THE EFFECT OF DENSIFICATION TEMPERATURE ON SOME PHYSICAL AND MECHANICAL PROPERTIES OF SCOTS PINE (*PINUS SYLVESTRIS* L.)

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As wood's density increases, strength properties tend to increase due to a decrease in cavity volume. This study aimed to determine the effect of temperature levels in the densification process with an open-system thermomechanical method on the density, bending, modulus of elasticity in bending, compression, shear strength, and Brinell hardness in radial/tangential directions of Scots pine. The densification process significantly increased the strength properties of Scots pine. This increase stemmed from the decrease in the rate of cavities with the densification process, which also resulted in an increase in cell wall elements that have load-bearing properties per unit volume. An increase in densification temperature decreased strength properties. The decrease in the strength values can be explained by increasing chemical degradation with a rise in the temperature level. The most suitable temperature level was 120°C for a higher bending, shear, and compression strength, and it was 140°C for a higher radial and tangential hardness in the densification of Scots pine. Increases of 42% in the bending strength, 20% in the shear strength, 47% in the compression strength, 242% in the radial hardness, and 268% in the tangential hardness were obtained after densification.

Keywords: Densification; Scots pine; Thermal process; Mechanical properties of wood

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INTRODUCTION

The native range of Scots pine extends from Great Britain and Spain east through Siberia, south to the Caucasus region and north to Lapland. Scots pine has been widely planted in New Zealand and the colder regions of North America. It is naturalized in the US Northeast, Midwest, and Pacific Northwest. Scots pine is the only pine native to northern Europe and once formed much of the Caledonian Forest of the Scottish Highlands. In its northern distribution, it ranges from sea level to 900 m; in its southern distribution, Scots pine is a high-elevation tree growing at altitudes from 1200 to 2500 m (Geils *et al.* 2009). The extent of Scots pine forests exceeds 28 million hectares, representing at least 20% of the commercial forest area of the EU. This is about 12 percent of the world distribution of the species. Nearly 80 percent of this area is to be found in two Scandinavian states (Sweden and Finland). Germany, France, and Spain also have substantial areas of Scots pine forests in excess of 1 million hectares (Mason and Alia 2000). Scots pine occupies about 1,038,435 ha (5 % of total forest area) in Turkey, growing mainly in the Black Sea coastal mountains on warm southern slopes, where the climate is humid (Turna and Güney 2009).

Densification of wood

The densification of wood is defined as a process involving compression by pressing heated wood. Densification is often used in combination with an impregnation process, whereby the cell lumens are filled with a liquid material (polymer, melted natural resins, sulfur, melted metal, *etc.*) (Kollmann *et al.* 1975; Kultikova 1999; Kutnar and Sernek 2007). Since increasing the density of wood improves its strength properties, there have been many studies on the densification of wood. By densification, a wood with low density can attain the strength properties of a wood material that has a higher density, and the applications and objectives of use change accordingly.

The species of wood, the temperature and period of softening or plasticizing, the densification method (thermo-mechanical, thermo-hydro-mechanical, and viscoelastic thermally compression), and the compression pressure and period are the most important variables that influence the strength of wood after densification. Application of these variables in a different manner can increase the strength properties of the densified wood materials at a rate reaching 100% (Seborg *et al*. 1945; Stamm *et al*. 1955; Kennedy 1968; Kunesh 1968; Trenard 1977; Hillis and Rozsa 1978; Bucur *et al.* 2000; Morsing 2000; Santos 2000; Navi and Girardet 2000; Lenth and Kamke 2001; Reiterer and Stanzl-Tschegg 2001; Tabarsa and Chui 2001; Blomberg *et al.* 2005; Kamke and Sizemore 2005; Kutnar and Sernek 2007; Kocaefe *et al*. 2008a,b; Korkut and Kocaefe 2009; Kutnar and Kamke 2012).

Many physical and mechanical properties of wood are affected when it is heated. As a result of heating, wood is momentarily transformed into a soft and rubbery structure from a stiff and glassy material. At short times, low temperatures, and low moisture contents, wood exhibits glassy behavior. At long times, high temperatures, and high moisture contents, wood exhibits rubbery behavior. The transition phase and temperature occurs between these two distinct regions. The glass transition temperature is also called the softening temperature. The decrease of the moisture content of wood increases the glass transition temperature (Kutnar and Sernek 2007). The glass transition temperature can also change according to the wood species (Calonega *et al*. 2010).

