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RESEARCH PAPERS

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INFLUENCE OF THERMAL TREATMENT ON SELECTED PROPERTIES OF CHESTNUT WOOD AND FULL RANGE OF ITS VISUAL FEATURES

The use of chestnut wood (Castanea sativa Mill.*) is highly relevant in Central Italy, as it is one of the most important and abundant broad-leaf species in this geographical area. The comprehension of the modifications induced by thermal treatment is of crucial importance to define the optimal temperature that could improve the mechanical and physical properties without affecting significantly the visual appearance. In this paper a careful and complete investigation on the effect of thermal treatment on chestnut wood (Castanea sativa* Mill.*) is reported. The aim of this study is addressed to understand the chemical-physical modifications occurred on the surface of wood samples, as a consequence of heating, in order to choose the most suitable temperature of treatment, also in the view of applying a possible coating. No such complete and homogeneous study on chestnut wood was found in the literature, so this paper contributes to add relevant scientific and technological information on it. Samples of chestnut were thermally treated 6 hours in a conventional oven at 140°C, 170°C and 200°C. Surface properties of heated wood, in comparison with untreated, were evaluated through the measurements of: roughness, colour, Vickers and Brinell hardness, surface profile and contact angle. The behaviour of earlywood and latewood was evaluated by studying separately the effect of heating on contact angle and surface micro--hardness. Fourier transform infrared spectroscopy was also used to evaluate the chemical modification of wood components due to thermal treatment. Heating at 140°C has little influence on wood characteristics whereas 200°C has a great impact on colour, mechanical properties and hydrophobicity behaviour. The*

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intermediate temperature, i.e. 170°C, seems to give the best results in term of improved mechanical properties and also aesthetical appearance of wood surfaces.

Keywords: *Castanea sativa* Mill., heat treatment, colour, surface properties, reflectance spectrophotometry, Fourier Transform Infrared spectroscopy

Introduction

Thermal treatment is widely used in wood industry because it should increase the stability of the material, as recently discussed in review papers [Esteves and Pereira 2009; Candelier et al. 2016]. This process causes a reduction of water adsorption due to the decrease of available free hydroxyl groups of carbohydrates [Budakçı et al. 2011]. The thermal treatment, by reducing the wood hygroscopicity, contributes to the dimensional stability of the treated material, as swelling and shrinkage are mainly related to water absorption and desorption phenomena [Enjily and Jones 2006; Korkut and Kocaefe 2009].

It is well-known that wood is a widely used material, from the domestic decorative market to industrial applications. Wood has been largely used in buildings indoor and outdoor, in flooring and in furniture. In addition to the many species of tree that provide wood, there is substantial use of various composite forms such as plywood and fiberboard [Bulian and Graystone 2009]. Generally wood surfaces require protection to improve resistance to moisture and solar radiation [Capobianco et al. 2017]. In fact, wood materials, even if widely used, can suffer degradation phenomena mainly due to moisture and UV component of solar radiation [Hon and Shiraishi 2001; Chang et al. 2002; George et al. 2005; Pandey 2005a; Oltean et al. 2008; Sharratt et al. 2009; Genco et al. 2011; Lo Monaco et al. 2011; Agresti et al. 2013a; Pelosi et al. 2013; Teacă et al. 2013; Živković et al. 2014; Calienno et al. 2014, 2015]. For this reason, different kinds of protective and stabilizing systems are used ranging from thermal treatment to surface protection by means of different kinds of commercial products such as solvent-borne, water-borne, high solids, powder coatings and radcure products [Bulian and Graystone 2009; Pelosi et al. 2013; Bonifazi et al. 2017a].

In this work, a careful investigation on thermal treatment was provided in order to evaluate the effect of the process on the surface properties of chestnut wood (*Castanea sativa* Mill.) [Akyildiz and Ateş 2008; Ateş et al. 2010] as relevant issues in the perspective of applying protective coating. The choice of this wood species was due to its wide representation in Central Italy [Calienno et al. 2015; Venanzi et al. 2016], and wide use for the construction of doors and windows due to the properties of its heartwood [Agresti et al. 2010]. In fact, chestnut heartwood contains extractives that give it a pleasant colour and a natural durability with regard to biotic agents [Annesi et al. 2015].

The effect of thermal treatment was evaluated by different tests; in particular, colour measurements, Fourier transform infrared spectroscopy, hardness measurements, roughness, surface profile and contact angle measurements were performed in order to understand the surface changes in wood untreated and thermally modified. A new approach was also applied for evaluating the different behaviour of earlywood and latewood in regard to thermal treatment, by measuring separately the contact angle and the micro-hardness.

Complete and homogenous studies on chestnut wood features cannot be found in the international scientific literature, so the aim of this study was to investigate the properties of chestnut wood after thermal treatment and in the perspective of applying a new water-based preservative coating.

