

Acknowledgement

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Pine wood modification by heat treatment in air

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Maritime pine (*Pinus pinaster*) wood has low dimensional stability and durability. Heat treatment was made in an oven using hot air during 2 to 24 h and at 170-200 °C. A comparison was made against steam heat treatment. The equilibrium moisture content and the dimensional stability (ASE) in radial and tangential directions were evaluated at 35%, 65% and 85% relative humidity. MOE, bending strength and wettability were also determined. At the same mass loss improvements of equilibrium moisture content and dimensional stability were higher for oven heat treatment but the same happened for mechanical strength degradation. A 50% decrease in hemicellulose content led to a similar decrease in bending strength.

Keywords: Bending strength, Dimensional stability, Heat treatment, MOE, *Pinus pinaster*, Wettability

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INTRODUCTION

Heat treatment is a wood improvement and preservation process that is facing a recent surge of interest. Despite having started in 1946 with the work of Stamm et al., it was only in the last decade or so that it was systematically researched and industrially applied in some European countries. There are different commercial heat treatment processes: the Finish process (Thermowood) uses steam (Viitanen et al. 1994), the Dutch (Plato Wood) uses a combination of steam and heated air (Tjeerdsma et al. 1998b), the French (Rectification) an inert gas (Dirol and Guyonnet 1993) and the German (OHT) heated oil (Sailer et al. 2000).

The heat treatment increases the wood value by decreasing equilibrium moisture content (Jämsä and Viitaniemi 2001; Wang and Cooper 2005; Esteves et al. 2007a, b), improving dimensional stability (Viitaniemi et al. 1997; Yildiz 2002; Wang and Cooper 2005; Esteves et al. 2007 a, b) and durability (Dirol and Guyonnet 1993; Kamdem et al.

2002) along with a decrease of the heat transfer coefficient (Militz 2002). The heat treated woods also acquire a darker color similar to most tropical woods which is an aesthetical advantage for some applications (Mitsui et al. 2001; Bekhta and Niemz 2003). The treatment is however detrimental to mechanical properties especially to static and dynamic bending strength (Yildiz 2002; Esteves et al. 2007 a, b), and also to compressive strength (Unsal and Ayrimis 2005).

When submitted to heating, wood changes its chemical composition through a thermal degradation that depends on temperature and time of exposure. For example, although wood presents a good thermal stability at 100 °C if the treatment time is long enough some chemical bonds begin to break, even for lower temperatures (Shafizadeh and Chin 1976). The temperature at which thermal degradation begins depends also on the wood species. For example, Kollmann and Fengel (1965) concluded that there was only mass loss for temperatures higher than 100 °C for pinewood, and 130-150°C for oakwood. The temperature at which the degradation starts depends on the molecular mass and crystallinity of the wood components (Belville 1982).

Maritime pine (*Pinus pinaster*) is a low valued timber species because of the relatively poor dimensional stability and durability of pinewood and the difficult preservation which is only possible for small diameters. Additionally it has an unappealing yellowish color. Heat treatment could improve some of these aspects and be a possible alternative to environmentally doomed chemical preservation treatments. The French process has already been applied to treat *Pinus pinaster* wood.

This paper presents results on the treatment of pinewood describing the properties change along the treatment, the decrease in equilibrium moisture content and increase in dimensional stability, the decrease in wettability and the degradation of mechanical properties mainly of MOE and bending strength. The selected treatment was heating in hot air at temperatures 170-200°C and variable duration leading to different treatment severity. The improvement of properties was related to mass loss. A comparison is made between this treatment and the steam heat treatment reported by Esteves et al (2007b) in the same conditions, therefore allowing analyzing the effect of an oxidative atmosphere which is likely to induce more intensive chemical changes on wood.

