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Steaming effects on selected wood properties of Turkey oak by spectral analysis

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Abstract Turkey oak (*Quercus cerris* L.) is characterized by some technological and aesthetical factors limiting its market value from its great potential. In this study, the effect of direct and indirect steaming on reduction in equilibrium moisture content (EMC) and colour variations was evaluated using a hyperspectral radiometer. Steaming treatments were carried out at 80°C for 48 h, and 120°C for 18 and 24 h, showing a reduction in EMC in the order of 8.1, 28.5 and 13.5, respectively, as well as very significant lightness (L^*) and hue (h°) modifications in comparison with untreated specimens. The spectral signature analysis confirmed that hydrothermal treatments modify wood sensibility to the light source in the entire spectrum range. The study supports the validity of hydrothermal treatments for improving technological and aesthetical properties of Turkey oak.

Introduction

Wood is widely recognized as the most frequently used material in constructions, thanks to its technological and aesthetical properties. However, its biological nature makes it susceptible to weathering degradation leading to alteration of some properties (Hon and Ifju 1978; Chang et al. 1982; Hon and Feist 1986).

A human-driven application of some environmental agents can be used to contrast wood's biological susceptibility, but also to improve some physical and technological properties. In the last years, heat and water treatments, performed first

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to reduce equilibrium moisture content and degradation by weather agents, have been usefully employed to also improve the texture characteristics of wood (Mitsui 2004; Johansson and Morén 2006).

Equilibrium moisture content and surface colour are very important wood properties that greatly influence its industrial usage (Esteves et al. 2008). The former is important because it is directly related to the dimensional stability of wood (Viitaniemi et al. 1997; Bekhta and Niemz 2003; Wang and Cooper 2005) and the latter because it has been observed that the final costumer pays much attention to the aesthetic texture (Mitsui 2004).

Turkey oak (*Quercus cerris* L.) actually arouses indifference in rich wood markets and the research world. Less dimensional stability, elevated internal tensions, strong swelling and shrinkage, low durability are its principal limiting factors (Giordano 1981). For these reasons, its wood is mainly used for energy purposes (i.e. firewood). Also from the colour surface point of view, Turkey oak is less appreciated than other oak species because of much more evident chromatic differences inside its wood structure, with white sapwood and dark grey heartwood (Tolvaj and Molnar 2006), frequently accompanied by the presence of heartwood discoloration known as "black-heart" (Giordano 1981).

Despite these limiting factors, Turkey oak wood has a great potential in the Apennines range system area of Italy. Turkey oak is one of the forest species with the largest planted area in Italy, especially on Apennines mountain range system (National Forest Inventory 2005), so that introduction in more profitable markets could represent an important opportunity to spark mountain economy in the Apennine area. In the Basilicata region (Southern Italy), where the study area is localized, Turkey oak timber forests cover 21% (23,400 hectare) of the total regional timber forest territory and 33.3% of the Italian Turkey oak high forest.

The great potential is indissolubly linked with the need of pre-treatments, in which heat and water treatments play a crucial role.

A large number of authors have observed, in fact, that heat treatments lead to a reduction in equilibrium moisture content, improving dimensional stability (Tiemann 1920; Stamm et al. 1946; Metsä-Kortelainen et al. 2006; Esteves et al. 2007), and mechanical resistance, proportionately to mass loss, which is in turn directly related to the treatment intensity (Zaman et al. 2000; Alén et al. 2002; Esteves et al. 2007).

It has been demonstrated that heat and water treatments can also improve aesthetical properties of wood. Several authors have reported a general darkening of tissues after these treatments (Mitsui et al. 2003; Bekhta and Niemz 2003). A little shift of Hue value from yellowish to red was also found on different hardwood and softwood species (Tolvaj and Molnar 2006; Esteves et al. 2008; Varga and van der Zee 2008). In most cases, the observed darkening is appreciated considering that a pale colour is often underestimated by a high percentage of the wood market, where "tropical flavour" is preferred (Esteves et al. 2008).

