Experimental study of thermal treatment in variable pressure conditions

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ABSTRACT

The paper reports preliminary results of tests on spruce and other species thermally treated by exposing the material to temperatures in the range of 140-180°C in saturated vapour pressure and in vacuum conditions under a convective heat transfer source.

Comparative data concerning drying quality, colour coordinates and residual physical and mechanical properties of treated wood are reported and discussed.

INTRODUCTION

The thermally modified wood has been on the European market for 10-15 years, above all for exterior use products because of its improved durability when treated at temperatures not lower than 212°C. Others favourable modifications concern the reduction of the equilibrium moisture content and the increase of the dimensional stability. Finally, the thermal treatment modifies the colour of wood which turns to dark brown-reddish depending on the temperature and time of exposition. On the other hand it is known that the thermal treatment reduces the mechanical properties of wood.

In Italy the colour modification makes the thermal treatment a very interesting process on the market of flooring and furniture even if the high price of energy and of treatment plant itself has discouraged the industry which results in a few thermal treatment plants working in Italy at present.

Moreover, some other known problems such as quality of drying at very low final MC for large thickness sawn timber, decrease of mechanical properties, instability of colour under UV irradiation but even durability of wood in specific exposure conditions still affect the demand and use of the thermally treated wood.

The thermal treatment is performed in special kilns where the wood is exposed to high temperature in modified atmosphere conditions. During the first phase of the process the wood is dried at 100-130°C and the MC decreases to nearly 0%. In the second phase the heat treatment takes place for some hours and finally in the third phase the temperature is lowered and the MC is increased to 4-7%.

In order to avoid fire risks and to reduce thermal oxidative degradation phenomena, the oxygen must be substituted with another gas, usually vapour (superheated steam at atmospheric pressure) or nitrogen.

The effect of various substitutive atmospheres was explored in this work. Treatment tests were performed at high temperature under high pressure in saturated vapour conditions and under vacuum using two different plants: a special corrosion and pressure resistant autoclave and a vacuum dryer designed for high temperature. The paper reports results of thermal treatment tests performed at CNR-IVALSA on spruce sawn timber of different thickness. Various combinations of temperature in the range from 140 to 200°C and time of exposition were used and the main physical and mechanical properties of the treated wood were measured.
The objective is to study the influence of the variables on the residual properties of the material and to compare the performance of different treatment processes.

METHODS

HT high pressure treatment (HT HP)

The laboratory plant used for the tests (Fig. 1) is composed by a small stainless steel autoclave (about 120 x 40 cm) with double wall. The internal wall of the cylinder is 6 mm thick. The gap between the two walls is filled with diathermic oil heated with an electric heater (resistance ?). The oil heats the internal wall of the cylinder and water located on the bottom of cylinder. Wood samples were placed inside the cylinder with metal sticks among the layers of stack. The cap of cylinder has a sealing-screw device which allows keeping the pressure within the cylinder in safe condition.

FIGURE 1. Schematic picture of the plant

The treatment has three phases: a warm-up phase needed to reach the schedule temperature within the cylinder, a treatment phase at constant conditions for a given time, and a cooling off phase, performed expelling the vapour and decreasing the temperature to 100°C. At the beginning, before the warm-up phase, the air is evacuated from the autoclave with a vacuum pump. The duration of the treatment (exposition time) is calculated since the pressure reaches the set value.

The saturated vapour conditions are reached keeping the water on the bottom of the cylinder at a given boiling temperature. In close conditions the boiling temperature and the saturated vapour pressure has an exponential relationships as showed in Figure 2.

FIGURE 2. Saturated vapour pressure vs. boiling temperature

This process does not dry the wood when it reaches a MC equilibrium with the saturated vapour at a given temperature (EMC in the range of 12-16%). For this reason, theoretically, it offers some advantages, above all treatment of large size timber which is not possible to treat with super-heated thermal treatment process because the drying phase produces high internal stresses and consequent degradations of the wood quality. Moreover, an industrial plant for saturated vapour treatment is probably less expansive than a superheated vapour plant. On the other hand, energy required for saturated vapour plants is probably higher and in some countries the use of equipment working at high pressures is restricted or requires special licence.

High pressure treatment tests were carried out treating the wood samples of different thickness and initial MC (MCi) at different combinations of pressure-temperature (p) and time of exposition (t) at a given pressure-temperature. The following combinations of variables were tested:

- s: thickness (30, 200 mm)
- t: exposition time (1,2,4,6,12 h)
- p: pressure-temperature (2,4,6 bar)- (120,150,170 °C);
- MCi: initial MC grouped in 4 classes:
  I: <18%; II: 18-22%; III: 23-28%; IV: >28%.

HT vacuum treatment (HT VP)

The plant is similar to a normal vacuum plant but designed to work at high temperature (max 250°C) with use of diathermic oil. The convective heat transfer is provided by two fans with external motors equipped with special mechanical transmission. Speed of fans is proportional to the vacuum pressure ranging from 635 rpm at atmospheric pressure to a maximum speed of about 1930 rpm at a vacuum pressure of 200 mbar. The characteristic curves of the fan at the maximum speed and at different vacuum and temperature conditions are reported in Figure 3. The vacuum pump is water ring type equipped with a heat exchanger.
The treatment consists in two phases:

1) Drying consisting in: a) a warm-up stage with a pressure kept below the boiling temperature ($T_b$) and vacuum conditions. The drying force [4] mainly depends on the superheating temperature ($\Delta T = T_h - T_b$) where $T_b$ is the boiling temperature which is a function of vacuum condition (see Fig. 4). In pre-dried material usually the set heating temperature was kept at 70°C for oak and refractory woods and 80°C for softwoods. The vacuum was set in the range 150 – 350 mbar which determines a $\Delta T$ in the range from 6 to 15 °C.