Materials and methods

Sample preparation and thermal treatment

Twenty four chestnut flat samples (10 cm length, 5 cm width an 2 cm thickness) were obtained from a board of chestnut, cut in the heartwood. After cutting, the samples were stored in darkness in a conditioned room at 65% relative humidity and a temperature of 22°C to reach 12% moisture content. The radial surface was used for the measurements. Density of samples had an average value of 0.720 ±0.017 g/cm^2.

To obtain regular surfaces and to satisfy the requirements for subsequent coating application, samples were sanded with 120 grid size sandpaper.

Before thermal treatment, samples were dried in a laboratory oven at 103 ±2°C for 24 h. Then, heat treatments were performed at three different temperatures for 6 h: 140°C, 170°C and 200°C in a laboratory oven controlled to within ±2ºC under atmospheric pressure. Four sample sets (each made of six pieces) were used: one remained untreated and the other three were heated at the three chosen temperatures. After heat treatment, samples were conditioned at 20ºC and 50% relative humidity (RH) to reach equilibrium moisture content. During this time, dimensions and weights of the samples were measured by using a precision caliper and an analytical balance respectively until they reached constant values. Tests and measurements were performed after mass stabilization. As selected tests and measurements, colour, surface roughness, curvature profile, contact angle, hardness and infrared absorptions were chosen to obtain visual, physical and chemical information, all relevant to understand the influence of thermal treatments on chestnut wood.

Colour monitoring

Colour was measured through X-Rite CA22 reflectance spectrophotometer according to the CIELAB colour system (ISO 11664-4:2008). The characteristics of the colour measuring instrument were the following: light

source D65; standard observer 10°; fixed geometry of measurement 45°/0°; spectral range 400-700 nm; spectral resolution 10 nm; aperture size 4 mm.

This parameter was measured before thermal treatment and after in order to evaluate the changes in chromatic coordinates induced by heating. For each sample, forty-five measure points were chosen, due to the high colour variation in wood surface, as previously discussed [Lo Monaco et al. 2011]. In each point three measurements were performed so that to have 135 values of L*, a* and b* coordinates for each sample. Then average values and standard deviations were calculated. Data are reported as L*, a* and b* values and total colour variation expressed by ΔE, which represents the geometric distance of two points in the L*a*b* colour space.

In order to evaluate the colour stability after thermal treatment, sample colour was measured during natural re-hydration of wood.

Roughness measurements

Roughness measurements were performed by a Taylor-Hobson TalySurf CLI 2000 apparatus, according to the standards [DIN 4768:1990; ISO 4287:1997].

A contact gauge surface measured the 3D morphology of the substrates before and after heat treatments. One hundred and one profiles were stored for each sample, with a resolution of 1 μm along the measurement direction and 2 μm along the perpendicular direction, over an area of 15 × 2 mm. The profiles were stored parallel and perpendicular to wood grain. TalyMap software Release 3.1 was used for data analysis and evaluation of the roughness parameters. This measure allows the main roughness parameters to be evaluated. In particular, average roughness R_a and ISO10 points height R_z are a measure of the amplitude parameters of the roughness profile. Spacing R_{Sm} is a measure of the characteristic wavelength of the roughness profile. The hybrid parameters slope $R_{\Delta q}$, R_{Sk} and R_{Ku} account for the average slope of the roughness profile and for its distribution and symmetry around the center line. 3D morphological maps were also stored, using a resolution of 3 μm along both the measurement and perpendicular direction, over an area of 8 × 8 mm.

Curvature profile measurements

The Hexagon Goddess Global Performance 05 series coordinate measuring machine (CMM) was used to measure the curvature profiles of the substrates before and after the heat treatments. The device is equipped with a continuous scanning head, Renishaw SP25M. Five profiles were stored for each sample, in the direction perpendicular to wood grain, with a resolution of 50 μm along the measurement and scan speed of 5 mm/s.

Contact angle measurement

Contact angle was measured in order to obtain the characteristics of wettability of the untreated and thermally modified chestnut surface. The method of direct observation was used: the values are obtained by measuring the angle, in the liquid phase, generated by the tangent to water drop profile and the wood solid surface. The measurement was performed by observing the drop through a FireWire camera with telecentric optics and 55 mm focus length. The measurements were taken for 120 s because the contact angle varies during the time after drop application. The software OneAttension elaborated directly the visual data supplying the values of contact angles every 0.72 s.

The measurements were performed separately on the earlywood and latewood areas.

Hardness tests

Brinell test

The hardness test allows to evaluate the material strength to the localized elasto-plastic deformation. The test that evaluates the penetration resistance according to the Brinell method has been applied according to the standard [UNI EN 1534:2011].

