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Effect of Heat Treatment on Wettability and Modulus of Elasticity of Pine and Spruce Wood

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This research was performed in order to determine how the heating process affects the wettability and mechanical properties of spruce (*Picea abies*) and pine (*Pinus silvestris*) wood. Studies were carried out using wood heated in laboratory. Specimens were cut out of planed beams. Then specimens were divided into the following four groups: specimens of one group were not exposed to heating, whereas specimens of three other groups were subjected to heating at the temperature of 190 °C for 1 to 3 hours respectively, in the air under atmospheric pressure. Both heated and unheated specimens were moistened and dried in a climatic chamber. Before and after treatment the mechanical properties of specimens were assessed using the original method of transverse vibrations and contact angle measurements were carried out using the sessile drop technique. The results showed a significant increase in wood hydrophobicity after treatment. Spruce contact angle after treatment increased from 1.3 to 1.45, pine from 1.4 to 2 times. Modulus of Elasticity (MOE) of pine wood decreased, while MOE of spruce slightly increased after heat treatment.

Keywords: heat-treated wood, pine, spruce, sorption, mechanical properties, nondestructive evaluation, wettability.

1. INTRODUCTION

Wood is a natural, renewable and environmentally friendly raw material, which consists mainly of cellulose, hemicellulose, lignin and extractive substances. It is one of the strongest and most widely used organic materials. Wood is easily processable and important material in furniture and construction industry. However, wood exhibits hygroscopic properties and its cell walls contain polymers with hydroxyl groups. Therefore, in the humid environment dry wood begins to moisten and in the dry environment damp wood starts to dry until it reaches its equilibrium moisture content. Thus, as a result of atmospheric humidity and other ambient parameters, wood moisture and dimensions begin to change [1 – 3].

Wood hygroscopicity can be reduced in different ways and one of them is thermal wood processing which involves the use of only three components, such as water, steam and high temperature, and, therefore, makes heated wood an eco-friendly alternative to chemically impregnated wood. Wood undergoes irreversible chemical changes due to the effect of temperature: the heat affects cell wall polymers and contributes to the destruction of polymer chains and the decrease in free OH groups. Consequently, excessive moisture absorption is prevented, biological resistance is improved and durability is enhanced [4 – 7].

However, temperatures above 150 °C have irreversible impact on not only chemical but also mechanical properties of wood. The higher the heating temperature, the worse the mechanical properties of thermally processed wood. Wood becomes more fragile and its bending and tensile strength decline by 10 % – 30 % [8 – 9].

Some earlier experiments demonstrated that heat treatment did not cause any significant changes in the values of the modulus of elasticity. In addition, it was observed that the modulus of elasticity of heat treated specimens was slightly higher than the one of untreated wood and it was determined that the transverse tensile strength of the specimens decreased by 26 % [10 – 11].

Wood is a biological material with a heterogeneous structure. During the analysis of its mechanical properties the extensive spread of data is obtained. One of the possible solutions is to use a large quantity of specimens and to subject data to statistical processing. Another solution is to apply non-destructive testing methods for the evaluation of mechanical properties. The main advantage provided by this method is that specimens remain intact and there is no need to cut out specimens with certain dimensions. In addition, the use of dynamic methods for the analysis of specimens allows quite accurate determination of their modulus of elasticity [12 – 13].

Many earlier experiments show that heat treatment reduces the equilibrium moisture content and slows down the water absorption and wettability of wood [1]. For example, contact angle measurements before and after 240 °C treatment observed a clear decrease in wettability for treated wood. Advancing contact angles of a water drop were in all cases systematically higher for heat-treated than for untreated wood. It was suggested, that the change in wettability might be due to the modification of conformational arrangement of wood biopolymers as a result of residual water or plasticization of lignin [14 – 17].

The purpose of this study was to establish the effect of heating on the mechanical and sorption properties of spruce and pine wood specimens.

