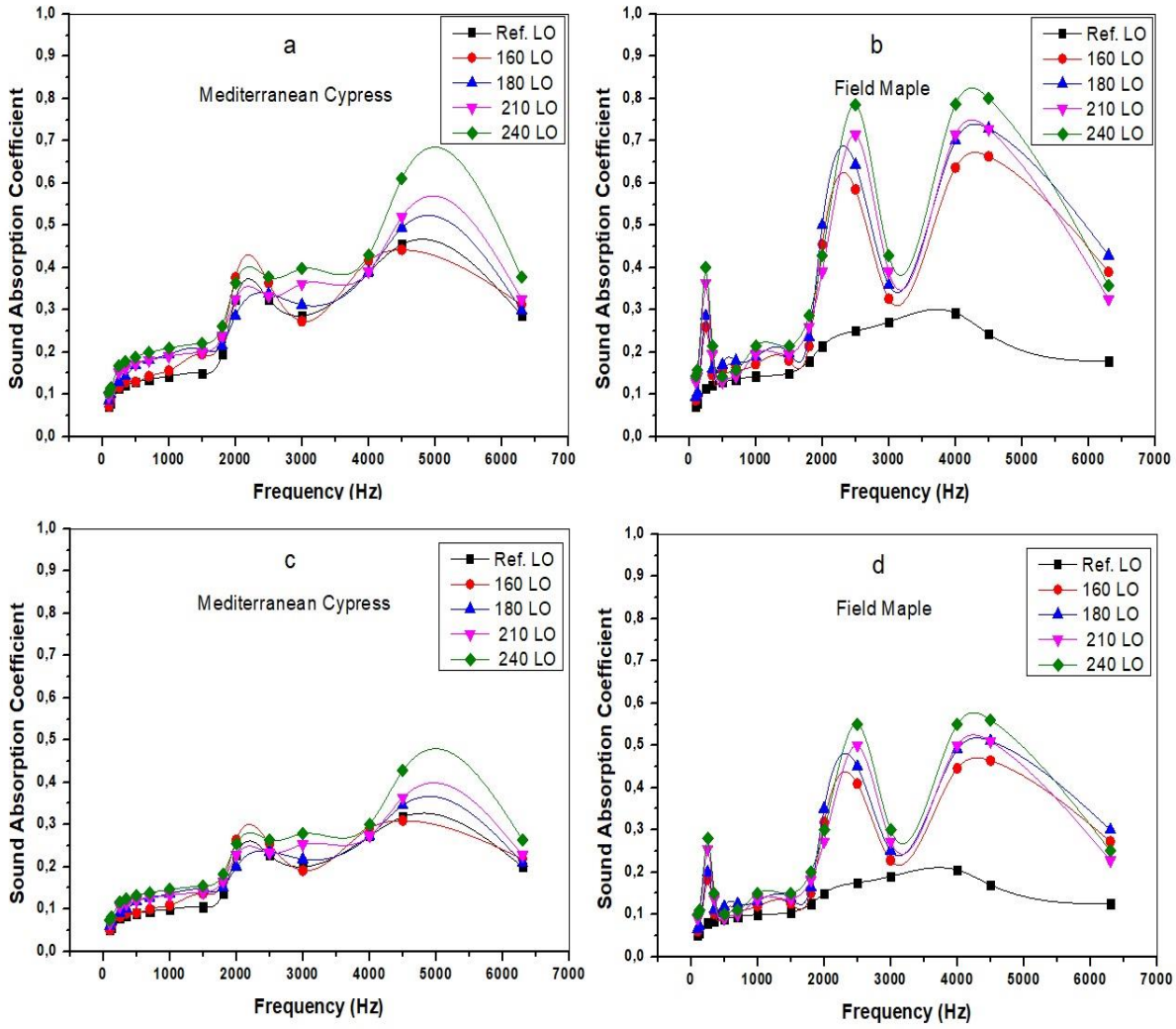

Fig. 9. The SAC data of the Mediterranean cypress wood and the field maple wood samples after impregnation with LO and after HT, longitudinal direction (a-b), tangential direction (c-d)

The SAC values in the longitudinal direction of the heat-treated LO impregnated field maple wood samples are given in Fig. 9b. The sound absorption of LO impregnated samples was observed to increase with the increase in the temperature. The two peak points for the SAC values, which were 0.78 and 0.80, were observed at 2500 and 4500 Hz, respectively. The average SAC value was found to increase with the temperature in the treated samples. For the samples processed at 240 °C, the SAC was found to be 0.37 on average. For the heat-treated Mediterranean cypress wood, the highest peak for the SAC in the tangential direction, which was 0.51, was observed at 5200 Hz (Fig. 9c). The average SAC value for the LO impregnated samples and the heat-treated field maple wood samples in the tangential direction was determined to be 0.19. The results of the SAC measurements showed two peaks, which were 0.58 and 0.59, at 2200 Hz and 4450 Hz, respectively (Fig. 9d).

This was realized in different ways in the low and high frequency bands. In the low frequency band the increase in the SAC values was slow. The low frequency band (0 to 2000 Hz) was affected by the temperature increase in the wood. The increase in the high frequency band (2000 to 6000 Hz) was faster but occurred in a narrow band range. Increase

in the temperature had prevented the collapse in the cell walls and the resulting porous structure might have prevented the sound waves. Then, continued sound absorption can be explained as being due to an increase in surface area.