EXPERIMENTAL

Material and wood treatment

Pinewood samples were cut from the sapwood of a radial board of one maritime pine (*Pinus pinaster* Aiton.) tree from the Portuguese region of Águeda. Cubic samples with approximately 40 mm edge and samples with 360mmx20mmx20mm were cut with clear faces, kept in a conditioned room at 20°C and 50% relative humidity for 3 weeks and weighed afterwards. The equilibrium moisture content and the dry mass of the samples were determined. The heat treatment was made in an oven heated by electric coils located in the walls and with exhaustion of the heated gases by natural convection through an opening in the oven wall. The treatment was made for 2 to 24 h and at 170-200°C. The treatment started by putting the samples at ambient temperature in the oven, and the period to reach the treatment temperature was about 60 min. Four replicates were

used for each combination of time/temperature of treatment and for each sample size. After treatment, the samples were cooled down in a dry environment and weighed. Mass loss was determined in relation to initial dry wood. Untreated samples were used as the control.

Wood properties

Treated and untreated wood samples were kept in a controlled environment at 20°C and sequentially equilibrated at 35, 65 and 85% relative humidity for at least 4 weeks in each relative humidity and until the mass variation was less than 5% in two consecutive days. Mass was determined and the equilibrium moisture content was calculated. The samples dimensions were measured in radial and tangential directions for all the relative humidity and dimensional stability of the heat treated samples was calculated as an Anti Swelling Efficiency (ASE). ASE gives the difference between the swelling coefficient of treated and untreated samples, from oven dry to 35% (ASE35), 65% (ASE65) and 85% (ASE85) relative humidity in percentage of the swelling values

for the untreated samples. For example $ASE_{35} (\%) = \left(\frac{S_{nt} - S_t}{S_{nt}} \right) * 100$

where S_{nt} and S_t represent the shrinking between 35% relative humidity and 0% relative humidity for untreated (nt) and treated (t) samples. The shrinking is determined in percent as

$$S(\%) = \left(\frac{L_{35\%} - L_{0\%}}{L_{35\%}} \right) * 100$$

with L representing the dimension of the sample.

Wettability was determined by the contact angle method in tangential and radial sections, with the contact angle measured 10 seconds after the contact of a 10 μ l water drop with the sample. Distilled water was used in this test.

The mechanical properties were determined with samples of 360x20x20 mm³ (axial x radial x tangential) by a three point bending device. MOE measurements were made using a constant velocity of 0.3 mm/min and for bending strength the velocity was estimated to cause rupture in about 3 min.

MOE and bending strength were determined according to the Portuguese standard NP-619 as:

$$MOE(N/mm^2) = \frac{\Delta F * L^3}{\Delta x * 4 * b * h^3}$$

$$Bending\ strength\ (MPa) = \frac{3 * F * L}{2 * b * h^{\frac{10}{6}}}$$

where F is the load on rupture measured in N/mm, $\frac{\Delta F}{\Delta x}$ is the slope of the elastic zone in N/mm, L is the arm length, h the height and b the width, all expressed in mm.

RESULTS AND DISCUSSION

Mass loss

Figure 1 presents the mass loss with heat treatment for temperatures between 170-200°C along the treatment time.

