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MECHANICAL STRENGTH AND SURFACE ROUGHNESS OF THERMALLY MODIFIED POPLAR WOOD

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Abstract:
The wood of poplar (Populous sp.) species seem to be of low quality, mechanical resistance and is susceptible to microorganisms attacks, therefore, the application of thermal treatment is necessary, in order to improve some of its properties and increase its duration. Thermal modification of poplar (Populous sp.) wood was implemented at 180^{o}C and 200^{o}C, for 3, 5 and 7 hours in the presence of air and some of the most crucial mechanical properties were investigated. As the intensity of heat treatment increases, the mass loss of poplar boards is increased, attributed to moisture loss and the degradation and loss of chemical wood components. The bending strength decreased (2.05%-44.65%) compared to control, due to the treatments, while the elasticity decreased in all the treatment categories (5.66%-24.86%). The tangential hardness of poplar modified at 180^{o}C for 3 and 5 hours, decreased by 10.24% and 14.59%, respectively and at 7 hours it grew by 2.35%, while at 200^{o}C a decrease of 17.04 -26.05% was recorded. The radial hardness decreased (2.81%-11.06%) at 180^{o}C (3 and 5 hours) and when the duration reached 7h, it increased (9.28%), while at the treatments of 200^{o}C radial hardness decreased (2.68%-18.86%). All the treatments reduced the impact bending strength of poplar specimens (24.27%-62.02%), even though they raised the resistance levels to compressive forces. Only the milder treatment achieved to decrease the roughness of poplar wood surface, while increasing further the treatment intensity, the degree of roughness showed a strong increase.

Key words: *mechanical properties; poplar; thermal modification; treatment; roughness.*

INTRODUCTION

The history of thermal treatment of wood, aiming at improving its properties, changing the shape or form, but also increasing its life duration, begins prior to several thousands of years. The results of the heat impact on improving dimensional stability and biological resistance of wood were already known several years ago. High temperature causes chemical changes in the wood components of large molecular weight, thus altering the physical and biological properties of it.

As the temperature increases, chemical changes are conducted in the components of the cell walls, resulting in further reduction of weight of the sample, and this mass loss corresponds to change of several properties, which is the result of the first stage of wood kinetic processes. Each type of wood reacts in a different manner to heat treatment, while large differences appear between softwoods and hardwoods. Researches have shown that the larger mass losses are presented in hardwoods, either referring to heat or in hydrothermal treatment (containing larger proportion of hemicelluloses) (Zaman *et al.* 2000). In each wood species the same moisture-heat interaction mechanisms take place, but the moisture transfer rate is different, because of the structure and porosity (Younsi *et al*. 2010). Additionally, the dimensional stability of wood is reduced, as well as the hygroscopic nature of wood (shrinkage-swelling), including the decrease in equilibrium moisture content (EMC) and wettability (Gonzalez-Pena *et al.* 2009, Ates *et al.* 2010).

Heating of wood at a temperature till 100°C, its mechanical properties decrease, but the effect is not permanent, while it begins to become permanent when the heating is at temperatures higher than 100°C, depending on the duration and moisture content of wood. Thermal modification at elevated temperatures has been proved to cause a reduction in mechanical strength, especially impact bending, tensile and flexural forces, but also reduces hardness, abrasion resistance and other properties (Hill 2006, Diouf *et al*. 2011). Acetic acid and other by-products of thermal degradation seem to help reduce the mechanical strength of the modified wood and as the temperature and the treatment duration increase, the thermal degradation is intensified. This has led to the development of thermal modification methods using temperatures till 200°C.

In Greece, the species of *Populus alba, P. tremula, P. nigra* and *P. canescens* grow, and mainly hybrid plantations appear in the region of Macedonia, Thrace and Thessaly. Indicatively, in 2011 the production of public and private forests in Greece in Populus was 13003, 10829 and 3783 m^3 (total: 27616 m^3) for technical, industrial and fuel wood, respectively (Ministry of Environment 2014). The wood of this species is of low quality, mechanical resistance and is susceptible to microorganisms attacks, therefore, the application of thermal treatment seem to be necessary, in order to improve some of its properties and increase its duration of use.

