

Effects of Heat Treatment Applied to Wood and Veneers of Various Wood Species

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ABSTRACT

Wood is one of the most important renewable materials with excellent physical and mechanical properties. However, its dimensional instability due the hygroscopic behavior is one of the shortcomings of wood and wood products. To enhance such disadvantage of wood many studies have been carried out using different modification processes and among them heat treatment is the oldest, the least expensive, and the most eco-friendly modification method. In this chapter the effects of heat-treatment on the properties of solid wood and veneers from different wood species are evaluated. It is expected that the heat treatment can add potential value on wood material to be used more effectively in further manufacturing steps.

Keywords: Colour changes; hardness; heat-treatment; roughness; solid wood; spectroscopy; veneer.

1. INTRODUCTION

Wood is one of the most important renewable materials with excellent physical and mechanical properties. However dimensional instability due its hygroscopic behavior is one of the shortcomings of wood and wood products. To enhance such disadvantage of wood many studies have been carried out using different modification processes [1-3]. Wood modification refers to a process that are meant to improve the properties of wood material producing a new material that will not damage more the environment at its life cycle end when compared to unmodified wood [3].

1.1 Considerations on Heat Treatment of Wood

Heat treatment is the oldest, the least expensive and most eco-friendly modification methods that has been popularly used along decades [4,5]. The interest in heat treatment processes has been renewed because the production of durable timber declines under a high demand for sustainable building

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materials, the tropical forests suffer from deforestation and the governments impose restrictions to reduce the use of toxic chemicals. There are in use worldwide five different commercial heat treatments (Table 1): one in Finland (Thermowood), one in Holland (Plato Wood), one in Germany (OHT-Oil Heat Treatment), and two in France (Bois Perdure and Retification). Two new heat treatment processes are also emerging in other countries, such as WTT in Denmark and Huber Holz in Austria [6,7].

Table 1. Commercial heat treatment processes in Europe

No.	Heat treatment process	Short description
1	Thermowood	Phase 1: by means of heat and steam, the kiln temperature is raised rapidly to a level of around 100°C; Phase 2: the temperature inside the kiln is increased to a level between 185°C and 230°C and maintained at that level for 2-3 hours; Phase 3: the final stage where temperature decreases to 80–90°C using water spray and remoisturizing and conditioning takes place to bring the wood moisture content to a useable content over 4% [2].
2	Plato Wood	Comprises a two-stage hydrothermal process performed in a stainless-steel reactor at relatively mild conditions with an intermediate drying stage in a conventional kiln. The process leaves high cellulose content in wood which is crucial to optimize final mechanical properties [7,8].
3	OHT-Oil Heat Treatment	The process involves heating of wood in vegetable oil (sunflower, rape seed oil, or linseed oil). In a closed process vessel, wood is immersed in hot oil and heated at temperatures between 180 and 220°C to ensure good durability at acceptable strength reduction [9].
4	Bois Perdure	The retification process is mild pyrolysis of wood under a nitrogen atmosphere, industrialised in France (Retiwood) The second French process is named Le Bois Perdure (the Perdure process). This process is relatively close to the retification process, and the properties of modified wood processed with both methods are similar. The wood is heated to 230°C in a steam atmosphere, the steam being generated from the water from the green wood [8].
5	Retification	

A typical heat treatment is applied at temperature levels and exposure times ranging from 120 to 250°C and from 15 min to 24 hours, respectively, depending on the process, species, sample size, moisture content and the desired target utilization. Physical and chemical properties of wood under heat treatment change at temperature near 150°C and it continues with increasing temperature [10,11].

1.2 Outline on Changes in Wood after Heat Treatment

Heat treatment is applied to wood to obtain enhanced properties, such as improved dimensional stability [12-14], increased durability, resistance to fungi

and natural weathering [15,16], smooth surfaces [17,18], and also a decorative specific dark colour [19,20]. But besides the advantages it has, heat treated wood presents reduced mechanical properties [21,22]. Heat treated wood is used for a large variety of indoor and outdoor applications, namely: parquet, wall and ceiling panels, saunas, kitchens, decorations and utensils, fences, joinery, claddings, windows, doors, while for structural applications it is not recommended. Table 2 summarizes the changes in wood after heat treatment.

