Thermal treatment of hardwood species from Italian plantations: preliminary studies on some effects on technological properties of wood

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ABSTRACT

High Temperature Thermal Treatment (HTTT) is generally used in order to improve both the technological performances of the raw material and its aesthetic appearance; as a matter of fact, wood modification by HTTT is well known as an interesting solution for improving the dimensional stability, the durability and the uniformity of the colour of the solid wood of coniferous species with limited decrease of the mechanical properties (Jämsä and Viitaniemi 2001). At present in Italy the aim of HTTT is to reach more interesting and remunerative uses of low quality hardwood, with potential applications for solid wood panels, flooring, furniture and cabinets or other uses in indoor conditions.

This paper reports a preliminary evaluation on the effects of HTTT carried out on two Italian hardwoods (European walnut and cherry) in terms of modification of some technological properties of wood, such as surface hardness, durability against insects and dimensional stability. Surface hardness of treated wood has been compared by applying the standards EN 1534 (Brinell method) and ISO 3350 (Janka method). Tests of durability against insects referred to EN 350-1, EN 20-1 and EN 46-1 standards and EN 335 for use class of wood assortments. All treated materials have also been characterised in terms of bonding quality by verifying the strength of the wood-to-wood joint when glued with polyvinyl acetate emulsions belonging to the D2 and D3 class of durability of the standard EN 204.

Dimensional and shape stability were then evaluated according to EN 1910, measuring the linear variations and warping of treated wood between dry (20°C, 30% RH) and wet conditions (20°C, 80% RH).

Cherry and walnut sawnwood treated with high temperature displayed notable changes in their properties. Hygroscopicity was reduced, and results also revealed an oven dry density reduction, a bending strength decrease, a modulus of elasticity increment in treated wood and an increase in surface hardness. After one month from the beginning of tests, the durability of cherry and walnut against insects was not modified by the thermal treatment. The HTTT in walnut reduced the quality of gluing with PVAc-dispersed products belonging to D3 and D2 classes of durability of EN204, but the application of acetone appreciably increased the performance of the D2-class adhesive. Finally, the thermal treatment stabilized the wood, and the varnish layer could contribute in the same way.

HTTT seems to be a good solution to reach more interesting and remunerative uses of the examined timber material and a method worthy of further studies.

Introduction

The Italian plantation programs, which began about 20 years ago, under the impulse of public financial contributions destined by the European Community policy to rural development, involved mainly four wood species (in order of importance): walnut, cherry, ash and maple. Recent estimations show that hardwood plantations in Italy, excluding poplar, extend for a total surface of about 35000 ha (INFSC, 2005). Highly selected timber from these hardwoods normally reach the highest prices in the local market, especially for furniture and decorative applications. On the contrary, all the timber derived from thinning does not achieve interesting economic value because it is affected by small diameters, a great number of knots, juvenile wood and other traits of low quality wood.

Costs of thinning are then higher than income and often the operation has not been carried out, with bad effects on the evolution of the plantation as a whole. In this framework, the goal of the paper is to search for a treatment that could help to promote and add more value to this material through the changing of some of its critical properties. HTTT is well known, in fact, as an innovative method able to modify the properties of wood, having the main effect of reducing its hygroscopicity (Tjeerdsma *et al.* 1998). This modification leads directly to an increased resistance to different agents of biodegradation and to an improved dimensional and shape stability. On the contrary there are some undesirable effects, such as loss of strength and an increased brittleness that prevent some utilization of the resulting products (Jämsä and Viitaniemi 2001; Repellin and Guyonnet 2003). Since the experience with HTTT up to now is well established only for softwoods, in this context the paper aims to evaluate the effects of the above treatment on some technological properties of European walnut (*Juglans regia* L.) and cherry (*Prunus avium* L.) sawnwood.

Materials and methods

A total of 15 walnut and 15 cherry logs were derived from thinning in a mixed plantation located in Central Italy; the age of the selected trees ranges from 20 to 22 years, with an average DBH of 23 cm for cherry and 21 cm for walnut. The basal log, 150 cm long, was cut from each tree: this material was then sawn to boards with a thickness of 30 mm which were further conditioned at 20°C and 65% relative humidity (RH) for 1 month. The thermal treatment was performed at an experimental drying chamber built by an Italian company (named Baschild and located near Bergamo) specializing in the design and production of wood kiln dryers. The capacity of the chamber is approximately 4 m^3 of lumber. The thermal process, originally developed by the Italian company, consists of four sequential steps with different temperature and moisture conditions which the material is maintained for a specific time (Figure 1); the temperature settings range from 70 to 210°C and the total cycle lasts 45 hours. During the first phase, wood is warmed up to a temperature of 70°C for about 4 hours; then temperature raised to 100°C and maintained constant for about 6 hours, during which the wood moisture content decreased to 3 to 4%. The real thermal treatment takes place in the following step when temperature reaches 210°C and is maintained for 24 to 25 hours while air in the kiln dryer is saturated by steam.

