Proceedings of the 5th International Conference on Processing Technologies for the Forest and Bio-based Products Industries (PTF BPI 2018)
Freising/Munich, Germany, September 20-21, 2018

Physical and hygroscopic properties of pine and poplar wood after heat treatment

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Key words (colour, hygroscopic properties, wood modification, pine, poplar, thermal treatment)

ABSTRACT

Thermal modification is a method that adds value to the existing timber and improves some of its most crucial physical characteristics, while it constitutes an alternative wood preservation method, since it produces an environmentally friendly product in comparison to chemically modified wood. The aim of the present research is to comprehend the response of poplar (Populus sp.) and pine (Pinus nigra L.) wood to thermal modification at the temperature of 180°C and 200°C, for 3 different treatment durations of 3, 5 and 7 hours, in the presence of air, by examining their physical and hygroscopic properties and comparing these species properties one another. Specifically, in this research, the mass loss of the samples due to the treatment, the density, the equilibrium moisture content (EMC) and the hygroscopic properties (radial, tangential and longitudinal swelling and absorption percentages) were examined. Generally, treatments of low energy consumption and cost were selected to be applied using simple equipment, which could be easily applied to many wood industries under the current financial conditions and given the shortage of timber of high quality.

1 INTRODUCTION

Although wood is proved to be considered one of the most appropriate construction material options, it presents disadvantages inherent in the hygroscopic nature. Thermal modification is a method that adds value to existing wood and improves some of its physical characteristics, such as dimensional stability and biological resistance, which could extend the life of the wood and the structures in which it participates, and produces an environmentally friendly product with regard to chemically modified wood.

The modification, depending on its duration and the temperature used, causes a loss of wood mass and a reduction in its volume. The mass that is lost at low temperatures is mainly volatile extracts and water from the mass of the wood, while above 100°C, loss of some macromolecular components of the wood is caused, resulting from the hydrolysis of their macromolecules, their decomposition and depolymerization, intensified by the rise in temperature (Kocaefe et al., 2008). Hardwood species show higher rates of mass loss due to treatment, compared to softwood ones. Esteves et al. (2007) report that a 3% loss of mass is the minimum loss to achieve improved dimensional stability and wood hygroscopicity and 5% for improved biological resistance. Even at low mass loss rates (0.4%) there is a decrease in hemicelluloses, arabinose, rhamnose and galactose (the glucan proves to be more resistant), while phenolic compounds are formed (Kartal et al., 2007).

Additionally, thermal modification has been proved to increase dimensional stability and reduce the hygroscopicity of wood, to a greater or lesser extent, depending on the treatment conditions, temperature level, duration, atmosphere, etc. The heat during treatment alters the chemical composition of the wood and hence its physical properties. Already from the temperature of 150°C and above, the hygroscopic properties of wood are being improved and the improvement increases proportionally as the temperature rises, marking a 55-90% improvement in wood stability (Gündüz et al., 2008), while the equilibrium moisture content (EMC) decreases by 40-60% (Gong et al., 2010), due to the dehydration reactions carried out, but for longer modification durations, it seems to increase again (Esteves et al. 2007, Hill 2006). A research of Johansson (2005) showed that at temperatures of 185-250°C EMC is reduced by 43-60%, while the shrinkage-swelling rate is reduced by 30-80%. Similar results were also recorded by Schneid et al. (2014), Kartal et al. (2007), Aydemir et al. (2011) etc.

The objective of this project research is to examine the way of responding of a softwood and a hardwood species to thermal modification of various conditions, recording their physical and hygroscopic properties, essential for the utilization of these species in various applications. Poplar was chosen because it constitutes a wood species found in abundance, due to the large number of plantations established in the last years, while parallel it is along with being a fast-growing species of low quality, physical properties and biological resistance that needs to be enhanced. Similarly, the species of black pine is an easily available raw material and was chosen due to its low biological resistance in the sapwood, as well as its intense hygroscopic nature.