Table 2. The changes in wood after heat treatment

No.	Property	Description
1	Mass loss	Heat treatment changes the chemical composition of wood, leading to mass loss that are quite high at higher temperatures.
2	Chemical transformation	Chemical and physical properties of wood start to change during heat exposure at temperature of about 150°C [6]. The decomposition of the main chemical compounds in wood occurs with high intensity at: 180°C for hemicelluloses, 270°C for lignin and 340°C for cellulose [23]. Organic polymers and extractives found in wood may deteriorate when exposed to elevated temperatures [24]. Hemicelluloses are the most affected compounds. Cellulose is more resistant to heat, which is attributable mainly to the crystalline fraction. Cellulose crystallinity increases due to degradation of amorphous cellulose. In lignin polycondensation reactions with other cell wall components, resulting in further crosslinking, contribute to an apparent increase in lignin content. Extractives are degraded or leave the wood at the same time that new extractable compounds emerge from wood degradation [6].
3	EMC	The reasons for the decrease of the equilibrium moisture are as follows: There is less water absorbed by the cell walls as a result of chemical change with a decrease of hydroxyl groups; there is enhanced inaccessibility of cellulose hydroxyl groups to water molecules due to the increase of cellulose crystallinity; and cross-linking occurs in lignin [6].
4	Anatomy	The effects depend on the wood species and on the process conditions used. Tangential and radial cracks, deformation on libriform fibres and collapse of vessels have been reported [6].
5	Dimensional stability	Dimensional stability increases due to cross-linking in lignin, due to the destruction of several hydroxyl groups, and due to decreased affinity with water in the case of treated wood. The reason for the improvement cannot be due to the cross linkages because treated wood shrinks in organic solvents such as pyridine or DMSO, as has been reported [6].

No.	Property	Description
6	Durability and weathering	<p>Wood durability is improved, increasing the resistance to rot, except in contact with soil, and slightly to weathering and insects, but it has little effect on termite resistance. Several reasons for the improvement of rot resistance have been reported: the transformation of hemicelluloses, which change from hydrophilic and easily digestible to hydrophobic molecules, and the fungal enzymatic systems do not recognize the substratum, the lower fiber saturation point which, by itself, leads to a better resistance against biological degradation, and there are changes in the external conditions affecting the microenvironment that affect the decay mechanism of heat treated wood. It is also mentioned that there might be an esterification of cellulose due to the acetic acid released by the degradation of hemicelluloses [6].</p> <p>Heat treated wood has better UV resistance than untreated wood, phenolic compounds are resistant to light aging. The heat treatment may not adequately protect the surface appearance and color stability in long-term outdoor conditions [25,26].</p>
7	Mechanical properties	<p>Mechanical properties are degraded. Brittleness of wood increases with the deterioration of fracture properties due to the loss of amorphous polysaccharides. The degradation of hemicelluloses has been identified as the major factor for the loss of mechanical strength, but also the crystallization of amorphous cellulose might play an important role. Polycondensation reactions of lignin, resulting in cross-linking, have been mentioned as having a positive impact mainly on longitudinal direction [6].</p>
8	Colour	<p>The wood colour changes due to heat are caused by thermal degradation of hemicelluloses and lignin. There are some differences between authors as regard to the temperature causing hydrolysis and oxidation of wood components. Such chemical changes may appear even at low temperature of about 65°C, depending on pH, moisture content, heating medium, exposure period and wood species [27]. Previous studies performed on solid wood have investigated the changes in wood colour that appeared during heat treatment and imply alteration of wood components [28-30]. The colour that wood gets after heat treatment was suggested to be an indicator of the degree of its modification and also a direct indication of chemical changes [31,32].</p>
9	Surface quality	<p>After heat treatment the surface roughness of the wood decreases. It was reported that the surface quality of wood species slightly improved with increasing both the temperature and time span of heat exposure [18].</p>

No.	Property	Description
10	Processing and finishing	The brittleness increases after heat treatment; it has a significant impact on its processing performance, local splits may appear [33]. The finishing properties decrease because the resin and other extracts come out and solidify on the wood surface. The bonding strength of wood and adhesive will be reduced [26,34]. The pH value of wood will change affecting the finishing performance of treated wood. An increase in acidity of wood surface can influence the wood wettability and the adhesion of water-borne coatings on treated wood [26].