Finally, a cooling period at 50°C for about 10 hours is needed to stabilize the wood that, when extracted from the chamber has a moisture content of 2 to 3%, and also to prevent the evolution of defects on the heat treated wood assortments.

The Baschild method is similar to the Finnish process Thermowood® (Syrjänen, 2001), except for the heat treatment phase that is longer and characterized by the presence of a tank, placed inside the heat chamber, containing water for the production of steam.

The treated material was then conditioned in a climate chamber at 20°C and 65% RH for four weeks.

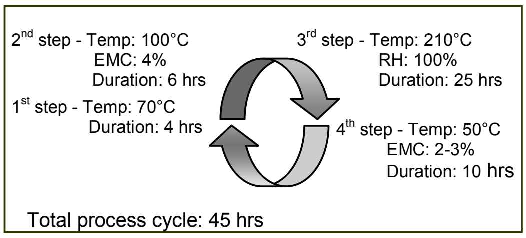

Figure 1 ***The high temperature treatment cycle***

Physical and mechanical tests

Sampling methods and general requirements for the physical and mechanical tests were based on ISO 3129. Small clear samples of untreated and treated walnut and cherry sawnwood were obtained for density (ISO 3131), bending strength and modulus of elasticity (ISO 3133), surface hardness according to EN 1534 (Brinell method) and ISO 3350 (Janka method) (Table 1).

Table 1 ***Summary of materials and methods for physical and mechanical tests***

PROPERTIES	SAMPLES DIMENSIONS	NUMBER OF SAMPLES
Density (ISO 3131)	20x20x50 mm	60
Bending strength and modulus of elasticity (ISO 3133)	20x20x320 mm	70
Static hardness (ISO 3350)	50x50 mm	40
Resistance to indentation (EN 1534)	50x50 mm	40

The tests were performed on a Galdabini testing machine PMA5. All specimens were loaded under displacement control. For all specimens the load and the crosshead displacement of the testing machine were recorded continuously.

After the mechanical strength test the moisture content of the samples was measured according to ISO 3130.

Durability against insects

The durability test against insects was evaluated as a function of the final use classes of the wooden products obtained from the HTTT hardwood: panel, flooring, cabinets and other objects for indoor uses. These destinations correspond to use class 1 in accordance with EN 335. All the wood species were tested against the Cerambycid *Trichoferus holosericeus* (Rossi); only walnut was also tested against *Lyctus brunneus* (Stephens). *T. holosericeus* is a Cerambycid widespread in the South European countries and Mediterranean basin and is specific to hardwood species. In Italy woods attacked by *T. holosericeus* are in order of preference: oak, black locust, beech, poplar, walnut, chestnut, alder, cherry (Palli and Gambetta 1962). The susceptibility to *L. brunneus* is correlated with specific anatomic features of the wood species: this insect can only attack hardwood species with sufficient starch content (ca > 3%) and with vessels of adequate size to allow female ovipositor within them (Eaton and Hale 1993).

The test against *T. holosericeus* was performed in accordance to EN 46-1. This standard describes a method for the determination of the preventive action of a treatment against recently hatched larvae of *Hylotrupes bajulus* (L.) and it is also used for the determination of the conferred durability by a treatment in accordance with EN 350-1. We applied the same methodology to the Cerambycid *T. holosericeus*. Preliminary results were observed after four weeks concerning the mortality rate and the tunnelling of the larvae.

The resistance to *L. brunneus* test was carried out in accordance with EN 20-1 and EN 350-1. At the end of the test the results are expressed by the number of new insects at different stages of development (adult, pupae, larvae) and the number of flight holes. Preliminary evaluation was carried out after one month from the beginning of the test with a visual examination of the surface tunnelling and frass production.