2 MATERIALS AND METHODS

Boards of black pine (*Pinus nigra* L.) and poplar (*Populus* sp.) trees obtained from Thessaloniki region (North Greece) were placed for about 8 months in an air-conditioned room at a temperature of 20 ± 2°C and 60±5% relative humidity and left there to dry naturally till a constant weight marking an equilibrium moisture content (EMC) of 10.50% for poplar (0.521 standard deviation SD) and 11.44% for pine wood (0.172 SD). The mean density (mass / volume, measured with moisture content at the reported levels) of the pine was 0.662 g/cm^3 (0.01 SD) and of the poplar – 0.385g/cm^3 (0.02 SD). The dimensions of the boards prior to the treatment were 35 mm x 70 mm x 400 mm, and consisted mainly of sapwood and a small portion of heartwood.

2.1 Thermal treatments

The treatment of the boards was carried out in a laboratory drying chamber (80 cm x 50 cm x 60 cm). Thermal treatment was carried out at a temperature of 180°C and 200°C under atmospheric pressure in the presence of air. Treatments of low energy consumption and cost were selected to be applied using simple equipment, which could be easily applied to many wood industries under the current financial conditions and given the shortage of timber of high quality. The moisture content of the boards was 10.5% and 11.44% for poplar and pine wood, respectively, when placed in the chamber (10 boards /operation, preheated to the final temperature chamber). The treatment lasted 3, 5 and 7h (counting 15 minutes more for the chamber temperature recovery). After thermal treatment, the boards were placed in desiccators to return to ambient conditions gradually and stacked in an air-conditioned room (60±5%, 20±2°C). Only defect-free material participated in tests.

2.2 Mass loss and density

Prior to the thermal treatments, the weight and dimensions of each board was measured, as well as after the treatments, to record the loss of wood mass. The weight and dimensions of the boards were also measured after 1, 2, 3 and 4 weeks after the treatment to monitor the recovery of the moisture content of the boards during their conditioning. After about 2 months of conditioning, EMC and density of the boards were tested again, based on ISO 3130:1975 and ISO 3131:1975, respectively.

2.3 Hygroscopic properties

The weight and the three dimensions (nominal: 2 cm × 2 cm × 2cm) of each specimen were measured with accuracy, before the specimens were immersed in water (20±3°C) using mesh and weights. The radial, tangential and longitudinal dimension of each specimen was measured again, as well as its weight, after 1, 3, 6, 24 and 72 h of immersion, in order to illustrate the swelling and absorption rate of the testing specimens (ISO 4859:1982).

For the radial, tangential and longitudinal swelling percentage calculation, the following equation was used:

$$S = (L_1-L_0)/L_0 * 100 \quad (1)$$

where S is the swelling (%), L_1 is the dimension after immersion (mm) and L_0 the dimension before immersion (mm).

The following equation was used to calculate the adsorption percentage of the samples:

$$WA = (W_1-W_0)/W_0 * 100 \quad (2)$$

where WA is the absorption (%), W_1 is the wet weight after immersion (g) and W_0 the weight before immersion (g).

3 RESULTS

3.1 Mass loss due to treatment

According to the results (Table 1), as the treatment intensity (temperature and duration) increases, the percentage of mass loss of the specimens increases as well.

Table 1. Mean percentage mass loss values of poplar and pine specimens after heat treatment

Wood species	Mass loss %					
	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Poplar	11.24 (2.51)	11.60 (0.64)	11.93 (0.97)	13.46 (2.19)	16.41 (1.97)	18.88 (2.64)
Pine	10.63 (0.27)	11.26 (0.51)	11.44 (0.57)	12.85 (1.03)	13.98 (1.17)	15.25 (1.51)

Standard deviation values within brackets

In the first stages of thermal treatment, heat contributes to the evaporation of wood moisture without raising its temperature, while the temperature of wood rises gradually on its surface and then inside its mass, causing component losses due to thermal degradation (Kocaefe et al., 2008). Given the fact that poplar had a mean initial moisture content of 10.50 %, it is easy to conclude that, even in the milder treatment, a percentage higher than its initial moisture content was lost, which does not ensure the total loss of the whole moisture content of wood, since there is a possibility that some extracts were also lost at this stage, besides moisture (180°C-3h). As the treatment intensity increases and, with it, the mass loss, it is assured that wood is dried and further loss is due to thermo-degradation of its components – evaporation of volatile extracts, loss of chemical constituents of wood, evaporation and loss of thermal degradation products (Kocaefe et al., 2008).