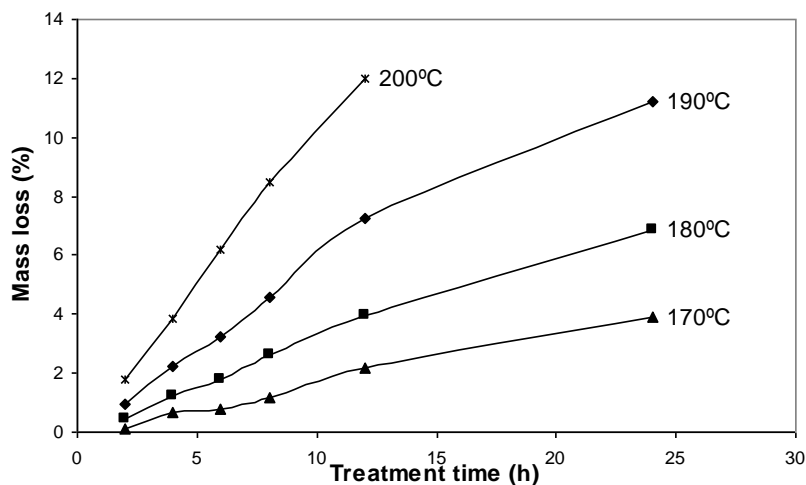


Fig. 1. Pinewood mass loss with heat treatment

Mass loss increased with the treatment time and with the temperature, and the same mass loss could be obtained with different temperatures, depending on the treatment time (Fig. 1). For example, a mass loss of 3% could be reached at 170°C in 17 h, at 180°C in 9 h, at 190°C in 5 h and at 200°C in only 3h. Similar results were reported by several authors, i.e. Zaman et al (2000) treated *Pinus sylvestris* and *Betula pendula* at temperatures between 200°C and 230°C during 4-8 h and determined that the mass losses for pine varied between 5.7% (4h) to 7.0% (8h) at 205 °C, and between 11.1% (4h) and 15.2% (8h) at 230 °C and for birch 6.4% (4h) and 10.2% (8h) at 200 °C and 13.5% (4h) and 15.2% (8h) at 220 °C. Alén et al. (2002) studied the mass loss of heat treated spruce at temperatures between 180°C and 225°C during 4 to 8 hours and concluded that they were between 1.5% at 180 °C (4 h) and 12.5% at 225°C (6h). At higher temperatures mass losses are quite higher; Bourgois and Guyonnet (1988) attained a mass loss of 18.5% in just 15 min, reaching 30% for 1 hour for maritime pine wood at 260°C. Órfão et al (1999) reported that *Pinus pinaster* wood starts to degrade at 140°C with or without the presence of oxygen.

The rate of mass loss was higher in the beginning of the treatment and decreasing for longer treatments. Since the mass loss showed an approximately linear variation with the treatment time until about 12 h, it was possible to adjust linear equations with statistically significant coefficients (R^2 between 0.972 and 0.998). The rate of mass loss (in h^{-1}) increased with the treatment temperature: 0.20 (170°C), 0.35 (180°C), 0.63 (190°C), 1.03 (200°C). The higher initial rate of mass loss was due to the thermal degradation of the more susceptible compounds, mainly hemicelluloses but also to the volatilization of some extractives as reported by Esteves et al (2007c). For example in a treatment at 190°C during 6 h the hemicelluloses content decreases 17.2% in relation to

the initial content and at the same time most of the original extractive compounds have disappeared (Esteves et al 2007c). Similar results for the degradation rate were also reported for the heat treatment of other species like cedar (González-Peña et al. 2004).

Mass loss of oven heat treated pine wood was higher than for autoclave steam heat treated pine wood at the same conditions as reported by Esteves et al. (2007b). As an example, for a treatment at 200 °C during 6 h the mass loss for pine wood treated in the oven was 6.2% while in the autoclave was only 3.5% (Fig. 1). These results are in accordance with Stamm (1956) who reported that wood degrades more in the presence of air due to oxidation reactions. It is also known that the acetic acid produced in this process acts as a depolymerization catalyst and it is possible that there is a higher content of acetic acid released on the oxidizing environment. Mazela et al. (2003) compared the mass losses with heat treatment in air or in an atmosphere with water vapor, using temperatures of 160°C, 190°C and 220°C during 6-24h and reported that the mass losses in the presence of air and water vapor for a treatment during 6 hours were similar but for 24h mass losses in air were much higher.

The extent of thermal decomposition is often measured by mass loss. In accordance to the Thermowood patent (Viitaniemi et al 1997), a mass loss of 3% is needed to improve wood dimensional stability and at least 5% to improve durability.

Equilibrium moisture content

The equilibrium moisture content of pine wood decreased with heating even for very short treatment times.