Sound Transmission Loss (STL)

When sound waves hit the material surface, some of the sound energy generated on the surface is absorbed, some is reflected, and the rest is transmitted by the surface. Sound transmission loss (STL) indicates the sound blocking capacity of the material. From an architectural point of view, the sound blocking capacity of the material is considered as an indicator of the sound insulation of the material. Depending on the frequency, the high sound transmission loss means that the sound insulation effect is also high (Lee *et al.* 2011; Kim *et al.* 2015).

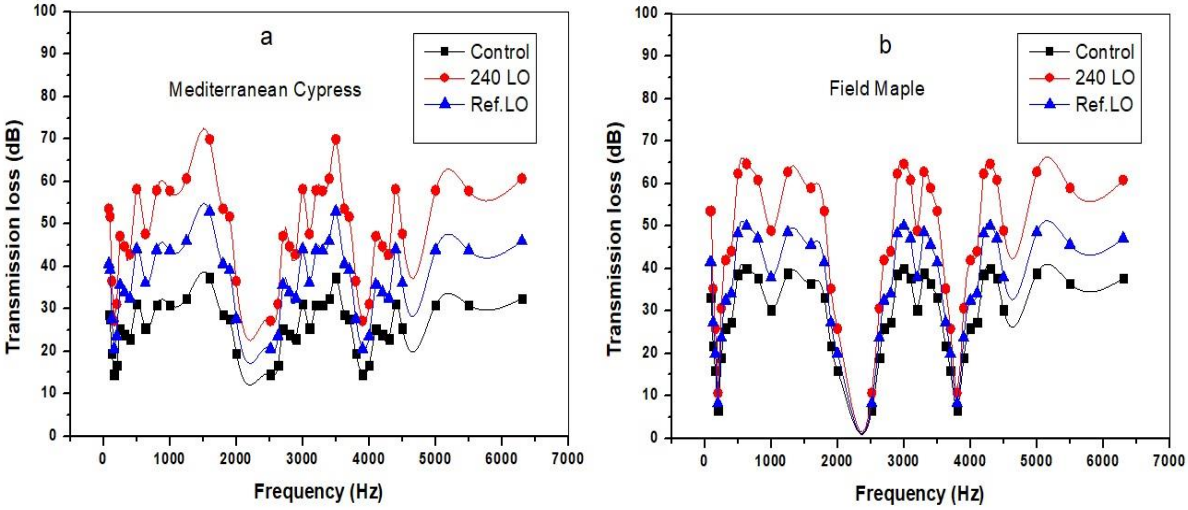

Fig. 10. The STL data of the Mediterranean cypress wood and the field maple wood, longitudinal direction (a), tangential direction (b)

Figure 10a shows the STL values of the Mediterranean cypress wood samples before (control) and after impregnation with LO in the frequency range 100 to 6300 Hz. The average STL of the Mediterranean cypress wood was found to be 48.6 dB. The average STL for the control (non-impregnated) group was 26 dB and the highest STL at 3500 Hz was 37.4 dB. In Fig. 9a, the highest average STL, which was 48.6 dB, was found with the group impregnated with LO. In this group, the highest STL value, which was 69.9 dB, was observed at 1540 Hz and 3600 Hz.

In Fig. 10b, the average STL values for the field maple wood was observed to be 37 dB and for the control group it was found to be 28 dB. In this group, the highest STL value, which was 64.7 dB, was observed at 640, 1100, and 4100 Hz.

When the average values were considered, it was observed that as porosity increased, the STL values for the Mediterranean cypress and field maple wood increased by 86% and 32%, respectively. Kang *et al.* (2019) obtained similar results in their study. They observed that for a 20 mm thick non-treated *Paulownia tomentosa* wood the average sound loss in the range 50 to 6400 Hz was 35 and 36.9 dB. Despite its low specific gravity and high porosity, the sound transmission loss for *Paulownia tomentosa* wood was high.

CONCLUSIONS

1. Compressive strength (CS) increased with increasing heat treatment temperatures. However, as the temperature increased, the compressive strength tended to decrease. Compressive strength (CS) increased with increasing heat treatment temperatures in impregnated heat treatment. The flexural strength (MOR) and flexural modulus of elasticity (MOE) decreased due to heat treatment. However, the flexural strength (MOR) flexural modulus of elasticity (MOE) increased due to impregnated heat treatment.

2. Density values decreased with increasing heat treatment temperature.

3. The percentage of average water absorption decreased significantly as the temperature increased. The lowest water absorption was observed for the samples impregnated at 240 °C. Water absorption was observed to decrease significantly as the temperature increased from 160 to 240 °C in heat treatment for each sample with and without impregnation.

4. Porosity decreased very little after the impregnation; in the HT, the increase in temperature had a positive affect (max. 35.3%).

5. For the wood samples, the average sound sorption coefficient in the longitudinal direction before impregnation (control) and after impregnation with LO were 0.19, and 0.28, respectively. In the tangential direction after impregnation with LO, it was found to be 0.12 and 0.17, respectively. The increase in temperature increased sound absorption in samples impregnated with LO. Two peak points were found as sound absorption coefficients which were 0.78 and 0.80 at 2500 Hz and 4500 Hz, respectively.

6. The highest STL was found at 640 Hz, 1100 Hz, and 64.7 dB at 4100 Hz in the group impregnated with LO.

7. The disadvantages that arise during use of wood samples were minimized *via* impregnation with LO and with TM. Thus, besides achieving ideal sound absorption it also attains properties of being durable, water-repellent, and dimensionally stable material during the desired service time. LO impregnated and the heat-treated wood can be used as natural, decorative, and environmentally sensitive wood material for sound insulation both indoors and as well as outdoor applications.

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