It is noted that, in all treatments, poplar recorded higher mass loss rates compared to those of the black pine, and this difference is more pronounced in treatments of 200°C. According to bibliography, hardwood species reveal larger mass losses compared to softwoods, probably due to their higher percentage of hemicelluloses. Softwood species hemicelluloses (hexoses) are more stable than hardwood species hemicelluloses (pentoses), due to different composition, and are being already depolymerized at 180-200°C. Furthermore, softwoods have a slightly higher lignin content than hardwoods, with lignin being more resistant to thermal degradation than other components, and softwood lignin seems to be less susceptible to thermal degradation than the lignin of hardwoods (Gonzalez-Pena et al., 2009). A similar response of pine was recorded by Esteves et al. (2007) and Zaman et al. (2000).

3.2 Mass recovery during conditioning

According to the monitoring of the mass rates of poplar and pine specimens after treatments, a decrease in the recovery rate of their initial weight is observed, which is more pronounced as the temperature and duration of treatment increases (figure 1). This means that EMC value of treated poplar and pine, after a conditioning period, seem to be in lower levels than that of unmodified wood. The specimens treated at shorter treatment durations exhibited higher weight gain, due to higher moisture absorption from the surrounding atmosphere, compared to samples of more intensive treatments.

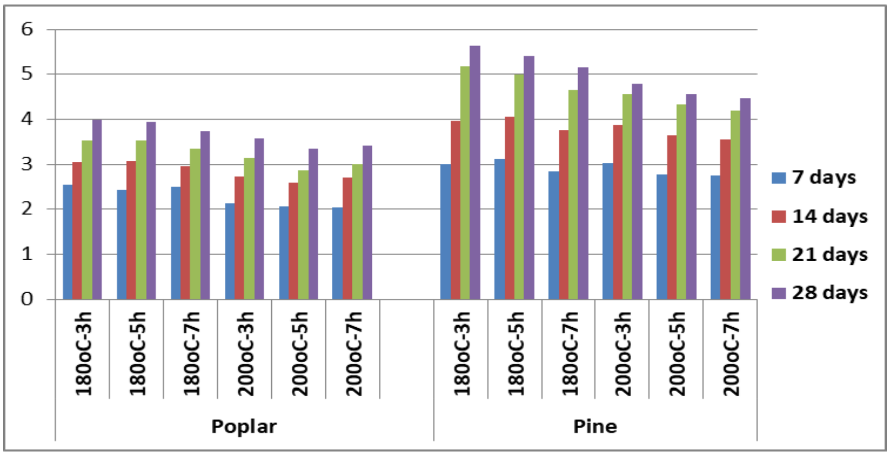

Figure 1. Mean weight gain percentages of poplar and pine boards, 7, 14, 21 and 28 days after treatment due to moisture content recovery

The percentage of weight increase of the poplar boards gradually increases between 7, 14, 21 and 28 days, while pine boards seem to absorb most of the moisture up to 21 days. Both species presented the highest moisture absorption rate in the 7th day.

3.3 EMC and Density

The EMC of all modified specimens was found to be reduced compared to control EMC, both for poplar and pine wood, even in the case of the mildest treatment (180°C-3h). As the treatment intensity increases, both EMC and wood density are reduced (Figure 2). Specifically, in treatments of 180°C, (3, 5 and 7h), the EMC of pine was reduced by 20.73%, 22.48% and 26.27%, respectively, while in treatments of 200°C (3, 5 and 7h), the EMC decreased by 29.89%, 34.22% and 38.43%. Poplar modified at 180°C (3, 5 and 7h) presented an EMC reduced by 18.76%, 24.12% and 28.42% compared to the control, whereas poplar modified at 200°C (3, 5 and 7h) demonstrated an EMC decreased by 41.17%, 45.83% and 49.20%, respectively. This EMC reduction clearly shows that thermal treatment greatly affects the dimensional stability of wood and the moisture absorption from the atmosphere, and is directly related to the reduction of hydroxyls in wood mass.