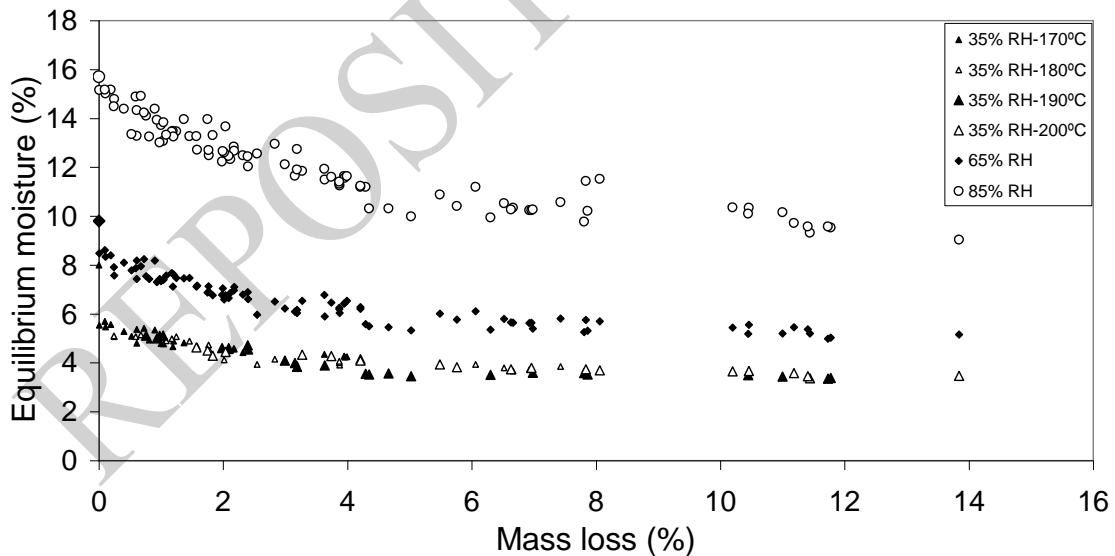


Fig. 2. Equilibrium moisture content of heat treated wood in relation to mass loss in different relative humidity environments.

Figure 2 presents the equilibrium moisture content at three different relative humidities (35%, 65% and 85%) as a function of mass loss. The equilibrium moisture content of heat treated pine wood decreased with the increase in treatment severity. The

rate of decrease was higher for lower mass loss reaching a minimum value for about 4% mass loss. The behavior was similar for the three relative humidity environments. Although the net reduction of equilibrium moisture content was higher for 85% relative humidity, the reduction in relation to untreated wood was higher for 35% relative humidity. These results are generally in agreement with Kamdem et al. (2002) for beech wood treated at temperatures between 200-260°C and conditioned at similar relative humidities (66% and 86%) and with Esteves et al. (2007a) for eucalypt wood.

A mass loss between 4-6% was enough to get the maximum reduction in equilibrium moisture and a higher treatment severity did not benefit the equilibrium moisture of wood (Fig. 2). Similar results were reported by Esteves et al. (2007b) with autoclave heat treated pine wood but in this case the minimum equilibrium moisture content was obtained at about 6-8% mass loss. This means that for a treatment with steam it is necessary to attain a higher mass loss to have a similar reduction on equilibrium moisture. This is possibly due to the somewhat different degradation reactions with heat occurring in air and in steam environment. Viitaniemi et al. (1997) also reported identical results for spruce wood, with the minimum equilibrium moisture content being reached for about 6% mass loss.

The reduction on equilibrium moisture content is due to several factors. The degradation of hemicelluloses, which are the most hygroscopic structural compounds, plays an important role but the degradation of the amorphous regions of cellulose and the cross-linking reactions also contribute to the decrease on equilibrium moisture content as reported by several authors (Bhuiyan and Hirai 2005; Tjeerdsma et al 1998a; Tjeerdsma and Militz 2005). Esteves et al (2007c) reported that hemicelluloses content decreased 17.2% and 10.4% in relation to initial content at about 3% mass loss for a treatment in air and in steam environment, respectively. A higher mass loss is needed for the steam treatment to attain the same hemicelluloses reduction and consequently a similar effect on equilibrium moisture.

The reasons for the apparent stabilization of the equilibrium moisture content for higher mass losses are not clear, although Bhuiyan and Hirai (2005) refer that cellulose crystallinity decreases for more severe treatments which might increase the accessible hydroxyl groups.

Dimensional stability

The heat treatment improved pine wood dimensional stability even for short time treatments, increasing with time and temperature of treatment. For example the radial ASE₃₅ of heat treated wood was 57% with 8 h at 170 °C, 4 h at 180 °C or 2 h at 190°C. The maximum values reached were between 63-73%. Similar results were reported by several authors, i.e. Yildiz (2002) with beech wood treated at 130-200°C during 2-10 hours.