Totally a number of 52 indentations, on untreated and thermally treated samples were performed. Before testing, samples were conditioned to the laboratory environment (22°C and 50% of relative humidity). The device was a loading head with a hardened steel hemispherical body (diameter D = 10 mm). A force (F) was applied reaching the value of 1 kN in 15 ±3 s, maintained for 25 ±5 s. At least 3 minutes after, the residual indentation, the concave permanent deformation of the sample surface, was measured as the surface of the spherical cap, averaging values of two perpendicular diameters (d).

Brinell hardness (HB) was determined according to:

$$\mathrm{HB} = \frac{F}{(0.5\,\pi D)\cdot\left(D - \sqrt{(D^2 - d^2)}\right)}\ \mathrm{N/mm^2}$$

After checking for normality, one-way ANOVA test was performed to compare HB in heated treated test.

Vickers test

Depth-sensing micro-indentation (Micro-Combi, CSM Instruments, Peseaux, Switzerland) was used to perform instrumented micro-hardness measurements. Standard micro-hardness test (micro-Vickers indenter) was performed on substrate by applying load of 15 N, to evaluate the heat treatment influence on the hardness. The measurements were performed separately on the earlywood and latewood areas.

Fourier Transform Infrared (FT-IR) Spectroscopy

Infrared spectra were obtained using a Nicolet Avatar 360 Fourier transform spectrometer. To perform FT-IR analysis, sample powder (10 mg) was mixed with spectrophotometric grade potassium bromide (KBr, 340 mg) and pressed in the instrument holder. For each sample 128 scans were recorded in the 4000 to 400 cm^{-1} spectral range (2500-25000 nm) in diffuse reflection modality (DRIFT) with a resolution of 4 cm^{-1}. As background the spectrum of the KBr powder was used. Spectral data were collected and elaborated though OMNIC 8.0 (Thermo Fisher Scientific Inc.) software. FT-IR spectra were recorded and compared with untreated and thermal treated wood at the three defined temperatures.

Results and discussion

Colour changes of wood samples

Colour is one of the most important properties of wood when choice is made by end users because aesthetic aspect is often the prevailing criterion of decision [Lo Monaco et al. 2015]. Thermal treatment causes darkening of wood as a consequence of production of coloured degradation compounds from hemicelluloses and extractives [Bourgeois et al. 1989; Jarosombuti et al. 2010; Matsuo et al. 2010; Chen et al. 2012; Candelier et al. 2016; Gurleyen et al. 2018]. In the present study, the visual appearance of wood sample colour has changed (Fig. 1) after mass stabilization of samples. The monitoring of colour after thermal treatments, during mass stabilization, highlighted that no further colour changes occurred.

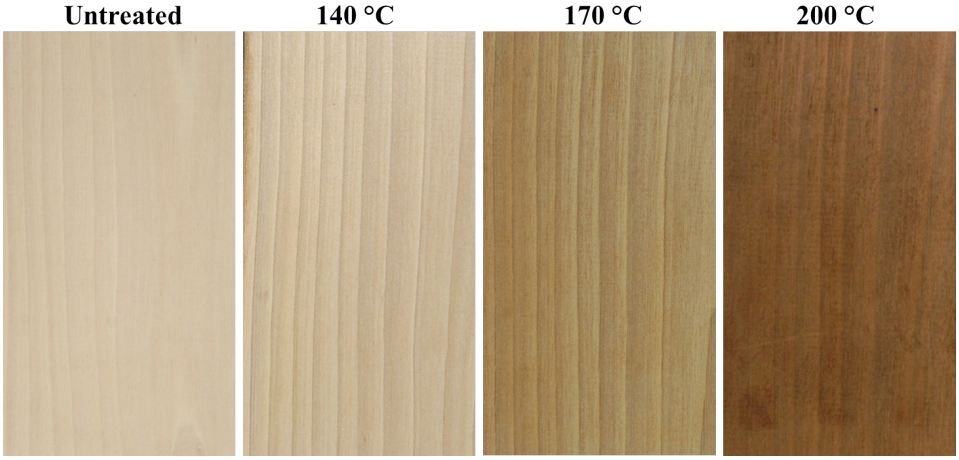

Fig. 1. Four wood samples untreated and thermally treated at 140°C, 170°C and 200°C

As it can be directly observed with naked eye, colour varies with thermal treatments and, as general result, wood darkens. This finding is confirmed by the chromatic coordinates' values (Fig. 2).