All property variations are induced by a change in chemical composition of treated wood. Esteves et al. (2008) have found a strong correlation between loss of lightness and glucose ($R^2 = 0.96$), hemicelluloses ($R^2 = 0.92$) and lignin ($R^2 = 0.86$) content,

while extractives contribution seems to be lower ($R^2 = 0.62$), but only in appearance. In fact, an important role in colour changing is assigned to extractives by Varga and van der Zee (2008), who found that the colour of wood changes proportionally to its water-soluble extractives content. According to Sundqvist (2004), extractives are the first compounds to be degraded during the treatment.

Chromatic differences between heartwood and sapwood in the wood structure of many timber species are due to the presence of extractives (Varga and van der Zee 2008), whose heartwood is generally richer than sapwood (Pandey 2005a, b); moreover, the presence of these external compounds makes it possible to homogenize and change wood colour through light irradiation and heat and water treatments (Mitsui 2006).

Also, homogenization of wood colour is very important since the final costumer pays much attention to the general homogeneity of colour and the absence of spots (Tolvaj and Molnar 2006).

The relevance of the general aspect of wood surface requires expressing colour in quantitative terms that is objective analysis of wood surface, which is central for many wood-processing decisions (Brunner et al. 1990). Tristimulus colorimetry can produce reliable results, especially by expressing colour parameters within the CIE Lab system, which is the mostly used system to graphically represent colour of an object and to determine percentage differences of colour (Brunner et al. 1990). Although spectral analysis can give more information than a simple portable spectrophotometer, it is possible to fix a real digital print of the surface and discriminate all colour variations not only in the visible range of the electromagnetic spectrum, but also in the near-infrared (NIR). By means of a spectroradiometer at high resolution, with a 0.2 nm sensibility, all minimal differences between the samples can be recorded.

On the basis of these evidences, a reduction in Turkey oak's limiting factors through the three hydrothermal treatments tested can be expected. In particular, a reduction in the equilibrium moisture content and consequently an improvement in the dimensional stability can be achieved.

From the surface colour point of view, it is hypothesized that the tested hydrothermal treatments can be a natural instrument to darken and homogenize the texture of Turkey oak samples according to the one observed by Tolvaj and Molnar (2006) on Magyar Turkey oak. Moreover, it is arguable that all chromatic differences inside the wood structure can be reduced into the boundary of statistical similarity by any of the treatments tested. At last, in this study, it was also hypothesized that colorimetric and spectral analysis can discriminate all colour variations between treated and untreated wood samples.

The aim of this work is to evaluate the effects of steaming treatments by spectral analysis, as well as to demonstrate that the potential of Italian Turkey oak can be improved when exposed to these treatments. Such objective would allow for a diversification of the commercial use of Turkey oak wood towards more appealing markets.

Materials and methods

Study site and wood specimens

Study areas are located in Croce dello Scrivano at 1140 m a.s.l. (Pignola, Basilicata Region, Southern Italy), geographical coordinates: 40° 34′ N, 15° 49′ E. The high forest studied is characterized by almost pure Turkey oak stands.

Turkey oak wood samples were obtained from nine trees. Diameter at breast height (dbh) and total height were measured for each sampled tree; the average values of dbh and height were 47 cm and 26 m, respectively. From each of the nine trees cut, two basal logs of 2.30-m length were obtained. In order to evaluate the physical characteristics, wood discs (3 cm thick, sampled at 0 and 2 m height) were obtained from each log. From each tree, only one log has been treated, while the second one represented the reference specimen. All the logs were later sawn to 80 mm of thickness and dried in a discontinuous vacuum kiln.