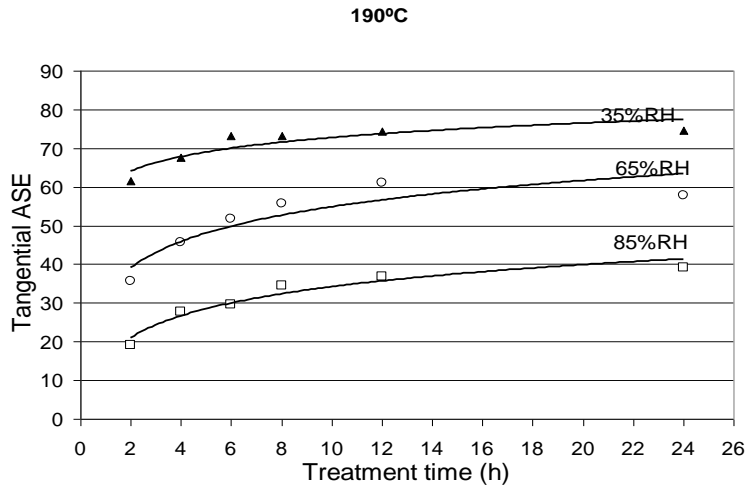


Fig. 3. Relationship between tangential ASE and treatment time for 35, 65 and 85% relative humidity. Each point is an average of 4 samples.

The improvements on dimensional stability were higher for lower relative humidities (Fig. 3). For example at 65% RH, the tangential ASE ranged between 25 and 38% reaching a maximum of 62% while for 85% RH, the maximum tangential ASE was 44%. The increase in dimensional stability is mainly due to the decrease in equilibrium moisture content.

Figure 4 shows the variation of radial and tangential ASE with mass loss, at 35% relative humidity. The improvements were slightly higher in the tangential direction with ASE₃₅ ranging from 73 to 80% for treatments at 170-200°C. Although stability improved more in the tangential direction, the swelling of treated wood samples remained higher than in the radial direction. The behavior was similar for 65% and 85% RH. Analogous results were reported by Tjeerdsma et al. (1998a) with beech, birch, spruce, Scots pine and Monterey pine.

For outdoor furniture it is important to have similar radial and tangential swelling, that is to say, a low anisotropy; therefore the decrease in anisotropy with the heat treatment as given by the comparatively higher increase in tangential ASE is an advantage for this type of wood use.

The increase in dimensional stability was higher for smaller mass losses until about 4%. The results were similar for both directions but with slightly higher values in the tangential direction.

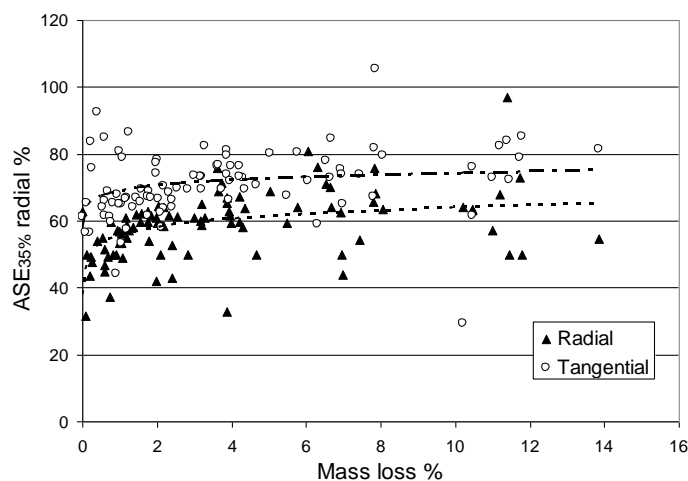


Fig. 4. Variation of radial and tangential ASE with mass loss at 35% relative humidity

The maximum ASE values were obtained at a mass loss of about 4-6%, and only in a few cases a small increase of dimensional stability was observed for higher mass losses. These results are in accordance with those reported by several authors i.e. Viitaniemi et al. (1997) with spruce wood, in which the maximum ASE was obtained for mass loss between 5-6% and Esteves et al. (2007b) for autoclave heat treated pine and eucalypt wood for 6-8% mass loss.