This chapter presents the effects of heat-treatment on the properties of solid wood and veneers from different wood species native in Romania, USA and Japan.

2. EFFECTS OF HEAT-TREATMENT ON SOLID WOOD PROPERTIES OF VARIOUS WOOD SPECIES

There is a constantly growing market for thermally modified timber, which challenges the industry to introduce faster and reliable methods for the quality control of their products. Although most of the properties of black alder wood, red oak, Southern pine and yellow poplar have been studied there is little information about the influence of heat treatment on some properties such as surface roughness, hardness and wood micro-structure of these species. Therefore, the objective of this chapter section is to evaluate these properties as function of heat treatment applied to these four wood species. It is expected that the heat treatment can add potential value on wood material to be used more effectively in further manufacturing steps.

2.1 Preparation of the Wood Samples

Four wood species, namely black alder (*Alnus glutinosa* L.), red oak (*Quercus falcata* Michx.), Southern pine (*Pinus taeda* L.) and yellow poplar (*Liriodendron tulipifera*) were used in this study. Defect free black alder samples were cut from commercially manufactured flat sawn timber supplied by a sawmill company in Romania while the other three wood species were supplied by a local sawmill in Oklahoma. Samples made of black alder had the dimensions of 70 mm by 70 mm by 16 mm while the others were of 55 mm by 38 mm by 19 mm. A total of 25 defect free samples were cut from each wood species. All specimens were sanded with 220 grit size sandpaper by applying several light strokes before they were heat treated. The moisture content of all samples prior to heat treatment was 8%. Five groups each of five specimens were tested for the experiments. One group of non-treated samples was kept as control samples while the others were exposed to heat treatment of two temperatures, 120°C and 190°C for 3 and 6 hours in a laboratory oven.

2.2 Changes in Surface Roughness of the Wood Samples

The increase in surface roughness of wood is important for many applications. It is fact that surface roughness of wood can be affected by various factors such as annual ring width, differences between juvenile and mature wood, density, variation between early and late wood and specific cell structures. Surface roughness was measured before and after the heat treatment. According to ISO 4287 [35] two well accepted roughness parameters, namely average roughness (R_a) and mean peak to-valley-height (R_z) calculated from digital information from the surface of each sample were used to evaluate the surface quality of the samples [18,36]. A portable profilometer device of Hommel T-500 type equipped with a TK-300 skid diamond stylus with a 5-micron radius and 90° of tip angle was employed for roughness measurements [18,37]. Six measurements were taken at a constant speed of 1 mm/s over a 15 mm tracing length across the grain of the samples from four species (Fig. 1). The typical roughness profiles of the samples are illustrated in Fig. 2.

Among the four wood species tested in this work red oak presents the most porous anatomical structure having the roughest surface with an average R_a value of $9.4 \mu\text{m}$. Exposing oak samples to a temperature of 190°C for 6 h resulted in 7.46% improvement in their R_a values when compared to that obtained for 3 h span time exposure at the same temperature level. A difference of 10% for R_a values was also recorded for same species exposed to a temperature of 120°C for 3 h span time when compared to non-treated samples. Priadi and Hiziroglu found 4.4% improvement of R_a values in the case of oak exposed to a temperature of 130°C for 2 h while mahogany had 4.3% reduction with extending exposure from 2 h to 8 h [38]. This observation is in good agreement with our study results. However, all four types of species kept in the oven at 190° for 6 h presented smoother surface quality. According to other scientists, the average mean peak-to-valley height, R_z is more convenient to characterize the wood surface roughness [39]. When compared to non-treated samples, the heat treatment applied at 190°C for 3 h in the case of red oak samples produced 6.1% reduction of R_z roughness values, while for black alder samples the highest difference of about 2.6% was noticed after 3 h of heat treatment at the temperature level of 120°C [18].