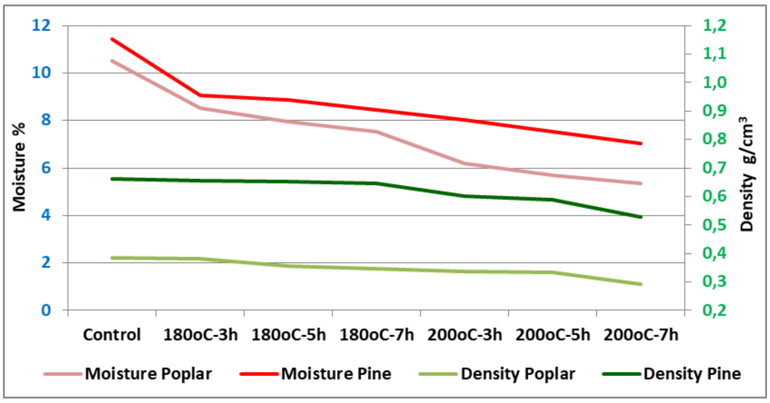

Figure 2. Mean moisture content and density values of modified and unmodified poplar and pine specimens after 4 weeks of conditioning

Additionally, the thermal modification process also caused a reduction in the density of poplar and pine specimens. The 180°C treatments (3, 5 and 7 h) led to a reduction in the density of pine wood by 0.76%, 1.36% and 2.57%, respectively, while in treatments of 200°C (3, 5 and 7 h), the density value was further reduced by 9.37%, 10.88% and 20.24%, compared to the control. Correspondingly, poplar treated at 180°C treatments (3, 5 and 7 h) presented density reduced by 1.04%, 7.53% and 10.13%, respectively, while the treatments of 200°C (3, 5 and 7 h) further reduced the density by 12.73%, 13.51% and 24.42%. This density decrease can be associated with a moisture content decrease after treatment and the mass loss caused by thermal decomposition of hemicelluloses and evaporation of volatile wood extracts, which leave empty spaces in wood tissue. This density decrease after treatment is related to the deterioration of mechanical properties, while the decrease in EMC contributes to strength increase (Hill, 2006). The findings of Güller (2012), Schneid et al. (2014), Ates et al. (2010), Telkki et al. (2010), Niemz et al. (2010) are in agreement with the results of the present research.

3.4 Hygroscopic properties

According to the results, the hygroscopic properties of the thermally modified poplar and pine appeared to be improved due to the applied treatments (Figure 3, 4, 5). This is probably attributed to the decomposition of hemicelluloses, the polymerization of molecules and the dissolution of hydroxyl groups from the amorphous areas of cellulose (Schneid et al., 2014).

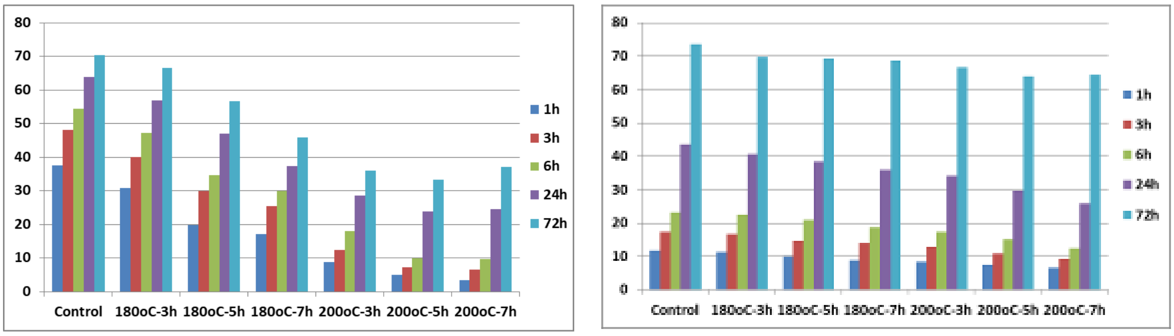

Figure 3. Mean absorption values (%) of modified and unmodified pine (left) and poplar wood (right)

The absorption test of the samples carried out after immersion in water for 1, 3, 6, 24 and 72 hours showed that even the mildest treatment (180°C-3h) improved the hygroscopic nature of wood and reduced the moisture absorption rates at all stages compared to control, due to the reduction of hemicelluloses, the number of hydroxyls and the branching of the lignin mesh in wood mass. It is remarkable that the decrease rate of absorption is intense up to 5 hours of treatment at 200°C, while the last and more intensive treatment (200°C-7 h) tends to cause a rebound of adsorption levels, which can be attributed to the extensive wood chemical composition changes carried out during high-intensity treatments (loss of polysaccharides, degradation of the cellulose-hemicellulose-lignin network).