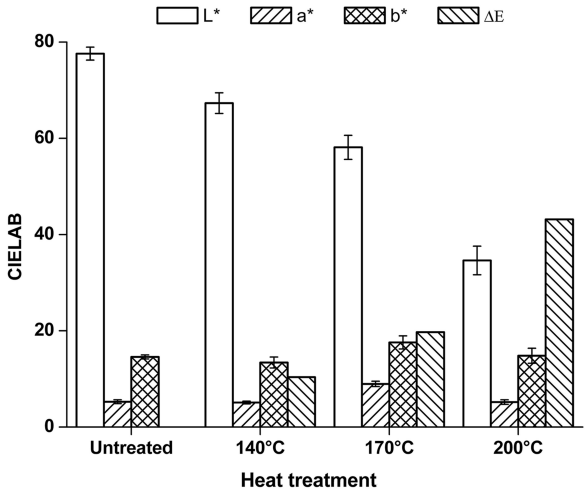

Fig. 2. L*a*b* values of untreated and thermally treated samples with the standard deviations of measurements

L* values clearly decrease with the increasing of thermal treatment ranging from 77 in case of untreated sample to 38 for the sample treated at 200°C as observed also by other authors [Correal-Mòdol et al. 2014; Gonzáles-Peña and Hale 2009a; 2009b; Tudorović et al. 2012; Marcon et al. 2017]. The chromatic coordinates a* and b* do not change significantly except for an increase clearly observed at 170°C and corresponding to an increase of both yellow and red components. As a consequence of these observations, it can be assessed that ΔE values, calculated for the three thermal treatments in respect to untreated samples, depend mainly on L* variations.

Roughness measurements and 3D mapping

The data reported for Ra indicate that average roughness increases with thermal treatments until 170°C, but it decreases at 200°C with values similar to those of untreated wood samples (Fig. 3).

On the other hand, R_a increases constantly with thermal treatment if measured in the perpendicular direction to wood grain (Fig. 4). This same trend can be observed for R_z values; in fact an increase of average roughness implies an increase of maximum profile height. The R_{Sm} parameter measured in the parallel direction (Fig. 3) exhibits maximum values at 170°C, whereas for the perpendicular one it has higher values at 200°C.

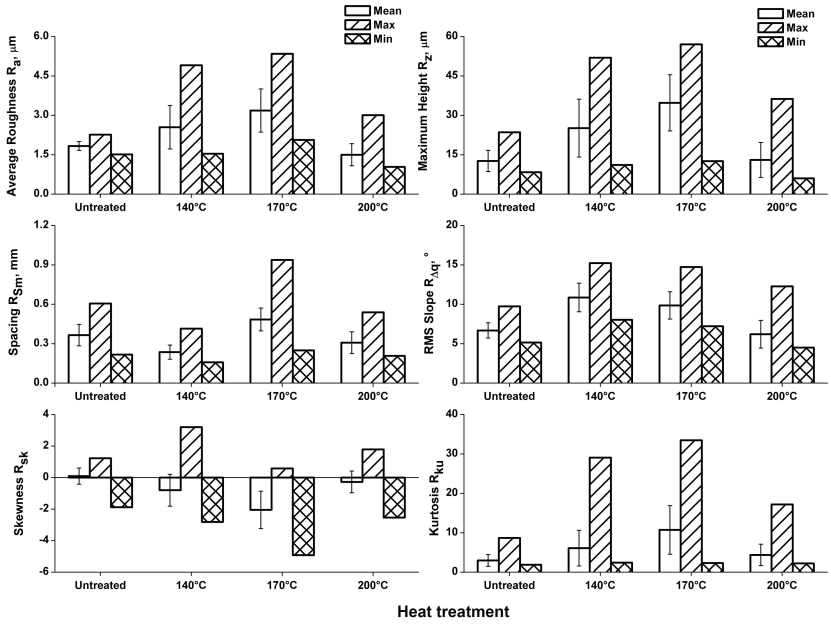

Fig. 3. Roughness parameters obtained for measurements parallel to wood grain at the different heat treatments

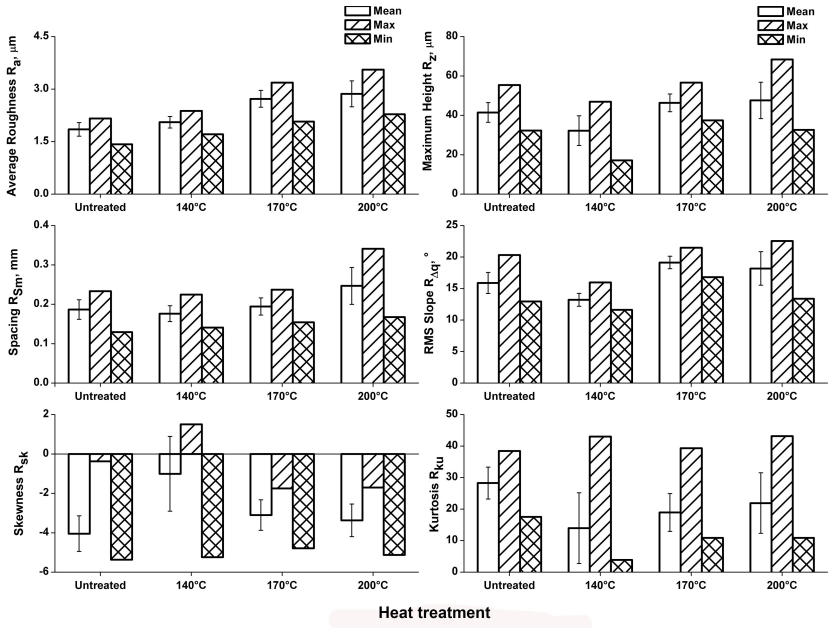

Fig. 4. L Roughness parameters obtained for measurements perpendicular to wood grain at the different heat treatments