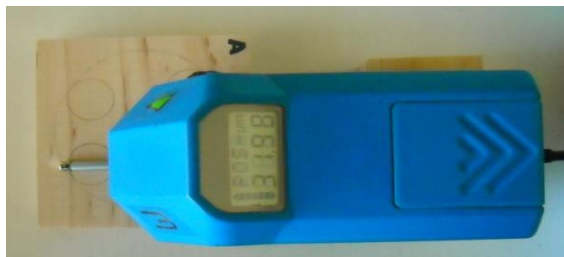


Fig. 1. Roughness profilometer Hommel T-500 [18]

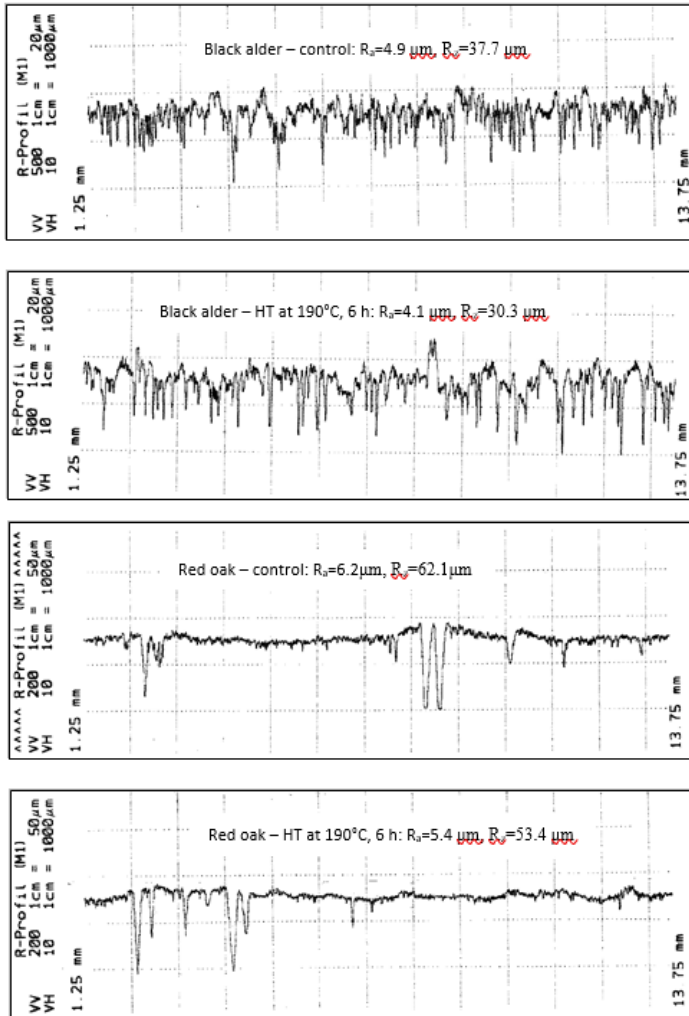


Fig. 2. Typical roughness profiles of the samples [18]

It was noticed that increased temperature from 120°C to 190°C for both exposure times showed significant differences from the surface quality of non-treated samples at 95% confidence level as shown in Figs. 3 and 4. It appears that surface quality of all species studied slightly improved with increasing both the temperature and time span of heat exposure [18].