Wettability

The surface wettability in relation to mass loss for radial and tangential sections is presented in Figure 5. The contact angle increased, and the wettability decreased, until about 3% mass loss for both sections, and after that stabilized for higher mass losses. Similar results were reported by Pecina and Paprzycki (1988) with Scots pine and Hakkou et al. (2003) with poplar, beech, spruce and Scots pine. The wettability decrease is due to the degradation of the most hygroscopic compounds, hemicelluloses and amorphous cellulose, but also to dehydration reactions. The change of the extractive composition might also play an important role on wood wettability. At 3% mass loss according to Esteves et al (2007c) most of the original pinewood extractives have disappeared and new ones have been formed. The new extractives formed are mainly some phenolic compounds and anhydrosugars.

An increase of wettability for higher mass losses is possible due to the degradation of macromolecular compounds as mentioned by Pecina and Paprzycki (1988). The differences in extractive chemical composition between 3% and higher mass losses, reported by Esteves et al (2007c) could also contribute to a change of wood wettability.

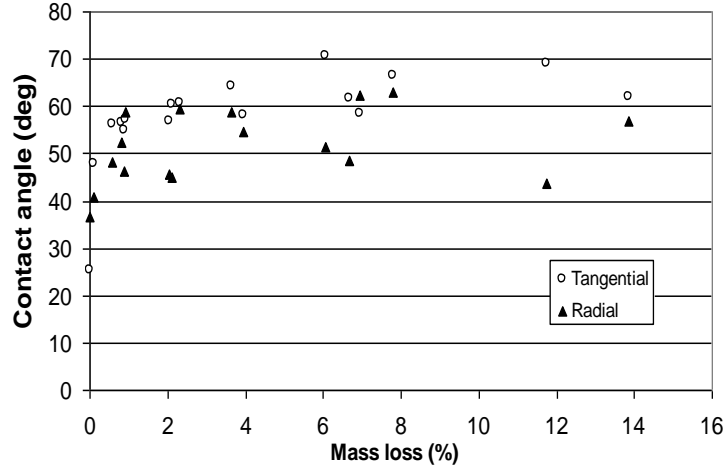


Fig. 5. Contact angle on tangential and radial sections in relation to mass loss during heat treatment .

Although wettability of untreated pine wood is higher for the tangential section, there are no significant differences between sections for heat treated wood.

Wettability influences gluing and finishing, mainly by increasing the absorption time of glues and varnishes; however, according to Vernois (2000) some varnishes can be adapted to this type of wood.

Mechanical Properties

MOE and bending strength decreased with heat treatment time and temperature. With 2 h of treatment, the MOE reduction was very small, with 2% (180°C), 0% (190°C) and 0% (200°C), and reached 6% (180°C), 12% (190°C) and 19% (200°C) with 12 h of treatment. The modulus of elasticity of pine wood decreased with mass loss during the heat treatment (Fig. 6). The decrease was less than 5% until about 4% mass loss, but increased subsequently and attained 16% for about 6% mass loss. Although the MOE decreased with heat treatment, at the mass loss necessary to obtain the maximum improvement on equilibrium moisture and dimensional stability (4-6%) the decrease was under 10% which is not significant. Yildiz et al. (2002) reported a decrease in MOE of about 45%, for beech wood treated at 130-200°C for 2-10 h but mass loss was not referred. Results reported by Esteves et al. (2007b) with steam heat treated pine wood showed a small increase until about 4% mass loss, followed by a decrease for higher mass losses. With the same treatment conditions, heating time and temperature, the reduction of MOE was higher for the treatment in air and the same happened when comparing at the same mass loss.

Bending strength of untreated pine wood was, on average 154 MPa, varying between 138-171 MPa and decreased in the heat treated pine wood samples more than the MOE. The relative decrease of bending strength was between 4- 38% with only 2 h of treatment at 180-200°C and 31% (180°C), 58% (190°C) and 58% (200°C) with 12 h of treatment.