The higher the temperature or the longer the treatment time, the more significant modification in wood is obtained. Based on the temperature and treating time, the process of heat treatment reduces the bending and tensile strength and makes wood more brittle. The brittleness of treated wood is attributed to the changes in viscosity and plasticity rather than in elasticity (Bekhta and Niemz 2003; Kubojima *et al*. 2000; Kubojima *et al*. 2000; Jiang *et al.* 2009). In general, both temperature and moisture content play leading roles in influencing mechanical properties of wood. Under the fiber saturation point, as the moisture content and temperature decrease, wood strength increases (Gerhards 1982).

Alen *et al.* 2002 states that at temperatures above 100 $^{\circ}$C, the intermolecular and intramolecular chemical bonds begin to break with a rate that would intensify as the heating time increases. Furthermore, this phenomenon could be due to the thermal softening and loss of amorphous polysaccharides, which are responsible for a tight combination of cellulosic fibers and the amorphous matrix including lignin. Bond breakage can also be attributed to the possibility of lignin relocation. Treatment at 160 $^{\circ}$C is probably enough to cause lignin molecules, which is located between the fibril aggregates, to change their position and damage the adhesive linkage of lignin with cellulose fibrils (Jiang *et al.* 2009).

As can be observed from the references cited, the temperature level in heating and densification is one of the important parameters. This study aims to increase the density of Scots pine (*Pinus sylvestris* L.) wood, which is one of the relatively soft (relatively low density) wood species, by pressing thermomechanically with a fixed pressure and

moisture content and different temperatures. This process can achieve physical and mechanical performance levels corresponding to medium density woods, such as oak, beech, and chestnut. This study also aims to determine the effect of densification temperature on physical and mechanical properties of Scots pine. Thus, since a less expensive wood material could be used instead of a more expensive wood material, it would form an economic alternative for the production of furniture and wooden structures. Furthermore, since the hardness of wood is increased to a significant degree by densification, it will be possible to use softwoods as floor coverings, and densified hardwoods could be used with even greater reliability. As the strength properties are expected to be much better, it is also possible to use the material obtained as bearing building materials such as columns and joists.

EXPERIMENTAL

Materials

Scots pine

In order to determine the effect of the temperature level applied in the densification with the thermomechanical method on some physical and mechanical properties, Scots pine (*Pinus sylvestris* L.) was selected as a wood species due to its relatively low density, the fact that it is one of the most widely distributed conifers in the world, and its widespread use in the wood products industry.

The Scots pine wood used for obtaining the test specimens was procured from the Siteler district in Ankara/Turkey. Importance was placed on selecting lumber that was first grade in quality and undamaged, and was naturally colored. Selected lumber had annual rings that were regular and parallel to each other and did not include defects and had not been subjected to attack from insects or fungi.

Densification press

A 100 ton Hursan brand laboratory press was used with a capacity to heat up to 250 $^{\circ}$C. The press was equipped with a plate surface of 60 x 60 cm^2 for the densification process of the Scots pine with an open system and thermomechanical method. Pressure and temperature of plates and stocks could be controlled and monitored automatically.

Preparation of the stocks for the densification process

The process given below was followed in the preparation of the test stocks that would be used in the densification process:

1. The lumber procured was stacked and kept in a manner suitable to the principles of air drying conditions in a ventilated and central heating system environment that did not receive direct sunlight.
2. Stocks were cut from the lumber in the dimensions of 560 x 80 x 60 mm (length x width x thickness) in order to conform to the measurements specified in the standards for the tests that would be conducted. The stocks were stacked and left to dry naturally according to standards.
3. The stocks were brought to the finished dimensions of 550 x 70 x 50 mm (length x width x thickness) with the cutting and planning processes.

4. The stocks were kept in a climatization chamber at a temperature of 20±2°C and a relative humidity of 65%±5% until they reached unchanged masses in order for them to reach a 12% air-dried moisture level .
5. The stocks were wrapped with plastic sheets to prevent a change in moisture content that could occur prior to the densification process.