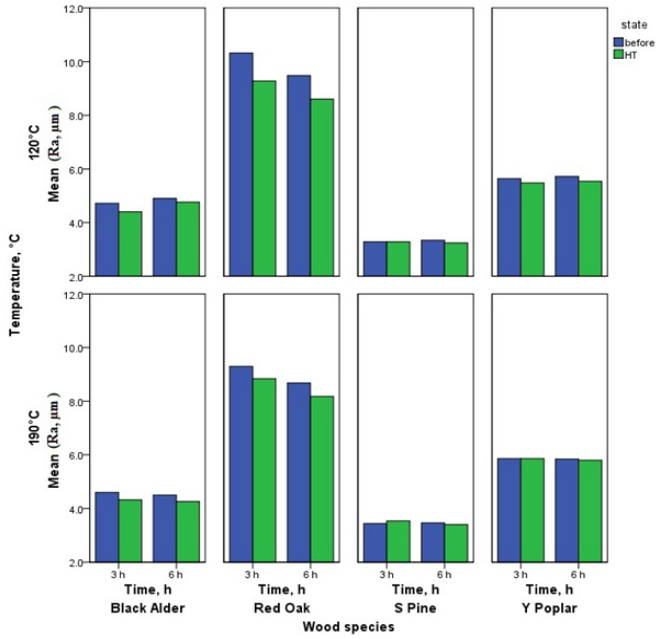


Fig. 3. Average roughness (R_a) values of species as function of heat treatment [18]

2.3 Changes in Hardness of the Wood Samples

Besides important advantages of heat-treated wood, an adverse influence of the treatment on mechanical properties of wood is still inevitable [40-43]. It was reported that all mechanical properties including modulus of elasticity (MOE), modulus of rupture (MOR), Janka hardness and compression strength parallel to grain of the specimens decreased with increasing temperature and exposure time [10]. Red bud maple presented 50% reduction in the hardness values when they are exposed at 180°C for 6 hours [10]. A Comten Universal Testing Machine was used to measure the hardness by embedding a steel sphere of 11.2 mm diameter into the sample perpendicular to the grain orientation (Fig. 5). Four measurements recorded in kg were taken from each sample of each wood species to evaluate the Janka hardness. The hardness was measured before and after the treatment.

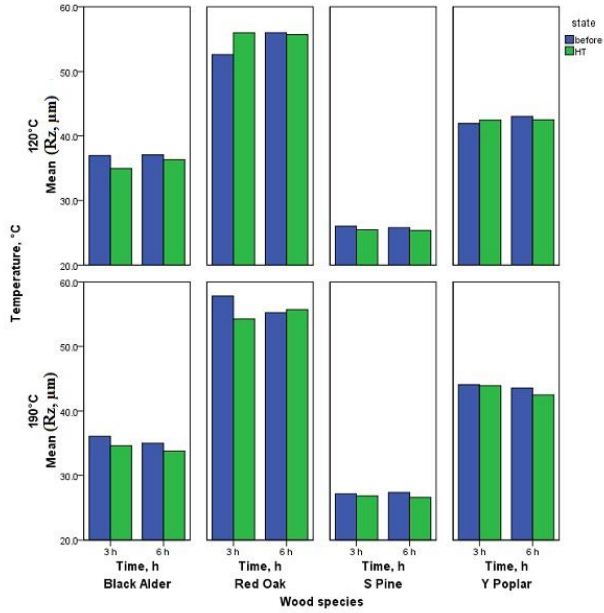


Fig. 4. Average mean peak-to-valley height (R_z) values of species as function of heat treatment [18]



Fig. 5. Comten 95 Universal Testing Machine [18]

The most significant reduction values in hardness due to heat treatment has been determined for red oak followed by black alder. When exposed to a temperature of 120°C for 3 h span time, samples of red oak recorded 18.7% lower average hardness value than before the heat treatment. But the average hardness value of same species exposed to a temperature of 190°C for 6 h was 41.7% lower than that recorded before the heat treatment. The magnitude of reduction in hardness of the specimens was more prominent with increasing exposure time to 6 h. In the case of black alder samples, a slow decreasing of hardness values was determined with increasing temperature and exposure time. The highest difference recorded for black alder in terms of hardness value when compared to non-treated samples was of about 4.3% at a temperature level of 120°C applied for 6 h span time. The temperature of 190°C reduced 7.9% hardness values of such samples with the increasing exposure time from 3 h to 6 h. Low significant differences were found between Southern pine and yellow poplar specimens before and after the heat treatment in terms of their hardness values, which could be related to their low density. Although red oak had the highest hardness values before the heat treatment, reduction of such samples was also higher than other wood species considered in this study [18]. Fig. 6 illustrates hardness values of the samples. Hardness results values found in this work were similar to those determined in a previous study [44]. Priadi and Hiziroglu found that oak samples had 42.7% reduction in their hardness values with exposure of 200°C for 8 h while in the case of pine no significant hardness reduction was found [38].