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2. MATERIALS AND METHODOLOGY

The studies were carried out using spruce and pine wood specimens cut out of planed beams with the following measurements: 315 x 20 x 20 mm. The beams were cut from trees at heights between 2 and 5 m. Specimens were divided into three groups (1, 2, 3) and heat-treated at the 190 °C temperature, in the air under atmospheric pressure for 1, 2 and 3 h respectively. Each group contained 20 psc. of specimens. Before the beginning of the heating process specimens underwent conditioning in a chamber at the 25 °C ± 1 °C temperature and 35 % ± 1 % relative humidity for 10 days. The moisture content of specimens was established using the weighing method [18]. After the completion of the heating process all the specimens were conditioned in the chamber under the same conditions. Before and after treatment the dimensions and mass of specimens were measured with the following accuracy: length – 0.05 mm, width and thickness – 0.01 mm, weight – 0.01 g. The special test stand was used to determine the modulus of elasticity and the damping coefficient on the basis of the non-destructive testing method, which also allowed assessing the mechanical properties of specimens. The modulus of elasticity *E* was calculated based on the following formula [19, 20]:

$$E = \frac{f_{rez}^2 4\pi^2 \rho s l^4}{IA^2}, \tag{1}$$

where E is the modulus of elasticity, f_{rez} is the frequency of transverse vibrations, ρ is the density of wood, s is the cross-sectional area, l is the beam length, I is the cross-sectional moment of inertia, A is the representing method of fastening coefficient.

The measurements of the contact angles of specimens were carried out applying the sessile drop method [21]. The values of the contact angles were determined by using a digital camera in combination with image analysis software. The selected liquid was distilled water. The contact angles between each droplet and specimen surface were measured both on the right and the left side of the droplet and the mean contact angle was calculated. The image was captured immediately after the 10 µL droplet was placed on the specimen surface, and then after 5 and 10 seconds other images were taken. Prior to contact angle measurements, for a ten day period all the specimens underwent conditioning in a chamber at the 40 °C temperature and 30 % relative humidity. Initially the contact angle of unheated specimens was measured and subsequently after the completion of the heating process it was repeatedly measured for the same specimens. Three tests were performed for each specimen in order to take into account the non homogeneous nature of wood and the mean of results was calculated.

After the all experiments statistical analysis of data was performed [22].

3. RESULTS

In order to determine the mechanical properties of specimens before and after exposure to heat treatment, the modulus of elasticity was assessed. It was established that prior to the heating process the dynamic modulus of elasticity of pine specimens was 10200 – 10521 MPa, whereas in the case of spruce specimens it was 8096 – 8416 MPa (Fig. 1 – Fig. 2).

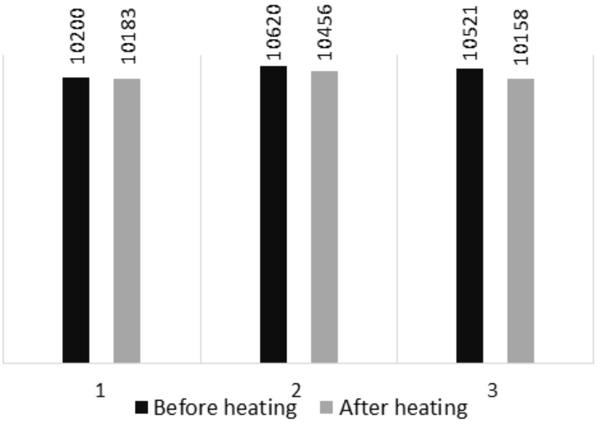

Fig. 1. The modulus of elasticity of pine specimens: 1 – 1 h heating; 2 – 2 h heating; 3 – 3 h heating

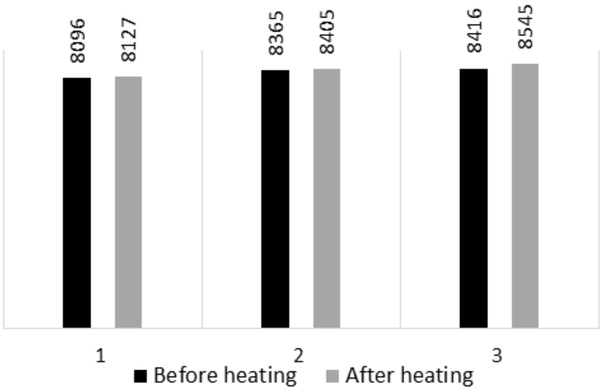

Fig. 2. The modulus of elasticity of spruce specimens: 1 – 1 h heating; 2 – 2 h heating; 3 – 3 h heating