Finally, for the colour analysis, 80 wood specimens were obtained from the boards, 20 for each treatment and of about $50 \times 50 \times 15$ mm^3 (longitudinal, tangential, radial). Specimens were sanded and equilibrated at 12% moisture content before measurements. Sapwood and heartwood were distinguished for each treatment. All samples were polished with an extra fine P400 sandpaper prior to the measurements.

Moisture content and wood density were preliminarily determined according to ISO 3130 (1985a) and ISO 3131 (1985b).

Steaming treatments

Steaming was carried out on only one log from each couple. Two types of treatments were performed. An indirect steam treatment was carried out in a cylindrical pressure chamber, purpose-made and of 1.5 m^3 in volume. The autoclave was realized with a basin of boiling water and equipped with air and wood temperature gauges and a pressure gauge. Seven logs were steamed under saturated conditions; a temperature of about 120°C and pressure of 2 atm were reached and total heating was extended to 18 (treatment named B) and 24 h (treatment named C), respectively.

Direct steaming was performed in a Maspell BS drying kiln of 4 m^3 in volume. It was a fan cell with a series of saturated steam sprinklers equipped, also in this case, with temperature gauges.

Two logs were steamed at 80°C and 90% relative humidity to atmospheric pressure. Heating time was 48 h (treatment named A).

Table 1 shows the parameters used for steaming treatments. Kiln dry-bulb and wet-bulb temperatures were controlled with thermocouples and a hygrometer connected to a computer at selected points in the cells. After treating the logs, they were cooled for several days in the same chambers.

Table 1 Steaming and drying treatments

Treatment	Max air temperature (°C)	Max pith temperature (°C)	Max air pressure (atm)	Heating time (h)	Cooling cycle (day)
Direct steaming	120	93	2	18	3
Direct steaming	120	100	2	24	6
Indirect steaming	80	76	1	48	12

	Air temperature (°C)	Centre board temperature (°C)	Vacuum value (mmHg)	Vacuum time (min)	Vacuum cycle (n)	Total process (day)
Drying	63	55	120	15	412	34

Drying process

Drying process was carried out on all boards at 63°C, 120 mm Hg of vacuum for 34 days. This vacuum drying process is affected by alternating vacuum phases with heating phases under atmospheric pressure. During the heating phase, boards were heated by fan circulated hot air until the centre of wood reached the desired temperature without significantly loosing or absorbing moisture, a vacuum was then pulled on them. This method has the advantage of creating a temperature gradient which is favourable to the mass flow. The distribution of the water is carried out from hot parts to cold parts; surface evaporation lowers surface temperature and thus provides the transfer of water from hot areas in the centre of the wood to the surface (Perré et al. 1995). The drying phase continued until the temperature difference between surface and centre of board is very small (±0.1°C). In this case, 15 min were used to decide on the time to terminate vacuum phase (Chen and Lamb 2003). Table 1 illustrates parameters of drying process. Small clear specimens were obtained from the boards for physical tests. The number of specimens taken from each log was nearly equal.

Spectral analysis

Spectral measurements were carried out with a Fieldspec Handheld Pro Spectroradiometer connected to a Plant Probe (ASD Inc., Boulder, Colorado—USA). The Fieldspec is equipped with high sensitivity 512-element photodiode array spectral sensors able to record the spectral reflectance of a sample's surface in the VIS/NIR wavelength range (325–1,075 nm). All the values are related to the reflected light of a white standard plate. The plant probe provides a halogen bulb light source, a spot size of 10 mm and a specular reflectance of less than 5%. The spectra obtained on each sample were averaged to provide a single spectrum for each treatment. In this study, no statistical preprocessing techniques (e.g. normalization) were used to transform data.

Colour measurements

Colour analysis was based on CIE $L^*a^*b^*$ and $L^*C^*h°$ values obtained by a Minolta CM-2002 Spectrophotometer (Minolta Corp, Osaka, Japan) designed in order to

scan different wavelengths of light reflected from a sample surface and to record the intensity of that reflected light relative to a white standard plate.