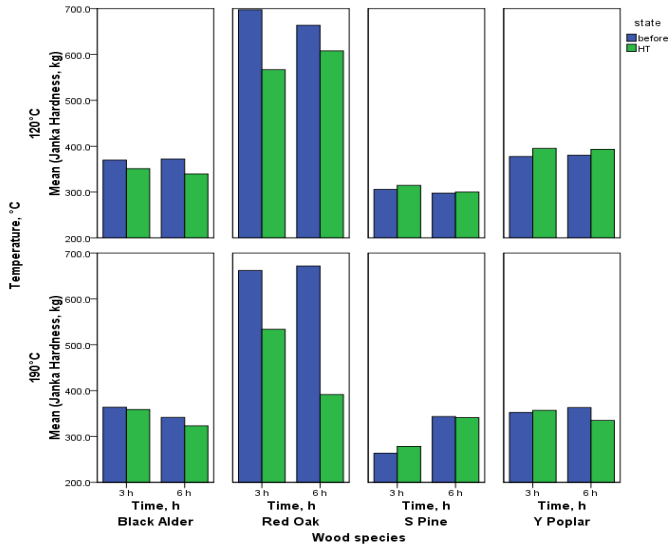


Fig. 6. Average hardness values of species as function of heat treatment [18]

2.4 Changes in Wood Micro-Structure of the Wood Samples

Micro-structural properties of heat-treated wood using scanning electron microscopy (SEM) were also studied in past works [45]. Boonstra pointed out that heat treatment influenced the anatomical structure of wood depending on the wood species and treatment schedule used [46]. It was found the cross section of heat-treated samples of Eastern red cedar presented rather smoother surfaces than that of control samples [36]. Huang et al. [45] determined that slight thinning of middle lamella of the cell wall took place on birch samples as result of heat treatment at temperature levels of 195°C and 215°C. Structural changes due to heat treatment were not distinct, but a certain level of plasticization of the cell wall occurred [38]. In this study the anatomical structure of samples was investigated by using SEM. The untreated and heat-treated samples with dimension of 3 mm x 3 mm x 3 mm were scraped with a blade. The samples were put under vacuum and coated with a thin film of gold using an ion sputtering device, before micrographs of the surfaces were taken. Fig. 7a and b show typical SEM micrographs taken from the cross sections of control samples and those exposed to a temperature of 190°C for 6 h of black alder and red oak samples. It appears that high temperature caused certain amount of damage on the cell walls of red oak and black alder as shown with arrows [18]. In the case of red oak control samples cell walls were complete and once they are exposed to heat treatment some distorted parts of red oak can be observed. Some damages of the cell walls were also noticed for heat treated black alder. The results are in accordance with that found in literature for oak samples and they are also supported by having hardness characteristics of heat-treated samples [18,38].

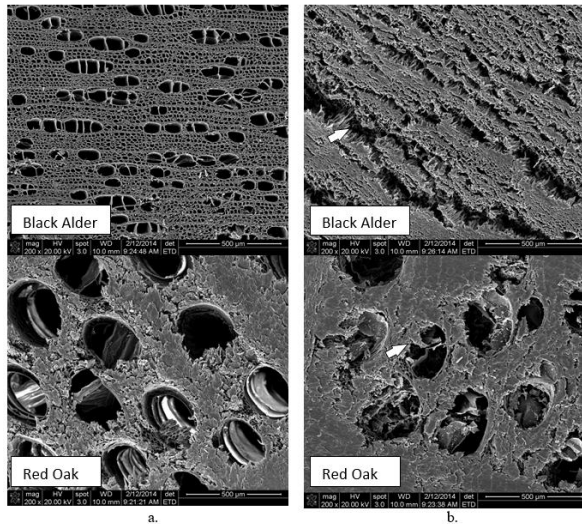


Fig. 7. Micrographs taken from cross-section of the samples by SEM (a-control samples, b- heat treated samples at 190°C, 6 h) [18]