Quality of gluing

The tests were carried out on the treated walnut according to EN 205 (tensile shear strength on lap joints for adhesives for non-structural applications). Specimens were cut from small thin boards of the approximate size of 500 x 130 mm. Thin boards thickness was 5.4 mm and it was obtained using a sandpaper-calibrating machine. However, just prior to gluing the surface was renewed by 120-grit hand sandpapering. The other gluing conditions were as follows: pressure 0.3 bar, single spreading of 150 g/m^2, open time less than 1 min, closed time 3 min, pressing time 2 hours. Two types of polyvinyl acetate (PVAc)-dispersion adhesives were used for gluing the thin boards, belonging to the D3 and D2 classes of durability considered in EN 204 (basically corresponding to interiors, respectively frequently and occasionally exposed to water and high RH). In particular, the products were KM (class D2) and 2252M (class D3), both provided by the company Vinavil. For each test, the thermally treated walnut was divided in two groups: one was glued as such, whereas the other was abundantly wetted by acetone just before spreading the adhesive. This procedure was chosen, considering the well known decrease in water wettability of the thermally modified wood (Follrich *et al.* 2006), in order to swell the wood and therefore to promote the penetration of the glue in the substrate. In the case of the D3-class adhesive, tests were also carried out on pieces of untreated walnut of the same provenance of those thermally treated.

Dimensional stability (and varnishing)

The aim of the tests concerning dimensional stability was to evaluate, through a comparative approach, the behaviour of treated wood during climatic variations of temperature and RH. This characteristic of the material is one of the most important for wood flooring applications.

After sanding, treated and not treated walnut and cherry sawnwood were varnished on an industrial painting line. The applied product was a layer of acrylic-polyester UV varnish, as an insulating layer (80 g/m^2), and an acrylic transparent finish (100 g/m^2). This coating system, based on a modified acrylic varnish, demonstrated an extreme resistance to light and abrasion and was especially formulated for wood surfaces with problems of wetting due to the presence of oil or natural extractives.

In order to check the wood dimensional stability, samples of 250x70x15 mm were exposed to different climatic conditions according to EN 1910. The samples, after one month at 20°C and 65% RH, were located in a conditioning room maintained at 20°C and 30% RH for 14 days, then to 20°C and 80% RH for 12 days.

The determination of the geometrical characteristics, carried out at the end of each conditioning time, was done according to EN 13647.

Results and discussion

Physical and mechanical properties

The results from physical and mechanical characterisation clearly underline the effects of the HTTT on the technological behaviour of walnut and cherry sawnwood. Previous studies showed that strength and density of the wood decrease with thermal treatment while this improves its dimensional stability (Jämsä and Viitaniemi 2001). Other works (Stamm and Hansen 1973) also determined a clear decrease in hygroscopicity of the wood material by applying the treatment to dried wood. This fact is due to chemical modifications in the wood structure occurring at high temperature (Tjeerdsma *et al.* 1998).

Yildiz (2002) and Sundqvist (2004) reported that crystallinity of the cellulose in hardwood samples increases with thermal modification. This effect is not only related to the temperature but is also time dependent. The reduction of the hygroscopicity is confirmed by the lower value of the equilibrium moisture content (EMC) reached by the heat treated wood after 4 weeks at 20°C and 65% RH. Normally for solid wood this value is around 12% while in this case we obtained 4%. Mass loss in wood material is another disadvantage of HTTT (Table 2). In line with the results achieved in a recent study on Turkish river redgum (*Eucalyptus camaldulensis* Dehn.) heated at a similar temperature of 180°C for 10 hours, oven dry density losses were 3.9%, and 4.2%, for cherry and walnut, respectively (Unsal *et al.* 2003; Unsal and Ayrilmis 2005).

Table 2 ***Density measurements of treated and untreated material at EMC (65% RH; 20°C) and oven dry (ρ_0)***

WOOD MATERIAL	DENSITIES	MEANVALUES g/cm^3	SD g/cm^3
Untreated Cherry	ρ_{EMC} ρ_0	0.608 0.571	0.03 0.03
Treated Cherry	ρ_{EMC} ρ_0	0.588 0.549	0.05 0.05
Untreated Walnut	ρ_{EMC} ρ_0	0.723 0.669	0.07 0.07
Treated Walnut	ρ_{EMC} ρ_0	0.679 0.641	0.05 0.06

Important changes in the mechanical properties of treated wood were also observed. Results revealed that bending strength decreases because of the treatment, while modulus of elasticity together with Brinell and Janka hardness increase. The maximum reduction in bending strength, compared to the untreated samples, was obtained for treated cherry (13.6%), while for the treated walnut the resistance is reduced to 6.9%. On the basis of Student's t-test changes in strength values are significant. At the same time modulus of elasticity increased up to 21.4% and 4.7% for cherry and walnut, respectively. These results can be explained by cellulose and hemicelluloses degradation due to the high temperature process (Yildiz 2002). Consequently the treated wood became a more brittle material than the untreated one of the same species. The increased brittleness and hardness of treated wood is confirmed by the diagrams showed in Figure 2 and Figure 3.