The Minolta CM-2002 spectrophotometer (Minolta Corp, Osaka, Japan) uses a pulsed Xenon arc light source, in a diffuse illumination system and an 8° viewing geometry. The measurement area is 8 mm in diameter. The instrument uses a double-beam feedback system with two photodiode array spectral sensors that measure the wavelength range between 400 and 700 nm (in 10-nm steps) and the $L^*a^*b^*$ (lightness, red/green, and yellow/blue chromaticity coordinate) values.

CIE $L^*a^*b^*$ system can be described as a uniform colour scale: distances between the points signed on the colour space correspond to the visual difference in colour. In the CIELAB system, the L^* axis represents the lightness. L^* varies from 100 (for white) to zero (for black), a^* and b^* are called chromaticity coordinates: $+a^*$ is assumed as red index, $-a^*$ as green index, $+b^*$ as yellow index, $-b^*$ for blue index. L^*, a^* and b^* values were used to calculate overall colour changes.

$L^*C^*h°$ values are obtained by conversion of $L^*a^*b^*$ values, using the following formulas:

$$h° = \tan^{-1}\left[(b^*)/(a^*)\right] \quad (1)$$

$$C^* = \sqrt{\left[(a^*)^2+(b^*)^2\right]} \quad (2)$$

$L^*C^*h°$ colour space supplies information on the three colour components: lightness (L^*), colour saturation (C^*) and hue angle (h°). Data were obtained using a standard illuminant (mean of solar radiation minus UV region) in the SCI mode (Specula Component Included).

CIELAB L^*, a^*, b^*, C^* and h° parameters were measured on each specimen and average value was calculated for each treatment. The final chromatic coordinates are the mean values of five measurements for each sample used.

Results and discussion

Table 2 shows the average of equilibrium moisture content (EMC) of 63 specimens, for each group, of steamed and not steamed wood. The MC at the end of the drying process (60°C and 80% RH) was lower in boards treated than in untreated ones and those treated at higher temperature. The EMC relative difference between untreated and treated samples was 8.1, 28.5 and 13.5% for 80°C and 120°C (18 and 24 h) treatment, respectively.

Esteves et al. (2007) reported that for *Eucalyptus globulus* Labill. steamed at 200°C for 6 h, a maximum reduction in EMC of 46% at 20°C—85% RH and of 61% at 20°C—35% RH was obtained. For 200–260°C heat-treated beech boards, Kamdem et al. (2002) have obtained a decrease in EMC from 10 to 5% (50%) at 23°C—66% RH and from 14.5 to 8% (45%) at 23°C—86% RH, respectively.

The large dimension of the logs and the low temperature used during the steaming process could be the reason for a lower reduction in the EMC recorded in this research. The reduction in EMC is related to the mass loss during the steaming treatments even if in this study no effect was observed on wood density. In fact, untreated wood shows a value of WD (0.834 ± 0.034 g/cm^3) not significantly

Table 2 Equilibrium moisture content and density of Turkey oak wood samples

Code	Steaming	Untreated EMC (%)	Treated EMC (%)	Treated WD (g/cm^3)
A	Indirect: 80°C—1 atm—48 h	13.15 (0.92)	12.17 (0.85)	0.866 (0.038)
B	Direct: 120°C—2 atm—18 h	13.48 (0.88)	10.49 (0.83)	0.871 (0.030)
C	Direct: 120°C—2 atm—24 h	13.58 (0.90)	11.96 (0.86)	0.847 (0.045)

Values in parentheses are the standard deviation

different compared to treated wood values (Table 2). Hitherto, there are no studies related to mass loss of Turkey oak and its thermal degradation, but for several woods, it has been demonstrated that wood treated at high temperatures has less hygroscopicity than natural wood. The change in properties is mainly caused by thermal degradation of hemicelluloses (Korkut et al. 2008). As a consequence, the reduced number of hydroxyl groups had an effect on some physical characteristics of wood (swelling, shrinking). In addition, the heat treatment affected the weight loss, and the amount depended upon temperature and time (Korkut et al. 2008).