The different behaviour between the two directions can be associated to the anisotropy of wood that changes its properties according to the anatomical directions. Moreover, heat treatment causes degradation of wood components, primarily hemicellulose and extractives that degrade thermally in the temperature range 180-200°C [Korkut et al. 2008a; Gonzáles-Peña et al. 2009; Laskowska and Sobczak 2018]. The degradation of hemicellulose is mainly due to the presence of acetyl groups that degrade under thermal treatment [Herrera et al. 2014; 2016]. Amorphous regions of cellulose are also rapidly changed structurally becoming less susceptible to reactions with water. Lignin suffers depolymerisation in a first and rapid phase then repolymerising in a more condensed material. Crystalline cellulose is largely unaffected below 300°C [Yildiz et al. 2006; Yildiz and Gümüskaya 2007].

The value of temperature 170°C is a sort of threshold over which hemicellulose degrades completely: this explains the increasing trend of roughness parameters until 170°C and the subsequent decrease at 200°C, for the measurements taken in the direction parallel to wood fibres.

On the other hand, the roughness parameters show an increase with temperature until 200°C in the case of measurements gathered in the perpendicular direction. This can be explained with the different degradation patterns of earlywood and latewood, as can be better observed on 3D maps of samples (Fig. 5).

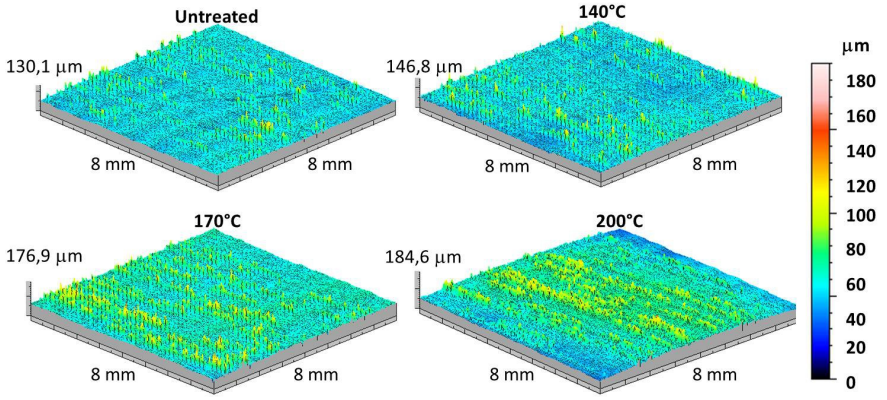

Fig. 5. 3D roughness maps of untreated and thermally treated sample surfaces, obtained for 8x8 mm areas

The 3D map of samples treated at 200°C clearly shows that surface underwent variations, specifically roughness increased along the earlywood zones (Fig. 5). So, if roughness parameters are measured parallel to wood grain, the result is a decrease at 200°C and an increase if taken in the perpendicular direction.

The degradation of earlywood in respect to latewood is probably due to the presence of higher content of hemicellulose in this zone of wood surface.

Contact angle and wettability of the surfaces

The contact angles were reported as function of time, until 120 s. Thermal treatment affected the wettability properties of wood surfaces by causing an increase of contact angle and consequently the hydrophobicity of the material (Fig. 6), which is also in accordance with the literature [Scheikl and Dunky 1998; Kocaefe et al. 2008; Kutnar et al. 2013; De Cademartori et al. 2015; Miklečić and Jirouš-Rajković 2016; Laskowska and Sobczak 2018]. In all cases the differences between earlywood and latewood are small. The hydrophobicity is particularly evident for samples treated at 170°C and 200°C whereas the heat treatment at 140°C seems to not change significantly the contact angles especially for latewood. Moreover, untreated samples exhibit a constant decrease of the contact angle as a function of the time, while the thermally treated wood samples presented low decrease rates, mainly in the 200°C treatment.