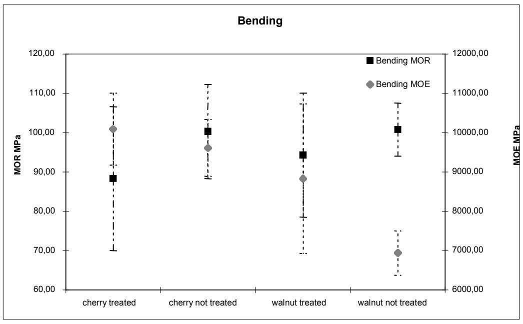

Figure 2 bending MOR and MOE of treated and untreated material

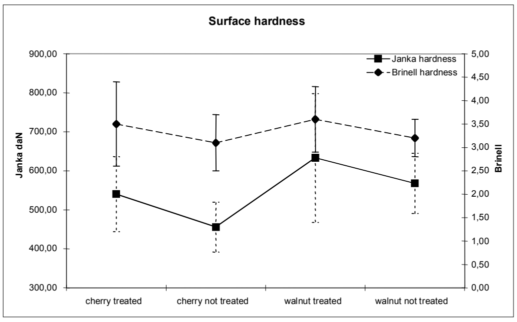

Figure 3 Janka and Brinell hardness of treated and untreated material

Several interesting results are evident. There is a significant difference in failure condition between treated and untreated wood. The load-deflection curve for a three-point bending test according to ISO 3131 for treated walnut shows, after a first linear region of elastic behaviour, a little region of apparently plastic deformation followed by a fast and sudden fracture of the specimens, whereas the typical curve for untreated walnut shows a defined and clear region of plastic deformation (Figure 4). The same trend was obtained for cherry.

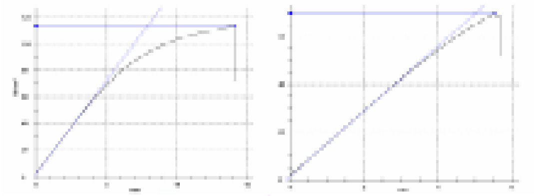

Figure 4 Load-deflection curve for three-point bending test according to ISO 3131 for the untreated (left) and treated (right) walnut

Durability against insects

Against *T. holosericeus*, preliminary results recorded after four weeks from the beginning of the test concerning tunnelling and the mortality rate of the larvae are shown in Table 3.

***Table 3 Preliminary results of the durability test against* Trichoferus holosericeus**

WOOD (NR. OF REPLICATES)	INITIAL NR. OF LARVAE	NR. OF ENTRANCE HOLES	DEAD LARVAE	LARVAE NOT RETRIEVED
Untreated Cherry (6)	60	39	3	18
%	*100*	*65*	*5*	*30*
Treated Cherry (6)	60	47	8	5
%	*100*	*76*	*16*	*8*
Untreated Walnut (6)	60	31	13	17
%	*100*	*51.7*	*21.7*	*26.7*
Treated Walnut (6)	60	34	5	21
%	*100*	*56.7*	*8.3*	*35.0*
Untreated Beech (3)	30	23	3	4
%	*100*	*76.7*	*10.0*	*13.3*

We can observe that the test is valid according to the standard because more than 70% of the larvae placed on the untreated beech had tunnelled. These observations are based only on the visual control of the wood blocks' surface where the newborn larvae were placed at the beginning of the test: we can recognize the number of larvae that have tunnelled by the small quantity of wood dust at the tunnel entrance and the dead larvae on the surface of the wood blocks. Based on these results we can affirm that there are no differences between untreated and thermally modified wood regarding the resistance against newly hatched larvae of *T. holosericeus*.