OBJECTIVE

Objective of this specific research work is to investigate the influence of thermal modification at the temperature of 180°C and 200°C, for the duration of 3, 5 and 7 hours on the mechanical properties and surface roughness of a fast growing wood species, such as poplar (Populous sp.) wood, in order to look into the possibility of improving some of the properties of this species, widening in this way the future range of its applications and uses.

MATERIAL, METHOD, EQUIPMENT

Boards of poplar wood (*Populus* sp.) obtained from plantations of North Greece (Drama) were transferred to the laboratory of wood science in the Faculty of Forestry and Natural Environment (AUTH), cut parallel to grain and placed for approximately 8 months in a climate chamber (20±2°C and 60±5% RH), until a constant weight, where an equilibrium moisture content (EMC) of 10.50% (0.521 standard deviation) was acquired. This is a relatively low level of moisture content, in order to avoid the creation of internal stresses and thus distortions of wood during the treatment process. The mean density of poplar before thermal treatment (oven dry weigh/volume in moisture content level mentioned) was 0.385 g/cm^3 (0.02 St. dev.). The dimensions of the plates intended to participate in heat treatment was 35mm thickness x 70mm width x 400mm length.

The thermal treatment of wood took place in a laboratory drying chamber (80cmx50cmx60cm), where the treatment was conducted at temperature of 180°C and 200°C, under atmospheric pressure conditions in the presence of air, while the chamber was pre-heated at the final temperature. The treatment lasted 3, 5 and 7 hours (counting 15 additional minutes each time for the recovery of temperature inside the chamber) and 10 plates were used in each treatment. According to the literature, the specific temperature levels and durations of treatments have not been tested so far. At the end of treatment duration, the plates were placed in glass desiccators to return gradually to ambient conditions and subsequently stacked in a conditioning chamber of stable conditions (humidity 60±5%, 20±2°C).

Before heat treatments, measurement of weight and exact dimensions of each of the plates was performed, just as it was measured directly after the treatments, in order to record the mass loss of wood due to the process. Weight and dimensions of plates were also measured 1, 2, 3 and 4 weeks after treatment, in order to detect the recovery rate of moisture of the plates during their conditioning.

The plates of treated and untreated wood were cut to final dimensions for the properties testing, according to relevant standards and the final samples were conditioned for 1 month more till their constant weight was achieved. For each variable, 15 specimens were prepared.

After the conditioning of the samples, EMC and the density of wood was determined again using the standards ISO 3130:1975 and ISO 3131:1975.

The resistance tests to static bending forces, as well as the elasticity tests, were conducted in accordance to standard ISO 3133:1975 in Universal Testing Machine, with a loading piston speed of 5mm/min. The loading was applied tangentially to specimens in the middle of their length.

The standard on which the test of the tangential and radial «Janka» hardness of the specimens was based, was ISO 3350:1975, using an Amsler Universal Testing machine and applying a piston speed of 6mm/min. The resistance test of the specimens to impact bending forces was conducted according to ISO 3348:1975 standard, in Amsler Machine, by performing loading at the center of each specimen, perpendicularly to the tangential surface of the samples. The compression test of the specimens was conducted according to DIN 52185:1976, in the same machine, with a piston speed of 6mm/min.