The lack of significant differences in wood density is probably due to the temperature. The study was performed considering a max air temperature of 120°C, while for most oak essences, the mass loss occurs at temperatures higher than 130°C (Sundqvist 2004).

Nevertheless, Kollmann and Schneider (1963) reported for oak wood an effect of heat treatment already at 100°C, and sorption capacity decreased with increased treatment time and temperature.

This study confirms reduction in EMC for steamed Turkey oak, but the size of the sample used may not be suitable to define its behaviour. Further investigations are required to confirm physical and technological properties of heat-treated Turkey oak wood.

Colour measurements of the wood samples show a general darkening of the surface, with a little switch to red hue for all three treatments (Table 3). Statistical analysis confirms that hydrothermal treatments lead to significant colour modifications for all colour parameters, with a great difference between treated and untreated wood samples for both lightness (L^*) and hue angle (h°). This is partially in agreement with Sullivan (1967a, b), who suggests that colour variation focuses itself on lightness and saturation.

Darkening was more evident for the strongest treatment (C samples), while the fastest one (B samples) showed the lowest reduction. The results indicate that both temperature and time of treatment affected lighting reduction with equal weight, giving evidence that darkening of wood is in all cases related to the treatment intensity, as also demonstrated by Tolvaj and Molnar (2006). However, under this point of view, there are no significant differences between treatments with a lightness reduction in the order of 6–7%.

Similar behaviour could be observed for reduction in hue angle (h°), which translates itself in a red shift of the wood colour.

Table 3 CIE Lab and LCh colour parameters analysis for different treatments

Code	Treatment	L^*	a^*	b^*	C^*	h (°)
A	80°C—48 h—1 atm	56.24^{b} (2.05)	10.76^{a} (0.53)	17.76^{ab} (0.43)	20.77^{ab} (0.36)	58.79^{b} (1.63)
B	120°C—18 h—2 atm	56.70^{b} (3.08)	9.52^{b} (0.67)	16.99^{b} (1.38)	19.50^{b} (1.14)	60.62^{b} (3.14)
C	120°C—24 h—2 atm	55.77^{b} (2.30)	10.81^{a} (0.44)	19.31^{a} (1.29)	22.13^{a} (1.30)	60.71^{b} (1.09)
D	Untreated	60.17^{a} (2.64)	9.10^{b} (1.04)	19.31^{a} (1.69)	21.37^{a} (1.70)	64.73^{a} (2.76)

Values in parentheses are the standard deviation. The different letters indicate significantly different means at $P < 0.05$

Varga and van der Zee (2008) have observed that treatment time has a greater effect than temperature, especially on mechanical resistance. Values of treated samples are statistically different compared with untreated ones, but among them there are no significant differences. In absolute terms, hue has fluctuated around 6 and 9% compared with untreated wood.

Most of the variability inside the three treatments are focused on chromaticity coordinates (a^* and b^*) and colour saturation (C^*). Statistical analysis highlights significant variation of red index in relation to treatment time: in fact, only longer treatments have caused a relevant increase in red component, in the order of 18% in both cases, A and C treatments, while fasted treatment statistically does not separate itself. For the yellow index and for colour saturation, the treatment time produces opposite effects, with little variation, not significant for the strongest one, and more evident reduction for the longest and the fastest ones.

Comparison between sapwood and heartwood submitted to the same treatment points out that the different hydrothermal treatments cannot reduce colour differences inside the same plant. Sapwood remains considerably different in comparison with heartwood for all colour parameters (Table 4). It is also clearer than untreated heartwood, where only a little red shift can be noticed.

This evidence confirms that extractives, whose heartwood is richer than sapwood, play a great role in changing colour by hydrothermal treatments. According to Sundqvist and Morén (2002), they are the first compounds to be degraded during the process.