According to the statistical analysis of Two-Way ANOVA of the pine specimen absorption values (at 72 hours of immersion), the effect of temperature was statistically significant and influences its variability by 87.7%. The factor of treatment duration also has a statistically significant effect on absorption rates, affecting its variability by 51.4%. Interaction between these two factors was also statistically significant and influenced the adsorption variability by 57%. In the treatment of 3 hours, the highest intensity effect of temperature was observed, recording statistically significant differences.

The hygroscopic properties of heat-treated poplar, like pine wood, were also improved due to the thermal treatments applied. Even the mildest treatment (180°C-3h) improved the hygroscopic nature of wood and reduced the moisture absorption rates at all stages compared to the control sample.

Remarkable is the fact that the reduction rate of absorption is relatively constant until the most intensive treatment (200°C-7h). In all cases (modified and control specimens), wood absorbs smaller amounts of water in the first hours (1, 3, 6h), it absorbs much more water between 6 and 24 hours, and even more water from 24 to 72 hours of immersion in water.

The factor of temperature was found statistically significant and influenced variability of absorption by 22.5%. Duration did not have a statistically significant effect on the adsorption level. The treatment duration of 5 hours showed the greatest effect of temperature, which is marginally statistically significant. The mean tangential swelling values of the pine control sample seem to be significantly higher than the corresponding radial swelling values at all steps of measurements (1, 3, 6, 24 and 72h). However, as it is evident, thermal treatment tends to improve hygroscopic behavior and to further reduce swelling levels tangentially compared to the radial direction of wood, thereby contributing to limiting the anisotropy of wood, which is of great importance for its utilization.

The reduction rate of tangential and radial swelling is quite intense up to 5 hours of treatment at 200°C (as in absorption), while the most intensive treatment (200°C-7h) tends to slightly increase the swelling rates again without approaching the control level, due to permanent changes in chemical composition occurring in wood, and due to intense mass loss, density reduction etc. Therefore, if the reduction of its hygroscopic nature is a priority in the use of pine wood, and the conditions of the specific experiment approach the material used (dimensions, treatment conditions, etc.), this tendency to increase should be taken into account and treatments over 5 or 7 hours should not to be chosen. In a previous research (Kamperidou and Barboutis, 2012), heat modification of poplar was performed, showing a mass loss that corresponded to the wood EMC reduction and a decrease in hygroscopicity and dimensional changes which were recorded even in the shorter treatment of 2 hours. When the treatment duration exceeded 6 hours, a slight increase of radial swelling and absorption rate was observed.

According to the TWO ANOVA statistical analysis of tangential swelling values of pine specimens (at 72 hours of immersion), the effect of temperature was statistically significant and influenced the variability by 61.6%. Treatment duration also has a statistically significant effect on swelling, affecting it by 23.5%. The interaction between these two factors was also statistically significant and influenced the fluctuation of swelling by 22.5%. In 3-hour treatments, the greatest effect of temperature was observed, recording statistically significant differences.

Similarly, the analysis of the radial swelling values of pine specimens showed that the effect of temperature was statistically significant and influenced its variability by 62.2%. The factor of duration also has a statistically significant effect on the swelling levels, affecting its variability by 36.4%. Interaction between temperature and duration was also significant and influenced the swelling variability by 36.5%. The highest effect of temperature was observed in treatments of over 3 hours and was statistically significant.