The surface roughness of the samples was determined using a profilometer apparatus (Mitutoyo Surftest SJ-301) and a diamond stylus device. The measuring speed, the pin diameter and the top corner of the pin tool was 10mm/min, 4μm, and 90°, respectively. The roughness indexes values were determined with an accuracy of ± 0.01μm. The points of roughness measurement were randomly chosen on the surface of the samples. Measurements were implemented in a direction perpendicular to the fiber direction of the samples. Three roughness parameters were recorded, the mean arithmetic deviation of profile - Ra, the mean peak-to-valley height - Rz, and the root mean square deviation of the assessed profile - Rq (ISO 4287, DIN 4768). All the plates were subjected to sanding before the measurment and then, cut to smaller dimensions (50mmx50mmx50mm) for the roughness test, which was carried out in the tangential surface, because it usually presents lower roughness, compared to radial one (Budakci *et al*. 2013). For the statistical analysis of the results, SPSS Statistics PASW was used in order to analyze the variability of mean resistance values using the method Bonferoni and Tamhane (One way ANOVA), as well as the Least Significant Difference method (LSD - two-way ANOVA).

RESULTS AND DISCUSSION

According to the results (Table 1), as the treatment intensity, referring to the temperature and duration, increases, the rate of mass loss of the specimens is increased due to the processes taking place during wood modification and can be attributed to the moisture loss, the evaporation of volatile extracts, the loss of chemical constituents due to their degradation, evaporation and loss of thermal degradation products (mainly hemicelluloses) from the mass of wood (Kocaefe *et al.* 2008).

Table 1

Average mass loss percentage values of poplar specimens after heat treatment

	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Mass Loss	**11.237 (2.512)**	**11.601 (0.645)**	**11.935 (0.975)**	**13.461 (2.192)**	**16.409 (1.974)**	**18.884 (2.642)**

standard deviation values within the brackets

Corresponding researches has shown that hardwoods present larger mass losses compared to conifers, probably due to the higher percentage of hemicelluloses (pentoses), which are more susceptible to thermal degradation and already depolymerized at 180-200°C. Additionally, hardwoods have a slightly lower lignin content, which seem to be more resistant to thermal degradation than the other components (Gonzalez-Pena *et al.* 2009).

According to statistical analysis, all the mean loss values of the samples were found to differ statistically significantly from one another, except the cases between control and the two milder treatments (180°C - 3 and 5h) and also the case between samples of treatment of 180°C (5 and 7h).

Significant mass loss, proportional to the treatment temperature, was recorded by corresponding investigations: Esteves *et al.* 2007, Korkut and Budakci 2010, Schneid *et al*. 2014 etc.

There was a decrease in the recovery rate of the initial weight of the specimens, which is more pronounced as the treatment temperature and duration increase (Table 2). This means that the EMC obtained by the samples after their conditioning tends to stabilize at lower levels than those of control, and as is evident, the modified samples of shorter duration treatments exhibit higher weight increase, due to higher absorption of moisture from the surrounding atmosphere, compared to the samples of more intensive treatments.

Table 2

Conditioning progress (moisture recovery rate) of boards indicated by values of weight gain percentage 7, 14, 21 and 28 days after treatment

Treatment	Weight increase % (7 days)	Weight increase % (14 days)	Weight increase % (21 days)	Weight increase % (28 days)
180°C-3h	**2.533 (0.335)**	**3.057 (0.336)**	**3.532 (0.371)**	**3.983 (0.389)**
180°C-5h	**2.427 (0.350)**	**3.066 (0.393)**	**3.523 (0.457)**	**3.932 (0.478)**
180°C-7h	**2.509 (0.405)**	**2.958 (0.419)**	**3.355 (0.395)**	**3.727 (0.397)**
200°C-3h	**2.125 (0.312)**	**2.730 (0.384)**	**3.142 (0.498)**	**3.585 (0.462)**
200°C-5h	**2.073 (0.333)**	**2.586 (0.301)**	**2.865 (0.319)**	**3.349 (0.272)**
200°C-7h	**2.033 (0.196)**	**2.704 (0.253)**	**2.992 (0.249)**	**3.412 (0.227)**

Standard deviation values within brackets

As the treatment intensity increases, both EMC and density of wood are reduced (Table 3). EMC of specimens, modified at 180°C for 3, 5 and 7 hours presented a decrease of 18.76%, 24.12% and 28.42%, compared to control, whereas those modified at 200°C for 3, 5 and 7 hours, a decrease of 41.17%, 45.83% and 49.20%. This reduction in EMC clearly indicates that thermal treatment greatly affects the dimensional stability of wood, the absorption of moisture from the atmosphere and is directly related to the reduction of hydroxyls in wood mass due to treatment.