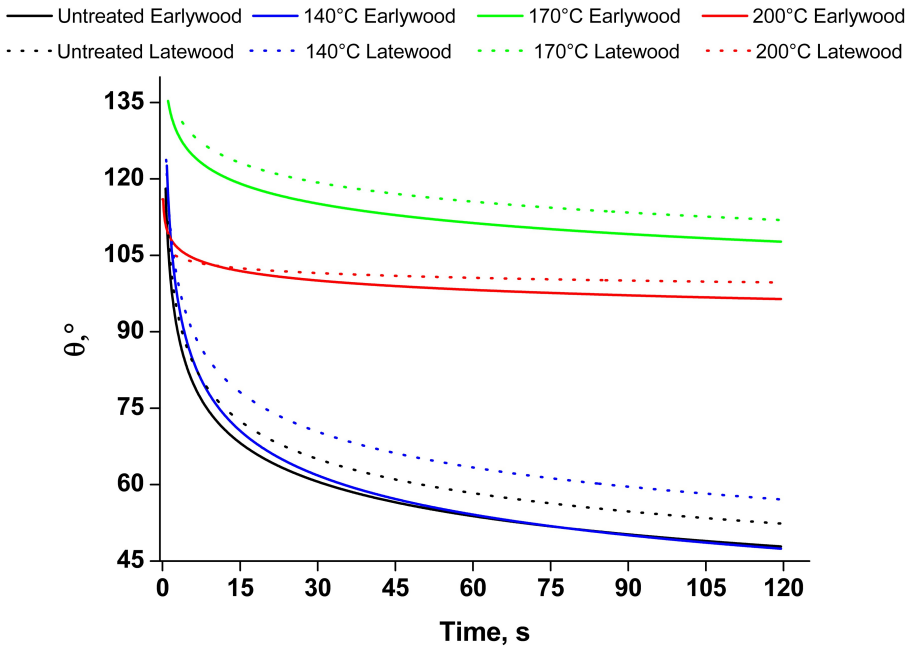

Fig. 6. Trends of the contact angles as function of time after water drop application

The increase of contact angles in thermally treated samples is due to hemicellulose degradation and amorphous cellulose re-organization causing a reduction of water adsorption due to the decrease available free hydroxyl groups of carbohydrates [Pétrissans et al. 2005; Oliveira et al. 2010]. Some

authors proposed also a different reason for justifying wettability change especially at lower temperatures [Hakkou et al. 2005]. In particular, they suggested the plasticization of lignin and consequently the reorganization of the lignocellulosic polymeric components of wood [Hakkou et al. 2005].

In the present paper the changes of contact angles at lower temperature, i.e. at 140° C, are observed for earlywood and can be associated to its higher contents of cellulose and hemicellulose polymers that could be involved in the above mentioned process of lignocellulosic polymeric reorganization.

At 200°C the contact angle showed lower values, both for late- and earlywood, in respect to those measured at 170°C. It must be considered that wettability could depend on macroscopic characteristics, such as porosity, surface roughness, moisture content and fibers orientation [Kocaefe et al. 2008]. Considering the roughness values, it was observed that they decreased for thermal treatment at 200°C in respect to 170°C in the direction parallel to wood fibres, whereas they increased in the perpendicular ones. This behaviour was explained with the higher degradation of earlywood at 200°C as clearly observed in the 3D map of the surfaces (Fig. 5). Moreover, the 200°C treatment probably generated small cracks in the samples surface [Boonstra et al. 2006; Priadi and Hiziroglu 2013], which could influence wood properties such as roughness and porosity and consequently contact angle values. So, lower contact angles at 200°C in respect to those measured at 170°C could be explained by the surface morphology modifications in addition to the hydrophobicity increase due to OH decrease.

Surface curvature profiles

There was an increase of curvature caused by heating, with no significant difference between 140°C and 170°C, in respect to untreated samples (Fig. 7). On the other hand, the curvature is much higher for the sample treated at 200°C, indicating evident warping of the surface. This result gives further evidence that the heat treatment at 200°C modifies the chemical and structural characteristics of wood causing also cupping of the sample boards.

Hardness test

Brinell hardness of chestnut slightly increased, about 5-6%, with temperature up to 170°C, decreasing with 200°C treatment (about 10%, Fig. 8). Thermo-treated samples showed no statistically significant differences compared to the untreated ones, although heat treatment clearly influenced wood colour.

As observed by other authors, heated wood properties depend on both species and processes [Esteves and Pereira 2009]. In fact, Poncsak et al. [2006] studied the hardness of birch thermo-treated in an inert gas ambient and they found a small increase with temperature, observing a maximum of the hardness