Table 1 provides the standard deviations of the modulus of elasticity of heated and unheated specimens. The obtained results demonstrate reliable data and the dispersion of results obtained before and after exposure to heat treatment was not so wide.

Table 1. The standard deviations of the modulus of elasticity

Standard deviation, MPa				
Group	Spruce		Pine	
	Before heating	After heating	Before heating	After heating
1 group	2005	1989	1209	1196
2 group	1694	1754	1058	1052
3 group	1735	1907	856	867

There was no significant change in the modulus of elasticity of pine specimens subjected to 1-hour heating: it declined to 17 MPa on average, which is equal to 0.15 %. The similar result was obtained in the case of specimens subjected to 2 h heating: their modulus of elasticity decreased by 1.5 % and reached up to 10456 MPa. When pine specimens were subjected to 3 h heating, the modulus of elasticity declined by 3.5 % and its value was 10158 MPa.

The examination of spruce specimens revealed the opposite result: there was an increase in the modulus of elasticity of all the groups of specimens after heating. It was determined that after exposure to 1 h heating the modulus of elasticity of spruce specimens was slightly higher than it was before heating (i.e. by 0.4 %). The similar result was obtained when spruce specimens were subjected to 2 h heating: after heating their modulus of elasticity was higher than it was before heating, i.e. by 0.47 %. The greater impact of the heating process was also noticed of spruce specimens exposed to 3 h heating: after heating their modulus of elasticity was higher than it was before heating, i.e. by 1.5 %.

Table 2. The densities of specimens

Group	Density, kg/m^3			
	Spruce		Pine	
	Before heating	After heating	Before heating	After heating
1 group	473.6	474.5	563.2	561.5
2 group	475.5	475.0	565.4	558.6
3 group	478.9	476.7	562.4	551.8

It is probable that such result was obtained because of the existing dependence between mechanical properties of wood and density, moisture and other parameters. The higher the densities of wood, the better the mechanical properties of it. A decrease in the moisture content of wood leads to an increase in its strength [23]. It is known that the heating process has a greater impact on high density wood [24] and that the equilibrium moisture content of wood declines after exposure to heating [25]. In comparison to spruce wood specimens, pine wood specimens had higher density and during the heating process they exhibited the greater loss of mass and the greater decrease of density. After exposure to 3 h heating the density of spruce specimens declined only by 0.5 %. Meanwhile, after heating the density of pine specimens was lower than it was before heating, i.e. by 2 %. Thus, it can be stated that the heating process had a greater impact on pine wood specimens (Table 2).

Table 3. The equilibrium moisture contents of specimens

Group	Equilibrium moisture content, %			
	Spruce		Pine	
	Before heating	After heating	Before heating	After heating
1 group	7.54	6.84	8.43	6.13
2 group	7.54	5.66	8.43	4.23
3 group	7.54	4.79	8.43	3.07

It was found that during the heating process, spruce wood specimens demonstrated a lesser reduction in strength properties. There was also a decrease in the equilibrium moisture content (EMC), which has effect on the strength properties of wood. After heating the EMC of spruce specimens subjected to 3 h heating was lower than it was before heating, i.e. by 1.6 times, whereas in the case of pine specimens it was lower by 2.7 times. In general it can be claimed that the modulus of elasticity, which declined during the heating process, could have been compensated by the fact that after heating the equilibrium moisture content of specimens was lower than it was before heating (Table 3).