Concerning the resistance against attack of *L. brunneus*, preliminary results observed one month after the beginning of the test showed that there is not a clear and marked difference between untreated and treated walnut blocks. In fact all the wood blocks showed the sign of a new generation: the presence of wood dust in the bottom of containers. More reliable results will be available after ten weeks from the beginning.

Quality of gluing

Results for the 2252M and KM adhesives are shown in Figure 5 and Figure 6, respectively. In the figures, 'Gdry' refers to specimens that were maintained in an atmosphere of 20°C and 65% RH ("dry" conditions) for one week after gluing. 'Gwet' refers to specimens that were submerged in water for 4 days at 20°C and they were tested in wet conditions as indicated by the reference standard. Conversely, 'Gcond' refers to specimens that, after their immersion in water for 4 days for the D3-class or 3 hours for the D2-class, were re-equilibrated at normal conditions (20°C/65% RH) for one week.

Values of shear strength of 2252M for tests of untreated walnut in dry conditions are very high and are above the minimal limits reported in EN 204 (10 MPa for Gdry specimens and 8 MPa for Gcond), whereas values of Gwet are barely under the limit of 2 MPa. However, the dispersion of data is quite high (COV of 25 to 28%), independently of the values of the cohesive wood failure (CWF) that for Gdry is very limited (30% only) while for Gcond is 80%.

As expected, the thermal treatment of walnut reduced the gluing quality: both Gdry and Gcond were appreciably lower (from 15 to 11 MPa and from 15 to 9 MPa, respectively), but both of these values are higher than minimal values considered in EN 204. On the other hand, Gwet was almost unchanged. The main differences with the untreated wood were the lower COV values (5 to 15%) and the higher CWF values for Gdry (70%, more than double), both attributable to the lower tensile strength of treated walnut and also confirmed by bending tests.

The surface pre-treatment made by acetone did not seem to improve the quality of the adhesion, considering that Gcond was unchanged and Gdry was even lower (but this can be attributed to the differences in the substrate). Nevertheless, the treatment by acetone greatly increased the uniformity of the results (many fewer specimens were discarded because of adhesion problems) and the Gwet values reached the minimum allowed by EN 204.

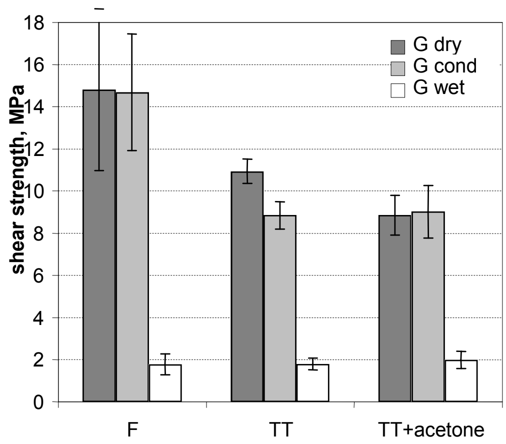

Figure 5 Values of shear strength for 2252M (D3-class). F = untreated wood, TT = Thermally Treated wood

The effect of acetone on gluing with adhesive KM is more appreciable than for 2252M (Figure 6). In fact both the values of Gdry and Gcond increased after the application of the solvent. The low value of shear strength observed for Gdry was entirely attributable to bad quality of the wood pieces. In that case 100% CWF was observed in approximately half of the specimens, whereas the adherends broke in the others. In such occurrence the shear value on the gluing area at failure was considered, even if we were aware that this is an underestimation.

However, acetone has a double effect: Gcond value slightly increased and the scatter of data decreased (COV was reduced from 25 to 30% to 8 to 15%). This positive outcome (differently from 2252M) was probably due to the different viscosity of the two adhesives (15,000 mPa·s for 2252M and 43,000 mPa·s for KM), and therefore to the fact that acetone is more able to enhance the penetration for the more viscous product.

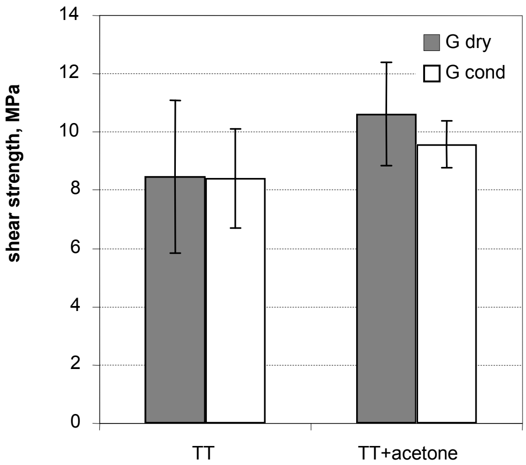

Figure 6 ***Values of shear strength for KM (D2-class). TT = Thermally Treated wood***

Dimensional stability (and varnishing)

The maximum change in the two transversal dimensions between 30% to 80% RH are shown in Table 4.