Reflectance curves obtained by the spectroradiometer fortify tristimulus values. Spectral analysis shows that hydrothermal treatments modify wood sensibility to a

Table 4 CIE Lab and LCh colour parameters analysis between sapwood and heartwood for the same treatment

Wood Type	Treatment	L^*	a^*	b^*	C^*	h (°)
Heartwood	80°C—48 h—1 atm	56.24^{a} (2.05)	10.76^{ns} (0.53)	17.76^{a} (0.43)	20.77^{a} (0.36)	58.79^{a} (1.63)
Sapwood	80°C—48 h—1 atm	62.79^{b} (3.29)	10.02^{ns} (1.09)	21.12^{b} (0.94)	23.38^{b} (1.24)	64.67^{b} (1.84)

Values in parentheses are the standard deviation. The different letters indicate significantly different means at $P < 0.05$

light source in the entire spectrum range, but the response to different treatments can be considered very similar (Fig. 1). The mean spectral reflectance curves of treated wood samples are fairly distinct in respect of the untreated wood curve, but they appear almost equal in shape among them, with only a progressive shifting with increasing of the wavelength. This indicates that lightness differences are accomplished by a change of hue to red depending on treatment temperature and time.

Looking at the spectral reflectance graph, it is possible to identify two distinct trends between treated and untreated wood samples across the visible boundary.

In the visible region of the electromagnetic spectrum, all treated curves are below the untreated one, indicating a general reduction in lightness. The differences among treated and untreated curves tend to fall with increasing the wavelength. The reflectance reduction is less evident in the yellow–red region, but very significant in the red, so that the final effect on the colour is a general darkening of wood surfaces with a little shift to the red. The shifting of hue values is more evident for the strongest and the longest treatments, rather than the fastest one, for which a very strong darkening of wood surface can be pointed out, accomplished however by any change of hue. The differences in lightness and hue state that pressure and temperature of treatment have the same influence on this parameter. On colour modifications, this influence is quite equal for both treatment parameters, confirming last findings by Varga and van der Zee (2008) on colour of black locust and oak surfaces.

These evidences are in accordance with previous findings by Tolvaj and Molnar (2006) on darkening of Turkey oak after hydrothermal treatments, measured by traditional instruments.

In the near infrared, over 700 nm, the curve trend changes drastically. The mean of untreated wood curve is still above the only faster treatment curve (B), whereas the spectral reflectance of the two last ones reaches values beyond those of untreated wood samples. In this region, the strongest treatment reflectance exceeds all the other curves, indicating deep modifications in wood chemical structure.

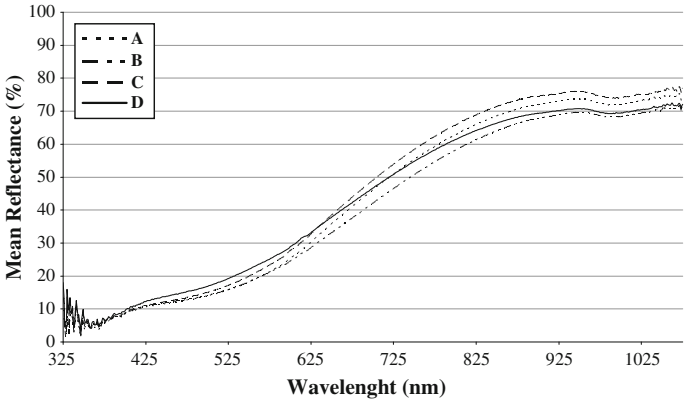

Fig. 1 Spectral reflectance of treated and untreated wood

Comparing sapwood and heartwood response to hydrothermal treatments, the spectroradiometer's data highlight a great difference in reflectance between the two kinds of wood, with sapwood curve generally over the other one in the visible spectrum, and the contrary in the NIR (Fig. 2). Also in this case, heartwood samples exceed the other one, indicating that most of the heat-degraded compounds are sensitive at the near infrared component of the solar light. These are represented by the so-called extractives, which are very rich in heartwood. Similar results in the NIR spectra have been found by Kelley et al. (2004).