Method

Densification

The following process was applied for the densification of Scots pine with the open system thermomechanical method:

1. The press was operated and the thermostats were adjusted so that the platens of press reached the planned temperature with a sensitivity of ±5°C.
2. The stocks were placed on the lower platen in a manner so that the pressure would be applied in a radial direction. Furthermore, in order to check if the internal temperature of the stocks had reached the planned temperature, three temperature control stocks with thermometer were placed in a manner so that they would reach both sides and the middle of the lower platen of the press. The thermometers were in the middle and side parts of the stocks.
3. In order to ensure heat transfer to both sides, the upper flat surface was contacted on the stock surfaces without applying pressure.
4. The stocks were kept until they reached the planned internal temperature values of 120 °C, 140 °C, and 160 °C. The internal temperatures of the stocks were monitored from the temperature control stocks with a thermometer.
5. The pressure gauge of the press was adjusted to 6 MPa providing a densification ratio of 50 %. The stocks were densified by being compressed with a 3 m/minute loading speed under automatic control. Thicknesses of the stocks were decreased from 50 mm to 25 mm after the densification process.
6. The press was kept closed for 10 minutes by preserving the pressure in order to prevent an increase in volume of the stocks with the spring effect. Residence time of 10 minutes after densification was the most appropriate time for less spring-back effect, as found in preliminary tests
7. The stocks were removed from the press and kept in a closed environment for a period of 10 days.
8. Average moisture content of the stocks (4%) was determined according to TS 2471.

Preparation of the specimens for the physical and mechanical tests

The following process was applied in the preparation of the specimens for the physical and mechanical tests:

1. The stocks on which the densification process was implemented were machined into specimens having the measurements and numbers specified in Table 1 in conformance to the general principles in the TS 2595, TS 3459, TS 2474, and TS 2479 standards related to the determination of the compression, shear and bending strengths, and the Brinell hardness values. The preparation of specimens complied with the principles stated in the TS 2470 standards.

Table 1. Measurements and Numbers of Specimens According to the Tests

Tests	Measurement of Specimen (mm)	Number	
		Densified	Control
Density	20 x 20 x 20	24	24
Compression Strength	30 x 20 x 20	18	6
Shear Strength	55 x 50 x 22	18	6
Bending Strength	360 x 20 x 20	18	6
Brinell Hardness	30 x 20 x 20	18	6

2. The average moisture content of the specimens after densification process was 4%. The specimens were kept in a Termal A11680F brand climatization chamber at a temperature of 20 ± 2 °C and a relative humidity of 65% ± 5% until they reached unchanged masses, such that they attained a 12% air-dried level of moisture content.
3. The specimens were wrapped with plastic sheets to prevent a change in moisture content that could occur before the tests were made.

Physical and Mechanical Tests

Moisture content

The moisture contents of the specimens were determined according to TS 2471(1976). Each specimen was weighed. After weighing, the specimens were placed in an oven heated to 103 ± 2 °C, and they were kept there until no appreciable weight change occurred in 4-hour weighing intervals. The oven-dry mass and the mass of the specimen when cut were used to determine the percentage of moisture content using following formula:

Moisture Content (%) = [(Mass when cut – Oven-dry mass) / Oven-dry mass] x 100

Density

The density of Scots pine on which the densification process was implemented or not implemented (control) was determined in accordance with the TS 3459 (2012) standard. The density value is defined as the ratio of the oven-dry mass of a specimen to the volume of the specimen at the oven-dry moisture.