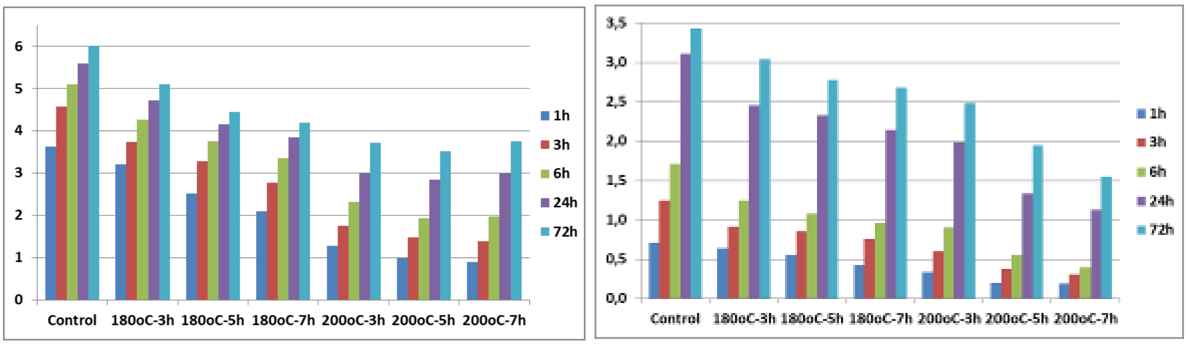

Figure 4. Mean values of tangential swelling (%) of modified and control pine (left) and poplar (right) specimens

The mean tangential swelling percentages of poplar control specimens appear to be significantly higher than those of the radial swelling at all measuring steps (1, 3, 6, 24 and 72h). Thermal treatment, however, tends to further reduce swelling levels tangentially compared to the radial direction of wood, putting a limit in wood anisotropy, as has been observed in the case of pine. Generally, the higher the temperature and the duration of the treatment, the greater the reduction of the tangential, radial and longitudinal swelling of poplar specimens, which is very important for the utilization of this species in various applications.

According to the analysis of the tangential swelling values of poplar (72h of immersion), the effect of temperature was found to be statistically significant and affected its variability by 80.6%. The duration also has a statistically significant effect on tangential swelling, affecting its variability by 62.8%. The interaction between the two factors is statistically significant and affects the swelling variability by 24%. In the 7-hour treatments, the most intensive effect of temperature was observed, which is statistically significant. The statistical analysis of the radial swelling values of poplar showed that the effect of temperature was found to be statistically significant and influenced the radial swelling variability by 87.4%. Duration also has a statistically significant effect on the levels of radial swelling, affecting its variability by 64.7%. The interaction between temperature and duration appeared weak. Generally, in 5- and 7-hour treatments, the greatest effect of temperature is observed, which is also statistically significant.

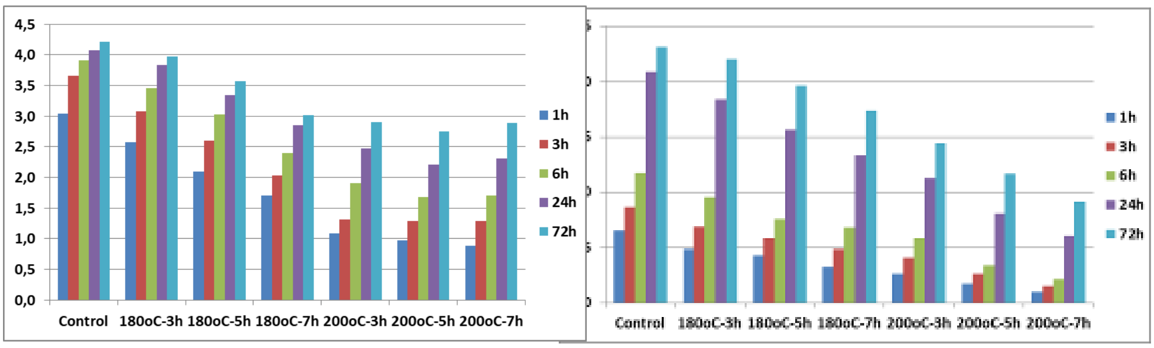

Figure 5. Mean values of radial swelling (%) of modified and control specimens of pine (left) and poplar (right) specimens

The thermal treatments even reduced the percentages of the longitudinal swelling of poplar and pine specimens, which were initially low compared to the respective tangential and radial swelling rates, demonstrating its beneficial effect on the hygroscopic nature of wood. In general, the higher the temperature and the duration of treatment, the greater the reduction in tangential, radial and longitudinal swelling of the pine specimens was observed, except for the last treatment that exhibited a slight increase tendency in the swelling level.