The rate of bending strength decrease was higher for small mass losses, about 40% for 3% mass loss, decreasing afterwards, but reaching 60% for mass losses above 6% (Fig. 6). The reduction of bending strength was higher than the reported by Kim et al. (1988) for radiata pine treated at 180 °C during 2 h with a reduction of the modulus of rupture (MOR) of only 21% for dry wood and 27% for green wood. Bengtsson et al. (2002) obtained a similar reduction in bending strength of 50% for spruce wood and 47% for Scots pine wood treated at 220 °C. The reduction on bending strength is mainly due to the degradation of hemicelluloses. The close relationship between hemicellulose content and bending strength was also reported by several authors (Winandy and Morrell 1993; Winandy and Lebow 2001). For 7% mass loss only about 50% of hemicelluloses remained in wood which has a high impact on bending strength (Fig. 6).

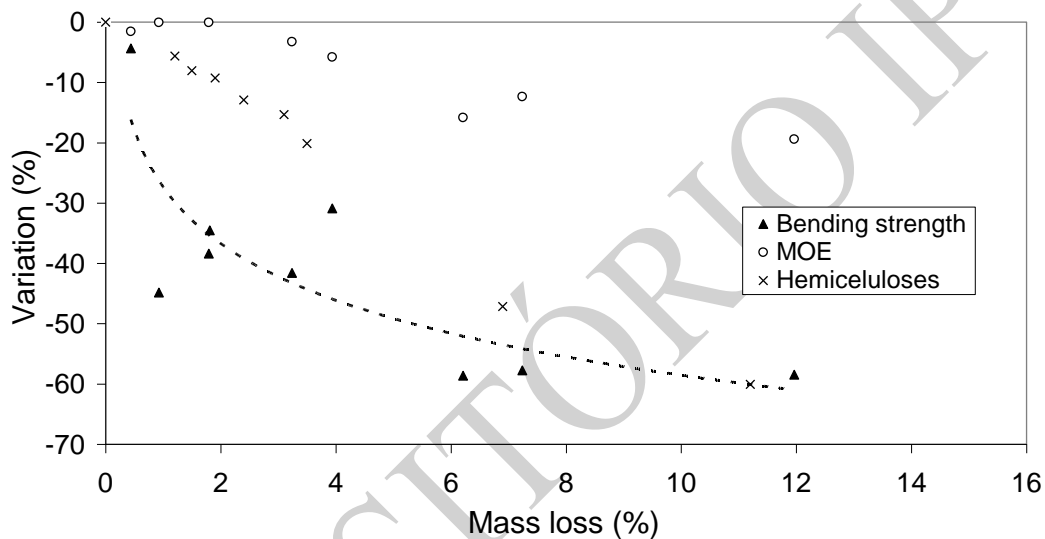


Fig. 6. Variation of bending strength, MOE and hemicelluloses content with mass loss during heat treatment. Each point is the average of 3 samples.

At about (4-6%) the reduction of bending strength would be about 40-60% higher than the reported by Esteves et al. (2007b) for steam heat treated pine wood (25%). It seems that a treatment with steam affects less the mechanical properties of wood with the same mass loss. Nevertheless, at the same mass loss the degradation of macromolecular compounds is different for wood treated with hot air or with steam. For example, at about 3% mass loss there is a decrease of hemicelluloses content of 17.2% and 10.4% for oven and autoclave treatment, respectively (Esteves et al 2007c).

CONCLUSIONS

1. The heat treatment of pine wood improved some of its properties: equilibrium moisture content decreased, the dimensional stability increased and the anisotropy

and the surface wettability decreased. In relation to mechanical properties, MOE was little affected but bending strength decreased much more.

2. Mass loss of oven heat treated pine wood was higher than for steam heat treated pine wood at the same conditions.
3. At the same mass loss equilibrium moisture content decreased more than for the steam treatment due to the higher degradation of hemicelluloses and amorphous cellulose.
4. The oven heat treatment improved more the dimensional stability but also affected more the mechanical properties of wood than steam heat treatment with the same mass loss, possibly due to the oxidation reactions and to the higher hemicellulose degradation. A 50% decrease in hemicellulose content led to a similar decrease in bending strength.

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