It is possible to observe, especially for walnut, that the treatment stabilized the wood: the maximum percentage change was higher in the untreated samples. Moreover, the varnishing generally had the effect of decreasing the wood dimensional variations. However, considering the short time samples were in the conditioning room, the varnish could only have had the effect of increasing the time required for the wood to reach the final EMC.

These variations were not so clear for cherry because of the different orientation (radial or tangential) of the board. Moreover EN 1910 prescribes a casual sampling and this leads to a high source of variability in the results.

Table 4 ***Mean values of maximum change (%) in width and thickness (EN 1910) between RH 30% to 80%. Minimum and maximum values in brackets***

	VARNISHED	WIDTH	THICKNESS
Untreated Cherry	No	*1.10* (0.87-1.42)	*1.87* (1.41-2.24)
	Yes	*1.18* (0.92-1.36)	*1.53* (1.08-2.29)
Treated Cherry	No	*0.53* (0.47-0.56)	*0.75* (0.46-1.07)
	Yes	*0.68* (0.57-0.82)	*0.43* (0.32-0.64)
Untreated Walnut	No	*1.52* (1.36-1.68)	*1.89* (1.41-2.30)
	Yes	*1.41* (1.07-1.92)	*1.43* (1.2-1.71)
Treated Walnut	No	*0.65* (0.55-0.73)	*0.74* (0.59-0.89)
	Yes	*0.50* (0.49-0.52)	*0.48* (0.44-0.57)

Conclusions

Actually, wood modification by HTTT is known to be an interesting (and sustainable) solution to improve dimensional stability, natural durability and wood colour uniformity (with a limited decrease of the mechanical properties) and a means to promote more valuable uses for low quality hardwoods with new potential applications.

Cherry and walnut sawnwood treated at the temperature interval 70 to 210°C displayed notable changes in their properties.

Chemical modifications in the wood occurring at high temperature were accompanied by several changes in the physical structure: mass loss, reduced shrinkage and swelling, low equilibrium moisture content, dark and homogeneous colour of wood. The reduction of the hygroscopicity were confirmed by the value of 4% of EMC reached by the heat treated wood after 4 weeks at 20°C and 65% RH. It was also confirmed by the wood EMC reached in dry and wet air conditions. Results also revealed an oven dry density reduction of 3.9% and 4.2% for cherry and walnut, respectively. Moreover, bending strength decreased and a modulus of elasticity increment in treated wood was recorded. Finally, an interesting increase in surface hardness was found. All these results can be explained by cellulose and hemicelluloses degradation due to the high temperature process.

As for adhesion ability, the treatment for walnut reduced the quality of gluing with PVAc-dispersed products belonging to D3 and D2 classes of durability of EN204, because of a well known inactivation mechanism of the surface induced by high temperature. Nevertheless, the D3 adhesive tested exhibited good values in the dry and conditioned specimens, whereas the measured values were low in the wet ones; this allows us to say that other gluing conditions could bring the adhesive to be still classified in the same durability class even if applied to treated wood. The application of acetone appreciably increased the performance of the D2-class adhesive whereas it left almost unchanged that of the D3-adhesive. This difference was attributable to the fact that acetone is able to increase the penetration of the former product, more viscous, while it had a very limited effect on the other one, whose viscosity is already sufficiently adequate.

The durability of cherry and walnut against insects, on the basis of the preliminary evaluation, was not modified by the thermal treatment or in any case this process is not a preventive preservative meant to control the development of new attacks by *Lyctus brunneus* and *Trichoferus holosericeus.*

The preliminary tests on dimensional stability showed that the thermal treatment stabilized the wood, and that the varnish layer could contribute in the same way.

In conclusion, HTTT is a way to drastically change the properties of wood and to obtain a new material. Thanks to the treatment, it seems possible to reach more interesting and valuable uses of the examined timber material, with potential applications for the production of solid wood panels, flooring, furniture and cabinets.

New studies are in progress on the application of HTTT modified methods in order to overcome some limitations in the use of this material and to optimize the process.

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