The results of contact angle measurements are provided in Fig. 3 and Fig. 4. Fig. 5 and Fig. 6 show a droplet which is placed on a spruce wood specimen before heating and after 1 h heating. It can be observed that after heating there was a considerable increase in the contact angle of both spruce and pine specimen surfaces, which means that there was a decrease in their moisture absorption. These results are in agreement with the results from the study by Esteves et al. [26] in which the wettability of pine and eucalypt decreased after thermal modification at 150 °C – 190 °C.

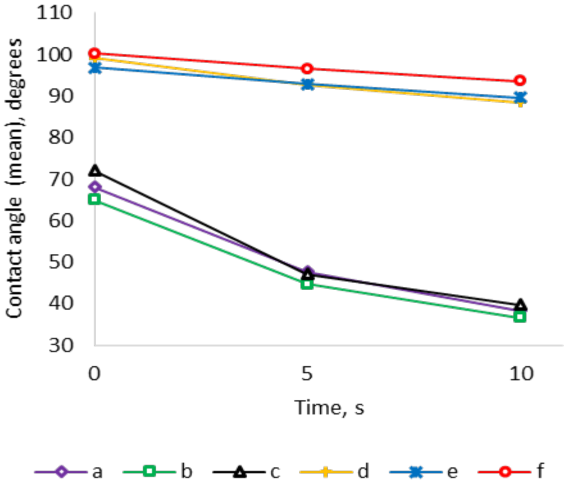

Fig. 3. The contact angle of spruce specimens: a – group 1 before heating; b – group 2 before heating; c – group 3 before heating; d – group 1 after heating; e – group 2 after heating; f – group 3 after heating

After heating the contact angle of spruce specimens, which was measured immediately after the droplet placement, varied between 100° and 109°, i.e. it was 1.4 – 1.56 times larger than the contact angle of spruce specimens before heating. The obtained results reveal that the duration of heating has no significant impact on the contact angle of specimens. Similar results were obtained by Candan et al. [27] where contact angle of untreated plywood panels were 40° which was half of contact angle value of panels treated at 190 °C.

The effect of the duration of heating becomes more noticeable when specimens are subjected to 2 and 3 h heating. After 5 s the contact angle of spruce wood specimens exposed to 1, 2 and 3 h heating decreased by 8.2 %, 3.8 % and 3 %, respectively. The results of the measurements of contact angle values after 10 s were as follows: the value of the contact angle of specimens exposed to 1-hour heating was lower by 13.3 % in comparison to the value of the contact angle measured immediately after the droplet placement. Whereas the value of the contact angle of spruce specimens subjected to 2 and 3 h heating was lower by 6 % and 6.5 %, respectively. The analysis of the contact angle data of pine wood specimens demonstrated that the values of the contact angles measured before heating ranged from 65 to 72 .

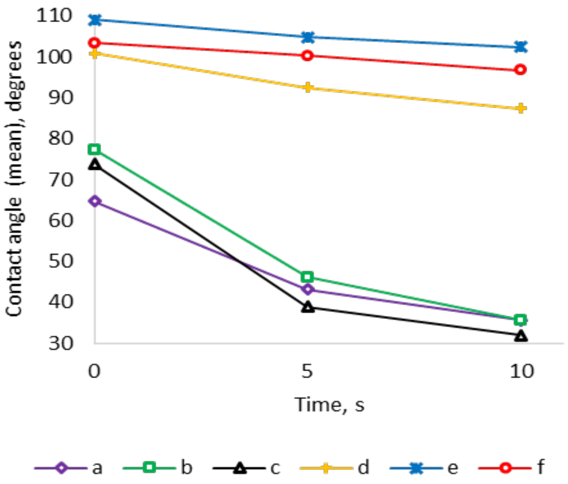

Fig. 4. The contact angle of pine specimens: a – group 1 before heating; b – group 2 before heating; c – group 3 before heating; d – group 1 after heating; e – group 2 after heating; f – group 3 after heating

The results of the measurements of the contact angle values of unheated pine specimens after 5 s were as follows: the value of the contact angle of specimens prepared to be heated for 1 h was lower by 34.4 % in comparison to the value of the contact angle measured immediately after the droplet placement. Meanwhile the value of the contact angle of pine wood specimens prepared to be heated for 2 and 3 h was lower than the value measured immediately after the droplet placement, i.e. by 31.2 % and 34.6 %, respectively.