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Table 3

Mean values of EMC and density of treated and untreated specimens after 4 weeks of conditioning

Treatment	EMC %	Density (g/cm^3)
Control	10.506 (0.521)	0.385 (0.002)
180°C - 3h	8.535 (0.377)	0.381 (0.006)
180°C - 5h	7.972 (0.500)	0.356 (0.007)
180°C - 7h	7.520 (0.871)	0.346 (0.007)
200°C - 3h	6.181 (0.654)	0.336 (0.005)
200°C - 5h	5.691 (0,246)	0.333 (0.002)
200°C - 7h	5.337 (0.402)	0,291 (0.005)

Standard deviation values within brackets

The samples modified at 180°C for 3, 5 and 7 hours showed a decrease in density of 1.04%, 7.53% and 10.13%, respectively, while the 3, 5 and 7 hours treatment at 200°C, a decrease of 12.73%, 13.51% and 24.42%, compared to control. Schneid *et al.* (2014), Günduz and Aydemir (2009), Esteves *et al.* (2007), Arnold (2010), Ates *et al*. (2010), Niemz *et al.* (2010) etc. have presented also similar results and behavior of thermally treated wood. This density decrese is also associated to the reduction in moisture content after treatment and the mass loss caused by the thermal decomposition of hemicelluloses and the evaporation of volatile wood extracts that leave vacant places in its mass. Wood density decrease is also associated to a potential of mechanical properties decrease, though the decrease of EMC contributes to the increase of mechanical strength (Hill 2006).

Thermal treatment at 180°C for 3, 5 and 7 hours seem to have decreased also Modulus of Rupture (MOR) by 2.05%, 7.62% and 21.89%, respectively. An even stronger decrease of MOR was marked in specimens that underwent treatments of 3, 5 and 7 hour at 200°C, revealing a decrease of 32.04%, 34.56% and 44.65%, compared to control (Fig. 1). Two Way ANOVA method applied to MOR values of the samples showed that temperature is a very important factor, which has a statistically significant effect on MOR affecting by 77% its variability.