This is in agreement with findings on UV light irradiation, which reinforce chromatic differences rather than reducing them (Mazet et al. 1993). Also Esteves et al. (2008) state that chromatic differences between early and late wood increase after treatment, and reinforce the assertion according to which hydrothermal treatments, object of this investigation, cannot be used in order to homogenize Turkey oak wood surface, in contrast to Tolvaj and Molnar (2006).

As reported by Sundqvist (2004), the major part of the change in colour is due to compounds emanating from hydrolysis of carbohydrates and extractives. Hydrolysis is an important reaction that occurs when wood is heat-treated and moisture is present in wood cells or under steaming conditions. The level of moisture and temperature during drying and steaming of hardwoods has been found to be the most important factor affecting darkening. Some extractives such as hydrolysable tannin in oak can be hydrolysed at temperatures above 30°C; obviously at higher temperatures the changes in wood compounds are stronger (Sundqvist 2004). The hydrolysis rate is also influenced by physical structure of wood, ring structure, pH, temperature, time and pressure during processing (Sundqvist 2004). Similar findings by Schmidt (1986) state that for the discolouration of oak, there is significant colour variation also at lower temperatures, but this situation occurs when wood is wet and when drying treatment was prolonged.

These hypotheses were also supported by Stamm (1956) who reported that thermal degradation of wood material was greater in a closed system and under steaming heating condition.

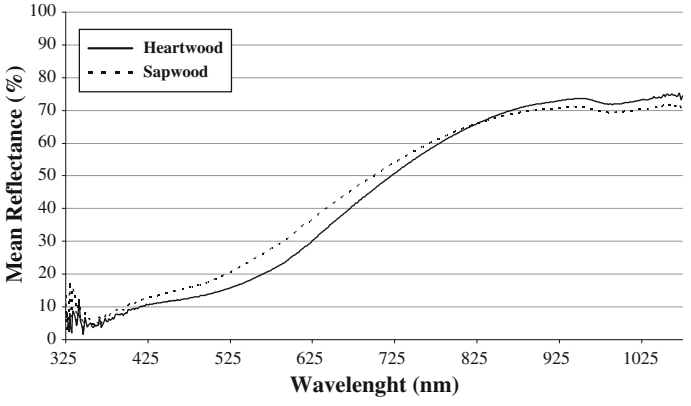

Fig. 2 Spectral reflectance of sapwood and heartwood samples

In this research, comparison of treatments showed that similar values of colour parameters can be obtained with different treatment conditions. In order to achieve the same effect on wood characteristics, such as colour, the hydro-thermal treatments can be performed over a wide temperature interval. Low temperature can be compensated by longer duration or pressure value. For a stronger reduction in EMC, higher temperature treatment seems to be required.

Conclusion

Quality of wood is a topic characterized by an increasing interest in the wood industry as much as in the research world, especially in the case of species which have more variability in technological properties like Turkey oak. The research carried out on Turkey oak wood samples in this study showed that moderate hydrothermal treatments, together with other benefits, can improve aesthetical properties of wood surface by a little darkening and red shift of the colour parameters.

Steaming treatments carried out at 80°C for 48 h, and 120°C for 18 and 24 h, show a reduction in EMC in the order of 8.1, 28.5 and 13.5%, respectively, and very significant lightness (L^*) and hue (h°) modifications in comparison with untreated specimens. The tested treatments confirmed the utility of hydrothermal treatment on woods affected by a strong swelling and shrinkage. The EMC of treated wood is reduced and as a consequence of this, the shrinkage and swelling of the material is also probably reduced.

Improved wood properties such as colour and dimensional stability depending on equivalent EMC is thought to be a possible approach for Turkey oak wood having no industrial usage.

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