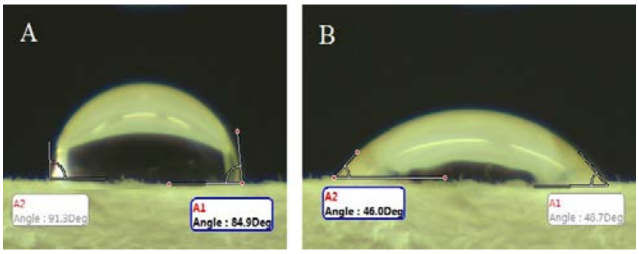

Fig. 5. The contact angle of the spruce specimen before heating. A – the contact angle measured immediately after the droplet placement; B – the contact angle after 10 s.

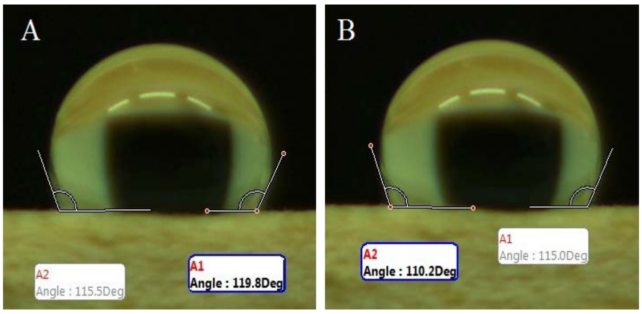

Fig. 6. The contact angle of the spruce specimen after heating. A – the contact angle measured immediately after the droplet placement; B – the contact angle after 10 sec.

The contact angle of heated pine wood specimens measured immediately after the droplet placement was larger than the contact angle of spruce specimens before heating, i.e. by 39 – 49 %, and varied from 97 and 100 . After 5 s the contact angle of pine specimens subjected to 1, 2 and 3 h heating declined by 6.5 %, 4 % and 3.7 %, respectively. The results of the measurements of contact angle values after 10 s were as follows: the value of the contact angle of specimens exposed to 1 h heating was lower by 10.7 % in comparison to the value of the contact angle measured immediately after the droplet placement. Whereas the value of the contact angle of pine wood specimens subjected to 2 and 3 h heating was lower by 7.3 % and 6.7 %, respectively. The same as in the case of spruce specimens, the duration of heating had inconsiderable impact on the size of the contact angle after heating in the case of pine wood specimens. The comparison of the contact angle values of heated and unheated pine specimens revealed similar results in the case of specimens exposed to both 1 h and 3 h heating. It was measured that after heating the contact angle of specimens subjected to 1, 2 and 3 h heating was larger than it was before heating, i.e. by 2.3, 2.44 and 2.35 times, respectively.

4. CONCLUSIONS

1. It was established that the impact on the sorption and mechanical properties of wood is proportional to the duration of heating.
2. It was found, that duration of heating has slight impact to the MOE of pine and spruce specimens.
3. It was found that after heating the modulus of elasticity of pine specimens decreased to 3.5 %, whereas in the case of spruce specimens it increased up to 1.5 %.
4. It was determined that the contact angle of unheated spruce specimens declined between 1.8 and 2.3 times within 10 s, meanwhile in the case of heated specimens it decreased from 1.06 to 1.15 times depending on the duration of heating.
5. The contact angle of unheated pine specimens declined approximately 1.8 times within 10 s, whereas in the case of heated specimens it decreased from 1.07 to 1.12 times depending on the duration of heating.
6. The experiments revealed that the duration of heating has a greater effect on spruce specimens: after 10 s the contact angle of specimens subjected to 1-hour heating was larger than it was before heating, i. e. by 246 %, and after exposure to 3 h heating it increased by 302 %. Meanwhile the contact angle of pine specimens was larger by 230 % and 235 %, respectively.

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