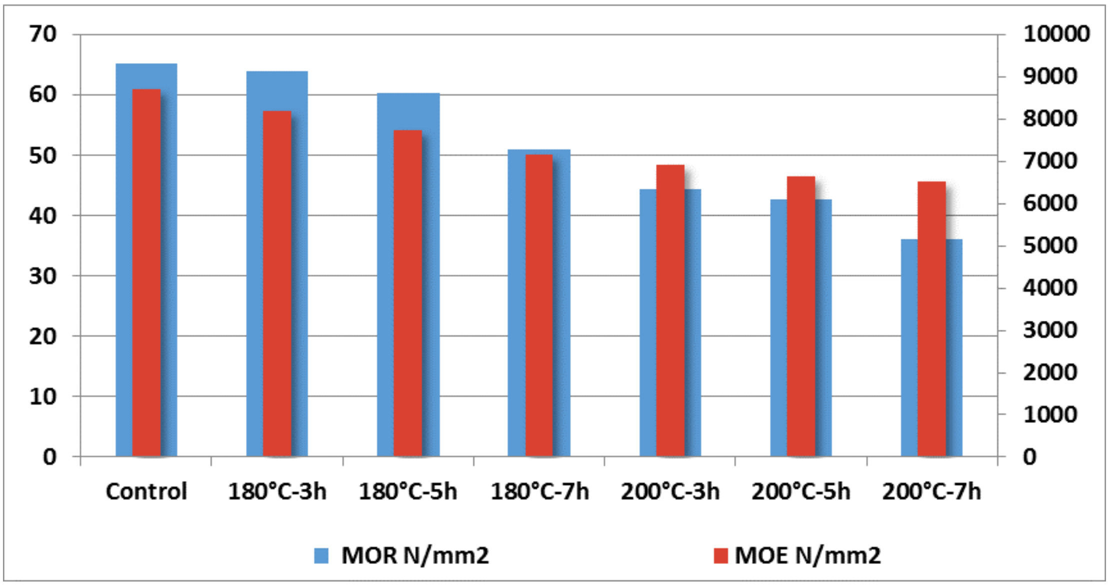

Fig. 1
Mean Modulus of Rupture and Modulus of Elasticity values (measured in the bending test process) of the control and thermally modified specimens (N/mm^2).

Heat treatments of 180°C for 3, 5 and 7 hours showed a 5,66%, 11,19% and 17,57% decrease in the modulus of elasticity (MOE), while the treatment of 3, 5 and 7 hour at 200°C recorded a 20.53%, 23.67% and 24.86% decrease. The factors of temperature and time have a statistically significant effect on MOE, while the temperature affects its variability by 26% and the time by 10.4%. Kocaefe *et al.* (2008) reported a relatively slight increase in MOR and MOE of Canadian birch and poplar at 160-200°C, probably due to the branching of lignin in wood, while they both decrease to temperatures above 200°C. Most researches, however, report a decrease in MOR and a slight increase in elasticity (Olek and Bonarski 2008, Li Shi *et al.* 2007, Németh and Miklós 2012, Goli *et al.* 2014, Akyildiz *et al.* 2009).

According to hardness tests results, all thermal treatments, except for 180°C-7hours, decreased hardness of wood in both tangential and radial cross-section (Table 4). More specifically, the tangential hardness of specimens, modified at 180°C for 3 and 5 hours, decreased by 10.24 and 14.59%, while by increasing the duration to 7 hours the hardness increased slightly (by 2.35%), compared to control. This increase may possibly be attributed to the formation of some components in woody mass, which probably enhances wood hardness. Samples treated at 200°C for 3, 5 and 7 hours presented reduced tangential hardness by 17.04%, 24.92% and 26.05%, respectively.

Table 4

Mean tangential and radial hardness values of thermally treated and untreated specimens (kN)

Hardness (kN)	Control	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Tangent.	**1.954 (0.221)**	**1.754 (0.174)**	**1.669 (0.157)**	**2.000 (0.285)**	**1.621 (0.183)**	**1.467 (0.118)**	**1.445 (0.129)**
Radial	**2.025 (0.213)**	**1.968 (0.189)**	**1.801 (0.157)**	**2.213 (0.262)**	**1.972 (0.161)**	**1.710 (0.170)**	**1.643 (0.139)**

Standard deviation values within brackets

Regarding the radial hardness of the specimens, it decreased by 2.81% and 11.06% in the mildest treatments (180°C for 3 and 5h), while at 7 hours treatment it increased by 9.28%. Samples treated at 200°C for 3, 5 and 7 hours revealed a reduce of 2.68%, 15.52% and 18.86%, respectively. In a similar research by Li Shi *et al.* (2007), thermally modified poplar showed a decrease in radial, tangential and transverse hardness by 26%, 39% and 15%, respectively, while Goli et al. (2014) recorded similar findings, as well.

The effect of temperature on the tangential hardness of poplar wood was statistically significant and the main factor affecting its variability by 39.6%. A statistically significant effect was also found in the temperature-duration interaction, in which 19.9% of the hardness variability is attributed, while the duration affects it only by 11.3%. Similar were proved the two-way ANOVA findings for the radial hardness.