Bending strength (modulus of rupture) and modulus of elasticity

Bending strength and modulus of elasticity (MOE) values of the specimens were determined according to the procedures described in TS 2474 (1976) standard. The test specimen was simply supported and applied by concentrated load. Observation of both load and deflection was made with a Universal Mechanical Test machine until failure occurred. The following equations were used to calculate bending strength and modulus of elasticity,

$$\text{MOE} = P L^3 / 4bd^3\Delta \qquad (\text{N/mm}^2)$$

where MOE is the modulus of elasticity, P is the load at some point below the proportional limit, L is the distance between supports for the specimen (mm), b is the specimen width (mm), d is the thickness (depth) of the specimen (mm) and Δ is the deflection (in mm) corresponding the load P, and where MOR is the bending strength (or modulus of rupture):

$$MOR = 3PL / 2bd^2 \qquad (N/mm^2)$$

Shear strength

Shear strengths of the specimens were determined according to the procedures described in the TS 3459(2012) standard. The specimen simply supported to the Universal Mechanical Test Machine and shearing load was applied to the specimens. The load at the moment of separation of the specimen parts from each other was observed and recorded. The shear strength of the each specimen was calculated by using following formula,

$$\sigma_m = P_{max} / A \qquad (N/mm^2)$$

where σ_m is the maximum shear strength, P_{max} is the maximum load, and A is the bond surface area.

Compression strength

Compression strength values were determined according to the procedures described in TS 2595(1976) standard. The specimen was subjected to an axial compressive load applied at its ends until failure occurred. The load at the moment of failure was observed and recorded. The compression strength of the each specimen was calculated by using the following formula,

$$S = P_{max} / A \qquad (N/mm^2)$$

where S is the compressive strength, P_{max} is the maximum load applied to the specimen, and A is the cross-section area of the specimen.

Hardness

The Brinell hardness values of the specimens were determined according to the procedures described in the TS 2479(1976) standard. The surface of the specimen was indented with a 10 mm diameter hardened steel ball subjected to a load of 5000 N for 15 seconds. The diameter of the indentation left in the specimen was measured with a microscope. The Brinell hardness number was calculated by dividing the load applied by the surface area of the indentation.

Experimental data analysis

The data obtained with the testing of the specimens according to different variables were statistically analyzed. The multivariance analysis was applied for testing whether or not the densification temperature level was effective relative to the selected physical and mechanical properties of Scots pine. At the end of this analysis, in case the p-value obtained was <5%, then the densification temperature was judged to be effective with respect to the variable; otherwise, it was judged to be ineffective. The Homogeneity Test was applied to determine whether or not the differences among the variables were significant, and the results were interpreted. The SPSS (Statistical Package for the Social Science) package program was used for analysis.

RESULTS AND DISCUSSION

Physical and mechanical properties obtained at the conclusion of the tests made on densified Scots pine in connection with the method explained above are given in Table 2.

Table 2. Some Physical and Mechanical Properties of Densified Scots Pine at a Pressure of 6 MPa and Different Temperature Levels

Specimens	Statistical Value	Density (gr/cm^3)	Bending Strength (N/mm^2)	Modulus of Elasticity (N/mm^2)	Shear Strength (N/mm^2)	Compression Strength (N/mm^2)	Hardness in Radial Direction (N/mm^2)	Hardness in Tangential Direction (N/mm^2)
Control (Undensified)	Average	0.43	69.13	8444.80	8.82	46.55	1.10	1.00
	Standard Deviation	.01	1.75	426.15	.74	1.51	.07	.06
	Variation Coefficient	1.23	2.53	5.05	8.43	3.23	6.28	6.00
Densified at 120 °C	Average	.79	98.55	9405.04	10.58	68.42	2.53	3.00
	Standard Deviation	.07	24.22	2025.39	0.95	11.41	.23	.43
	Variation Coefficient	8.46	24.58	21.54	8.99	16.68	9.02	14.46
Densified at 140 °C	Average	.81	94.69	9197.97	10.34	61.03	3.77	3.68
	Standard Deviation	.02	2.91	694.65	0.33	3.12	.41	.44
	Variation Coefficient	2.05	3.08	7.55	323	5.12	10.92	11.84
Densified at 160 °C	Average	.80	64.93	8336.42	8.34	57.45	2.55	2.48
	Standard Deviation	.02	1.60	461.51	1.29	4.93	.09	.34
	Variation Coefficient	2.81	2.46	5.54	15.43	8.58	3.37	13.74

The analysis of variance related to whether or not the temperature level applied in densification was effective relative to the density bending strength, modulus of elasticity, shear strength, compression strength, and hardness in radial and tangential directions is given in Table 3.