2) Thermal treatment (see Fig. 5): starts when the MC is 5%. It consists in a) a heating phase up to the treatment temperature ($T_t$) 6-10 h long, b) a high temperature heating treatment (from 4 to 8 h) phase at a $T_t$ in the range 170-210°C and c) a cooling phase down to 60°C. All the treatment phases are carried out under vacuum conditions and without use of steam. At the end of the process the MC of wood is 0%.

**RESULTS**

**Colour**

Statistics of the colour coordinates of untreated woods are reported in Table 1. In the Table 2 the global influence of the process variables temperature ($T$) and time ($t$) of
exposition (hours) in terms of $\Delta E$ measured at the inner core ($\Delta E_{core}$) and at the surface ($\Delta E_{surf}$) of high pressure treated spruce are reported. In Table 3, $\Delta E$ of surface and core are reported for HT VP treated species.

The initial MC did not influence the colour modification and it is neglected in the results description.

![Table 1. Colour coordinate of untreated woods](image)

![Table 2. Spruce in high pressure. $\Delta E$ for surface and core of sawn timber treated at different T-t (°C-hours) combinations.](image)

![Table 3. HT vacuum. $\Delta E$ for surface and core of sawn timber treated with different temperatures (HT4=180 °C-3 h; HT5=180 °C-4h; HT6=180 °C-5h).](image)

![Table 4. HT vacuum treatment. Colour modification at the surface for different species; average values for treatments HT 4-6.](image)

The treatments at high temperature make the wood darker (decrease of $L$) and in general make it reddish brown. The effect on global colour modification ($\Delta E$) is significantly higher for high pressure than for vacuum treatment at the same temperature. Moreover the modification of colour in HT HP starts at lower temperature for high pressure treatment: a temperature of 130°C is sufficient to produces colour modification in high pressure, while it does not produce any significant colour modification in vacuum condition. After 3 h of exposure at 170°C (6 bar) the surface of the wood starts to char.

**Physical properties**

A comparison of effect of the two treatment schedules (at identic conditions, i.e. 160°C 2 h) are summarized in Table 5. It can be observed that all the measured physical characteristics of all wood species are modified by the thermal treatment.

A clear positive influence of both treatments on the dimensional stability can be observed. As expected this is mainly due to the hygroscopicity which is reduced with approximately 20-30%. Another positive influence of the treatments on the dimensional stability is the change of anisotropy ratio.

The treatments modify the density of wood. As previously observed [3], the reduction of density is strictly related to a significant decrease of the mechanical properties of wood. For spruce HT HP treated at 150°C for 4 h the following mechanical properties reductions were observed: modulus of elasticity (-10%), compression strength (-9%), bending strength (-19%).

At identical temperatures and exposure time the reduction of density was nearly double in HT HP treated spruce compared to HT VP.

**Other observations**

The drying quality, i.e. distortion, splits and residual internal stresses was good for both treatment types but also for 200-mm thick HT HP treated spruce. As expected, refractory woods (oak) must be pre-dried to 15-20% MC before vacuum drying. On the contrary, HT HP treatment does not require a drying treatment (at least for spruce) and the wood can be directly treated without drying; the effect of the initial MC is negligible. However, it can be supposed that refractory woods (still not tested in HT HP process) need more attention during pre- and post treatment.

The HT VP treated wood did not have any odour after the treatment. This could be due to the work of the vacuum pump that evacuates the volatile substances during the treatment.

Results of reaction fire tests and durability tests with *Hylotrupes bajulus* on HT HP treated spruce did not show any difference between treated and untreated wood. More durability tests are planned.
CONCLUSION

Both thermal treatment tests showed interesting advantages. The main advantage of the HT HP treatment under saturated vapour condition is that wood does not dry to a very low MC, thus indicating that large thickness sawn timber could be treated. The treatment significantly modifies the physical properties and the colour even at relatively low temperatures (effects start at 140°C) and the time of treatment is short. On the other hand, the HT HP treatment is not probably convenient for temperatures higher than 180°C because of the high pressures. At the moment no results are available for other species.

The HT VP treatment produced moderate colour modification and total absence of the typical unwanted odour of the treated wood. The vacuum drying process before the thermal treatment offers short drying time and a high drying quality. The vacuum conditions during the exposure of wood at high temperature are probably more protective against the oxidative degradation compared to the thermal treatments in vapour. There is still not sufficient experience to evaluate the resistance of thermal vacuum plant to aggressive conditions. In particular a weak point can be the complicated ventilation system constructed with exterior motor fans and a high technological mechanical transmission that must be temperature and vacuum proof.

Ongoing tests are still in progress. They will provide further information of the effect of vacuum and other treatment variables. Laboratory tests are planned to measure mechanical and physical properties, durability and colour performance of different woods modified under various thermal treatment.

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<th>α&lt;sub&gt;V&lt;/sub&gt;</th>
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Table 5, Average physical properties of treated and untreated wood and relative variation: normal density (ρ<sub>norm</sub>), oven-dry density (ρ<sub>0</sub>), MC at normal conditions (MC<sub>norm</sub>), volumetric swelling from oven-dry to normal conditions (α<sub>V</sub>); volumetric swelling coefficient in the range oven-dry – normal conditions (α<sub>V/ MCnorm</sub>); anisotropy ratio (α<sub>T/αR</sub>).

REFERENCES