According to the statistical analysis of the swelling of the pine specimens (72h of immersion), the effect of temperature was statistically significant and influenced its variability by 14%. The factor of duration did not show a statistically significant effect on longitudinal swelling of pine. The interaction between temperature and duration was found to be insignificant. No treatment revealed a statistically significant effect of the treatment duration, which is easily explained by the fact that the values generally varied slightly.

The statistical analysis of the longitudinal swelling values of poplar showed that the effect of temperature was statistically significant and influenced the swelling variability by 82.4%. The duration also had a statistically significant effect on longitudinal swelling, affecting its variability by 31.6%. The interaction between the two factors was not found statistically significant. The greatest effect of temperature (180°C and 200°C) was observed in 3-h treatments, which corresponds to significant differences.

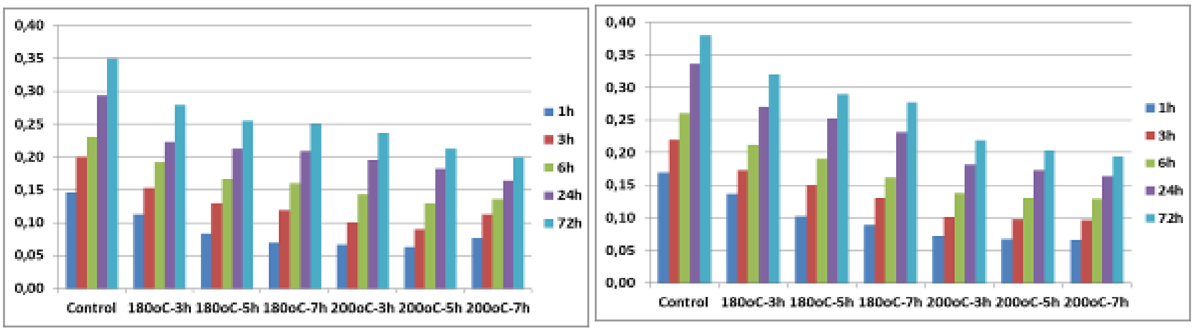

Figure 6. Mean values of longitudinal swelling (%) of modified and control specimens of pine (left) and poplar (right) specimens

Similarly, Gündüz et al. (2008), who thermally modified black pine at 120-180°C for 2-10 hours in the presence of air, recorded, even at low temperatures (150°C), a decrease in swelling levels and reported that the tangential swelling was improved more than the radial one, which was before treatment slightly smaller than the tangential. Güller (2012) also modified black pine at 190-225°C for 60-180 min. and recorded a reduction in dimensional stability of up to 66%, with temperature being the most critical factor in improving hygroscopicity and dimensional stability over the duration of the treatment.

As shown in the corresponding figures, the control specimens of pine recorded slightly higher levels of absorption and swelling (mainly tangentially and radially), compared to the corresponding percentages of poplar control specimens, which can be explained by the higher density of pine, whereas the absorption and swelling reduction due to treatments was found to be higher in pine wood.

4 CONCLUSIONS

As the treatment intensity increased, the mass loss of pine and poplar plates due to treatment also increased, which corresponds to the loss of moisture, and the chemical constituents of wood due to their degradation (evaporation of volatile extracts, loss of products through thermal degradation etc.).

The thermal treatment has led to a reduction in the recovery rate of the pine and poplar plates of their original weight due to reduced moisture recovery, which is more pronounced as the treatment temperature and duration increase.

The EMC of all heat-treated pine and poplar samples was reduced compared to the corresponding control value, even in the case of the less intensive treatment (180°C-3h) and as the intensity increased, EMC decreased further (20.73%-38.43% for pine and 18.76%-49.20% for poplar). Thermal treatments reduced the density of pine (0.76-20.24%) and poplar (1.04-24.42%), and as the treatment intensity increased, larger reduction was recorded.

The hygroscopic properties of heat-treated pine were improved in every case due to thermal treatments. The most intensive treatment tended to slightly increase the adsorption and radial-tangential swelling rates, without approaching the control levels. Referring to poplar, all treatments, even the less intensive ones, improved the hygroscopic nature of wood, reducing the adsorption and tangential-radial swelling rates compared to the control sample. Therefore, confirming the results of literature, the thermal treatment was found to improve the hygroscopic behavior of pine and poplar and to further reduce tangential swelling levels in relation to radial direction, contributing to the limitation of wood anisotropy.

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