Table 3. Analysis of Variance Related to the Effect of the Temperature Level on Some Physical and Mechanical Properties of Scots Pine

Variables	p-Value	Significant ($p< .05$)	Insignificant ($p>.05$)
Density	.000	X	
Bending Strength	.000	X	
Modulus of Elasticity	.28		X
Shear Strength	.001	X	
Compression Strength	.000	X	
Hardness In Radial Direction	.000	X	
Hardness in Tangential Direction	.000	X	

Table 3 shows that the temperature level affected the density, bending strength, shear strength, compression strength, and hardness in radial and tangential directions of Scots pine ($p< 0.05$). But the temperature level did not affect the modulus of elasticity, since the p-value was larger than 0.05 ($p>0.05$).

Table 4 shows the homogeneity tests results related to the temperature levels that created a difference according to the values of density, bending strength, shear strength, compression strength, and hardness in radial and tangential directions.

Table 4. Homogeneity Tests Related to the Determination of the Temperature Levels that Create a Difference According to the Values of Density, Bending Strength, Shear Strength, Compression Strength, and Hardness in Radial and Tangential Directions

Variables	Densification Temperature Values (^{o}C)	Number of Specimens	Homogeneity Group			
			4	3	2	1
Density	160	6			.80	
	120	6			.81	
	140	6			.81	
	Control	6				.42
Bending Strength	160	6			64.93	
	Control	6			69.12	
	140	6				94.69
	120	6				98.54
Shear Strength	160	6			8.34	
	Control	6			8.82	
	140	6				10.34
	120	6				10.58
Compression Strength	Control	6		46.55		
	160	6			57.45	
	140	6			61.03	61.03
	120	6				68.42
Hardness Value in Radial Direction	Control	6		1.10		
	120	6			2.53	
	160	6			2.56	
	140	6				3.77
Hardness Value in Tangential Direction	Control	6	1.00			
	160	6		2.48		
	120	6			3.00	
	140	6				3.68

After the densification process, the density of Scots pine increased from 0.42 g/cm^3 to 0.81 g/cm^3 at all the temperature levels, and a 93% increase in density was realized. Since the control specimens and densified specimens appeared in separate homogeneity groups, the densification process was judged to be significantly affected by density (Table 4). However, since the differences among the density values of the densified specimens heated to 120 °C, 140 °C, and 160 °C were not significant, it was found that the different temperature levels did not increase density at the 6 MPa pressure level. The fact that the densities were the same at all temperature levels likely stemmed from the Scots pine having reached a maximum compressible point upon application of a sufficiently high compression level (6 MPa). Low densities could emerge at low pressures because the plasticity of wood is different at different temperature levels.

The difference between the bending strength (69.12 N/mm^2) of the undensified Scots pine and the bending strength (64.93 N/mm^2) of the densified Scots pine at a temperature of 160 °C was not significant. Consequently, the densification carried out at 160 °C did not produce an increase in bending strength. In addition, the bending strengths (98.54 N/mm^2 and 94.69 N/mm^2) between densified Scots pine at temperatures of 120 °C and 140 °C were not significant (Table 4). From this, the conclusion emerges that the densification process increases the bending strength, but an increase in the temperature level causes a decrease in bending strength.

The shear strength (8.82 N/mm^2) of undensified Scots pine was not significantly different from the shear strength (8.34 N/mm^2) of densified Scots pine at a temperature of 160 °C. Consequently, the densification made at 160 °C did not affect shear strength. The differences in the shear strengths (10.58 N/mm^2 and 10.34 N/mm^2) between densified Scots pine at temperatures of 120°C and 140°C were not significant (Table 4). From this, the conclusion emerges that the densification process increases shear strength, but an increase in the temperature level is the cause of a decrease in the shear strength.

Since the lowest compression strength (46.55 N/mm^2) was obtained in the undensified Scots pine, the densification process increased compression strength. The differences between the compression strengths (61.03 N/mm^2 and 57.45 N/mm^2) of the densified Scots pine at 140 °C and 160 °C and the compression strengths (68.42 N/mm^2 and 61.03 N/mm^2) of the densified Scots pine at 120 °C and 140 °C were not significant (Table 4). It can be concluded that it is necessary to densify Scots pine at temperatures of 120 °C or 140 °C for higher compression strength.