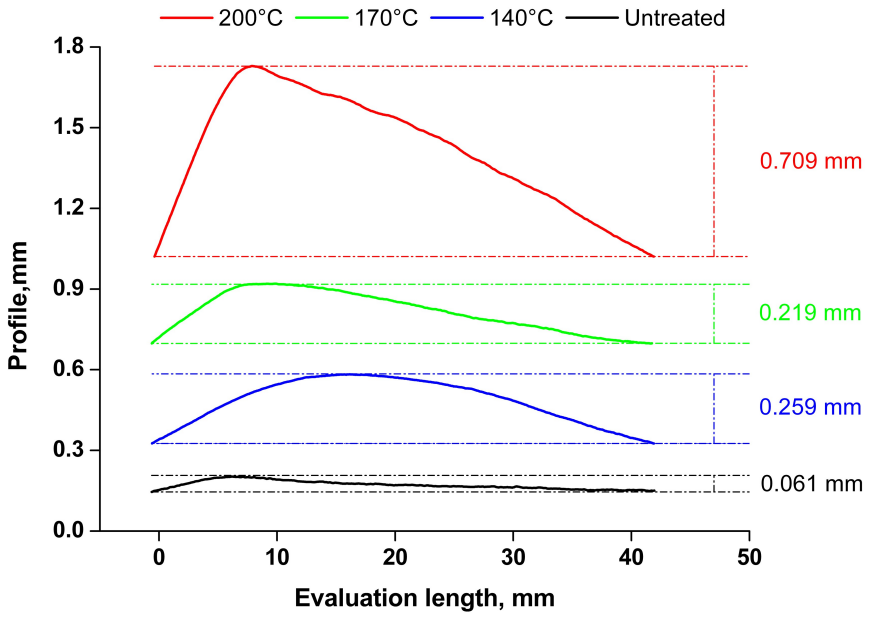

Fig. 7. Surface curvature profiles of untreated and thermally treated samples

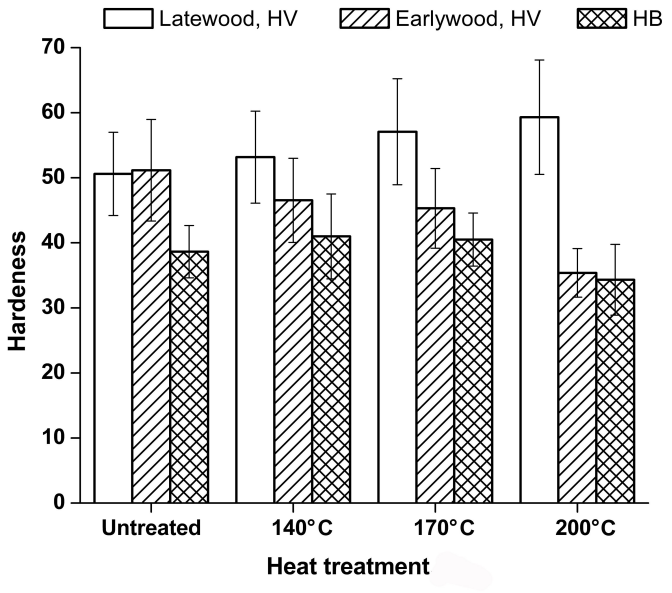

Fig. 8. Histogram of Vickers and Brinell hardness tests of untreated and thermally treated samples

at 160°C. Boonstra et al. [2007] detected a significant increase in hardness (48%) on *Pinus sylvestris* L. (Scots pine) during the heat treatment under modified atmosphere. On the other hand, Korkut et al. [2008b] ascertained that Janka-hardness decreased with heat-treatment. Contrasting behaviour on different species was noticed in broadleaf wood with ThermoWood process also by other authors [Shi et al. 2007]. Utilizing laboratory oven, as in our case, hardness was unfavourably influenced by heat treatment and time of exposition both in oak [Priadi and Hiziroglu 2013; Salca and Hiziroglu 2014] and chestnut [Ates et al. 2010].

In the case of micro-hardness obtained through Vickers testing, different results are observed for early and latewood. The first one exhibits a decrease of hardness with the increase of temperature of thermal treatment, whereas latewood has a completely opposite behaviour.

As previously assessed, earlywood is more degraded by heat treatment because it contains higher amounts of polysaccharides and this could produce hardness decrease. On the other hand, latewood, due to the presence of lignin, undergoes reorganization of the lignocellulosic polymeric components and this causes an increase of micro-hardness. Macro-hardness Brinell behaviour is a combination of micro-hardness in early and latewood.

FT-IR analysis

Band assignment (Table 1) was made according to literature references [Tolvaj and Faix 1995; Pandey 1999; Moore and Owen 2001; Colom et al. 2003; Nauman et al. 2007; Antonović et al. 2008; Bonifazi et al. 2015; 2017b].

The most significant change can be observed at 1735 cm^{-1} in correspondence of the carbonyl absorption that exhibits a decrease with thermal treatment, and at 1787 cm^{-1} (Fig. 9).

This last absorption appears well visible at 200°C. This behaviour can be associated to the cleavage of acetyl groups of polysaccharides and so to hemicellulose degradation [Hakkou et al. 2005; Tjeerdsma and Militz 2005; Miklečić et al. 2011], and also to extractives modification with the possible formation of coloured compounds [Helm et al. 1997; Pandey 2005b; Nzokou and Kamden 2006; Zahri et al. 2007; Chang et al. 2010].