Heat treatment at 180°C for 3, 5 and 7 hours reduced the impact bending strength by 24.27%, 37.56% and 47.40%, respectively, while treatments of 200°C resulted in a strength decrease of 45.73%, 49.54% and 62.02%, compared to control (table 5). Generally, the intensity of the particular thermal treatments applied in the present project was of such intensity, that caused a high decrease in the impact bending strength of poplar.

Table 5

Mean values of impact bending strength (J/cm^2) of heat-treated and untreated specimens

Impact Bending (J/cm^2)	Control	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
	2.620 (0.312)	**1.984 (0.395)**	**1.636 (0.290)**	**1.378 (0.186)**	**1.422 (0.177)**	**1.322 (0.153)**	**0.995 (0.086)**

Standard deviation values within brackets

Temperature and time appeared to affect the variability of impact bending strength (46% and 46.5% respectively), both of which had a statistically significant effect on impact bending resistance. The interaction between temperature-time can be characterized as not statistically significant.

According to compression test results, heat treated samples showed higher levels of resistance than control levels (Fig. 2). Even the milder treatment (180°C-3h) was sufficient to bring about a satisfactory improvement in the compression strength of poplar specimens, and as treatment duration increases (at 180°C), strength increases as well. Specifically, treatments of 180°C (3, 5, 7h) increased the compression strength by 13.70%, 15.84% and 18.14%, respectively, while treatments of 200°C (3, 5, 7h) resulted in an increase of compression strength by 23.26%, 23.18% and 22.13%, compared to control. Németh and Miklós

(2012), as well as Mazela *et al.* (2010), also recorded a slight increase in compressive strength of poplar wood after thermal treatment.

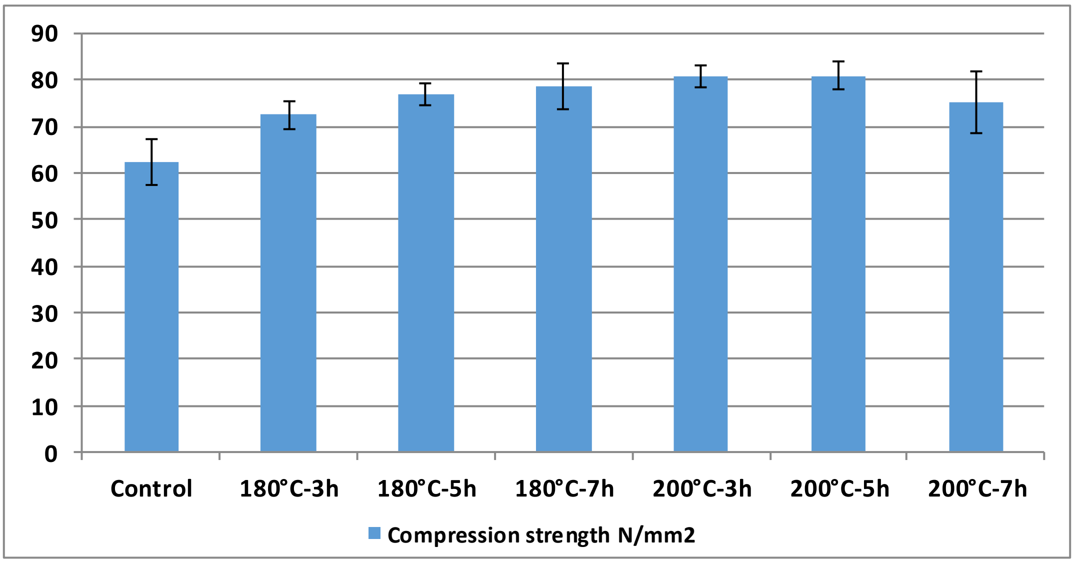

Fig. 2.
Mean values of compression strength (N/mm^2) of heat-treated and untreated poplar samples.

According to statistical analysis, only the compression strength of control specimens was found to differ significantly from all the modified categories of specimens. Temperature was found to be the major factor affecting the compression strength variability by 17.1%.