Since the hardness values (1.10 N/mm^2 and 1.00 N/mm^2) of the undensified Scots pine in the radial and tangential directions were the lowest, the densification process increases the hardness of Scots pine. The highest hardness values (3.77 N/mm^2 and 3.68 N/mm^2) were obtained in the densified specimens at 140 °C, both in the radial and tangential directions. The differences between the hardness values (2.56 N/mm^2 and 2.53 N/mm^2) in a radial direction of the densified Scots pine at 160 °C and 120 °C were not significant. The second highest hardness value (3.00 N/mm^2) in a tangential direction was obtained in the specimens densified at 120 °C. The lowest hardness value in a tangential direction emerged at 160 °C in the densified specimens (Table 4).

The increase of temperature in the densification process affected all of the strength properties of Scots pine. The increase of the densification temperature level from 120 °C to 140 °C in the Scots pine did not affect the bending strength (98.54 N/mm^2 and 94.69 N/mm^2), shear strength (10.58 N/mm^2 and 10.34 N/mm^2), and compression strength (68.42 N/mm^2 and 61.03 N/mm^2). However, it caused an increase in the hardness in a radial direction from 2.53 N/mm^2 to 3.77 N/mm^2 and in the hardness in a tangential direction from 3.00 N/mm^2 to 3.68 N/mm^2. The raising of the densification temperature to 160 °C decreased the bending strength value to 64.93 N/mm^2 , the shear strength value

to 8.34 N/mm^2, the compression value to 57.45 N/mm^2, the radial hardness value to 2.56 N/mm^2, and the tangential hardness value decreases to 2.48 N/mm^2. In this situation, when comparing the densification made at 120 °C, there was a 34% decrease in the bending strength, 21% decrease in the shear strength, and 16% decrease in the compression strength. A 32% decrease was observed in the radial and tangential hardness values with an increase in the densification temperature from 140 °C to 160 °C. In the densification made at 160 °C, the bending and shear strength values for Scots pine decreased to the same levels of bending and shear as undensified Scots pine (69.13 N/mm^2 and 8.82 N/mm^2). Consequently, the densification process applied at 160 °C became ineffective. In conclusion, the most suitable temperature level was judged to be 120 °C for a higher bending, shear, and compression strength in the densification of Scots pine, and it was judged to be 140 °C for a higher radial and tangential hardness. Under these conditions, an increase of 42% in the bending strength, 20% in the shear strength, 47% in the compression strength, 242% in the radial hardness, and 268% in the tangential hardness could be obtained after densification. This increase stems from the decrease in the amount of cavities with the densification process, which also results in an increase in cell wall elements that have load-bearing properties per unit volume.

The improvement of the strength properties with the densification process gives the possibility of bearing a heavier load with the same sectional area of wood or bearing the same load with a thinner section in buildings. Furthermore, the large increase in hardness will provide for even softwoods to be used as flooring after densification.

CONCLUSIONS

1. The densification process with a pressure of 6 MPa and temperatures of 120 °C, 140 °C, and 160 °C were found to affect the physical and mechanical properties of Scots pine, except for the modulus of elasticity in bending.
2. The temperature level for densification with a pressure of 6 MPa did not affect the density of densified Scots pine. After the densification process, the density of Scots pine increased from 0.42 g/cm^3 to 0.81 g/cm^3 at all the temperature levels, and a 93% increase in density was realized.
3. The increase of temperature in the densification process affected all of the strength properties of Scots pine. An increase in densification temperature decreased strength properties.
4. In the densification at 160 °C, the bending and shear strength values for Scotch pine decreased to the same levels of bending and shear strengths as undensified Scots pine (69.13 N/mm^2 and 8.82 N/mm^2). Consequently, the densification process applied at 160 °C was ineffective.
5. The most suitable temperature level was found to be 120 °C for a higher bending, shear, and compression strength, and it was 140 °C for a higher radial and tangential hardness in the densification of Scots pine. Under these conditions, an increase of 42% in the bending strength, 20% in the shear strength, 47% in the compression strength, 242% in the radial hardness, and 268% in the tangential hardness could be obtained after densification.

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