Another modification is observed at 1667 cm^{-1}, corresponding to the conjugated carbonyl and H_2O (Table 1); the decrease of this absorption may be attributed to water elimination caused by thermal treatment, as observed also by other authors [Hakkou et al. 2005; Naumann et al. 2007]. This could be explained with the decrease of OH groups available for water molecules absorption. The absorption at 1327 cm^{-1}, which is associated to syringyl ring

Table 1. Position and assignment of the bands of DRIFT spectra

Band position (cm^{-1})	Assignment
3411	stretching of O-H group
2938	C-H and CH_2 asymmetric stretching
2904	C-H and CH_2 symmetric stretching
1735	stretching of the carbonyl group C=O
1667	conjugated carbonyl and H_2O
1592	aromatic skeletal vibrations
1503	aromatic skeletal vibrations
1457	C-H deformation and aromatic skeletal vibrations
1420	C-H in-plane deformation
1374	C-H in-plane deformation for polysaccharides
1327	syringyl ring breathing and C-O stretching
1233	guaiacyl ring breathing and C-O stretching
1156	C-O-C antisymmetric bridge stretching vibration in cellulose and hemicelluloses
1118	C-O-C symmetric stretching, aromatic C-H in-plane deformation, glucose ring vibration
1044	C-O and O-H association bands in cellulose and hemicelluloses
899	C_1-H deformation of cellulose
621	C-OH out-of-plane bending in cellulose

breathing in lignin and C-O stretching of cellulose (Table 1), gives the appearance of a doublet in the spectrum of the sample treated at 200°C (Fig. 9) suggesting the modification of cellulose that increase its crystalline character [Tolvaj and Faix 1995; Hakkou et al. 2005]. The decrease of the absorption at 1420 cm^{-1} (Fig. 9), due to C-H bending in cellulose (in-plane deformation of C-H, Table 1) is a further indication of modification of this polymer as a consequence of thermal treatment.

Conclusions

This study demonstrated that thermal treatments on chestnut wood causes modifications of chemical and physical properties of its surface and that different behaviour can be observed for earlywood and latewood due to the different composition especially in terms of hemicellulose polymer. The tested temperatures have clearly different effects on the measured parameters. Specifically, 140°C has in general little influence on wood surface causing little changes; 200°C treatment modify drastically wood properties also causing warping of the samples; 170°C seems to be a sort of threshold temperature.

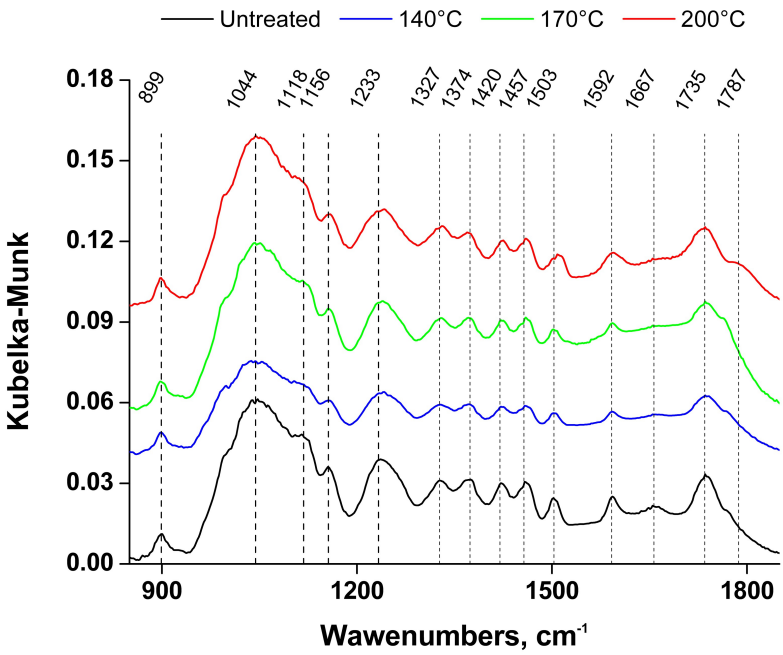

Fig. 9. FT-IR spectra of untreated and thermally treated wood samples. Focus on the 850-1850 cm^{-1} region

In fact, it provokes changes of wood characteristics but maintains visible the original wood fibres. Moreover, it increases roughness of the surfaces so allowing a better adhesion of a possible coating, and causes only little warping. Further, the 170°C temperature increases significantly the wettability so favoring the application of water based protectives. At last, the intermediate temperature increases surface hardness, and doesn't cause significant chemical changes.

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List of standards

DIN 4768:1990 Determination of values of surface roughness parameters R_a, R_z, R_{max} using electrical contact (stylus) instruments. Concepts and measuring conditions
ISO 11664-4:2008 Colorimetry - Part 4: CIE 1976 L*a*b* Colour space
ISO 4287:1997 Geometrical product specifications – Surface texture profile method – Terms, definitions and surface texture parameters
UNI EN 1534:2011 Wood flooring – Determination of resistance to indentation – Test method

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