According to the roughness tests on the surface of poplar specimens, it emerged that only the mildest treatment slightly reduced the roughness of wood surface, while increasing the temperature and duration of the treatment, the degree of roughness showed an intense increase compared to control levels. Specifically, the roughness index Ra value (most representative) of the specimens treated at 180°C for 3 hours, presented a slight decrease of 2.80%, while as the temperature and the duration increased, Ra showed an increase of 26.92% to 70.36%, compared to control level. This behavior may be attributed to the fact that thermal treatment causes a mass loss, making wood more brittle, reducing the number of hydroxyls and its moisture content, causing degradation of the hemicelluloses and all these affect the roughness of wood surface (Budakci *et al.* 2013). In contrast, in several previous researches (Korkut and Guller 2008, Korkut *et al.* 2008, Korkut and Budakci 2010, Schneid *et al.* 2014, Baysal *et al*. 2014) the surface roughness was found to be decreased as the temperature and duration of thermal treatment increases.

Table 6

Mean values of the surface parameters (Ra, Rz, Rq) of thermally modified and control specimens

Parameter	Control	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Ra	2.355 (0.347)	2.289 (0.181)	2.989 (0.235)	3.137 (0.314)	3.150 (0.304)	3.128 (0.384)	4.012 (0.302)
Rz	18.106 (1.772)	17.366 (2.455)	22.225 (2.396)	24.528 (1.689)	22.913 (1.852)	23.348 (2.527)	27.541 (1.885)
Rq	3.225 (0.449)	2.965 (0.223)	4.005 (0.339)	4.403 (0.433)	4.205 (0.291)	4.211 (0.500)	5.227 (0.314)

Standard deviation values within brackets

According to statistical analysis of Ra roughness values, the effect of temperature and time factors was found to be both statistically significant, affecting its variability by 55.5% and 61.2% for temperature and duration, respectively. The interaction between temperature-time was also statistically significant, affecting the variance of Ra by 27.4%.

CONCLUSIONS

According to the research, as the treatment intensity increases, higher mass losses, caused by the treatment, are being recorded, which corresponds to moisture and the wood components loss, due to thermal degradation. All treatments resulted in a reduction of the recovery rate of the initial weight, due to the reduced moisture recovery, which is more intense as the temperature and duration of treatment increases. EMC of all the samples was found reduced, compared to control, even in the case of the mildest treatment, while as the treatment intensity increases, a greater decrease is marked (18.76% -49.20%). Additionally, The bending strength decreased (2.05%-44.65%) compared to control, due to the treatments, while the elasticity decreased in all the treatment categories (5.66%-24.86%). Furthermore, the tangential hardness of poplar samples modified at 180°C (3, 5, 7h), declined by 10.24%, 14.59% and an increase of 2.35% in 7 hours, while treatments of 200°C reduced the tangential hardness by 17.04% -26.05%. The radial hardness decreased by 2.81% and 11.06% at 180°C treatments of 3 and 5 hours, respectively, while the duration of 7 hours caused an increase. Treatments of 200°C also reduced the radial hardness (2.68% - 18.86%). In each case of treatment, the impact bending strength of poplar wood was reduced (24.27%-62.02%), compared to control, while all treatments resulted in an increase of the compression strength of the samples (13.70% - 23.26%). Finally, only the less severe treatment managed to decrease slightly (2.80%) the roughness of wood surface, while increasing the temperature and duration of treatment, the degree of roughness sharply increased (26.92% to 70.36%).

In most of the cases, the effect of the temperature factor was found statistically significant and affected the variability of property values at a higher extent, compared to the treatment duration or the interaction between the two factors.

An ideal way of utilizing this modified species is to be used in applications where its new enhanced properties can be fully utilized, ensuring that the deteriorated properties will not be critical priority during the use. Wooden frames, floors, bathroom and kitchen structures, linings, decorative indoor/outdoor details, participation selectively in the construction of outdoor furniture under shelter or after additional protection, may be some possible applications of the specific materials.

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