Fig. 8. Discoloration of control and heat-treated samples [18]

2.5 Changes in Colour of the Wood Samples

It is fact that during the heat treatment wood becomes darker in color due to the considerable changes in its chemical composition, such as the degradation of hemicelluloses, lignin and certain extractive compounds. Wood discoloration is undesirable during drying but under heat treatment the darker color values are more evident. This simple and environmentally friendly modification method applied to wood does not cause any emission of hazardous volatile organic compounds [47]. Fig. 8 illustrates the progress of discoloration appeared as a result of heat treatment for all specimens. The higher the temperature of heat treatment, the darker the colour of specimens became.

The specialty literature provides relevant data regarding the discolorations of wood due to heat treatment. Japanese cedar wood was exposed to saturated vapor at temperatures up to 150°C, for 6 to 72 hours to determine the color

change and durability against brown rot fungus [48]. Red alder veneers were exposed at 30 to 90°C for 8 to 72 hours to establish the optimal schedule that can influence its color [49]. Johansson [50] predicted the mechanical strength using the color response of birch, spruce and pine wood species after heat treatment using ThermoWood. Discoloration of wild Pear wood was evaluated by Gunduz et al. [41] as a result of thermal treatment at temperature levels of 160, 180, 200°C for 3, 5 and 7 hours. Tuong and Li [19] also established the effect of heat treatment under nitrogen at temperatures over 210°C to 230°C for 2 to 6 hours on acacia hybrid wood color and its dimensional stability. The heat treatment applied to beech sapwood and hardwood at temperatures of 170, 190, 200°C for 4 hours has reduced the properties and equalized the color of the specimens [51]. Heat treated beech wood at 180°C and 200°C for 2, 3 and 4 hours generally undergoes a slight color change when exposed to visible light [30]. The heat treatment of black alder at temperatures around 120 up to 200°C for 2 to 10 hours delayed the rate of color change caused by weathering [52].

3. EFFECTS OF HEAT TREATMENT ON WOOD VENEERS OF VARIOUS WOOD SPECIES

Little information addressed the veneers modification of various wood species by heat treatment. Due to their small thickness, veneers need a shorter time of heat exposure at high temperature ranging between 180-200°C during their thermo-densification process [53,54]. Such process when applied to solid wood is a fairly long process [54]. It was found that the densification process applied to veneers for a shorter time affected their surfaces, particularly colour changes [54]. It appeared that the convection heat treatment caused stronger discoloration on veneer surfaces regardless the wood species when compared to press heat treatment [53]. The dark colour of veneers can easily mask some blemishes and other discolorations on the surface. Various processes, such as staining and varnishing, which involve mainly chemicals, may be skipped when using heat treatment as an eco-friendly alternative to obtain dark colour veneers for furniture manufacturing. Heat treated veneers may be successfully used as overlaying material for composites used in furniture manufacturing [55].

The objective of this chapter section is to evaluate the colour changes that occurred on veneers of black alder, beech and hinoki cypress when subjected to heat treatment by using the CIELab colorimetry technique and NIR spectroscopy. Colour measurement and vibrational spectroscopy are non-destructive, rapid and low-cost evaluation methods used to measure organic materials [56-60]. Black alder is a less valuable wood resource but a fast-growing species, while beech represents one of the most common wood species in Romanian wood industry.

Hinoki cypress is a representative tree species native to Central Japan. It is considered one of the most elegant types of wood in Japan used since ancient times for palaces, temples and shrines [61] (MAFF 2017). The best example is Horyuji temple in Nara, from the latter half of the seventh century, the oldest wooden structure in the world. When applied to such veneer species, heat treatment could therefore enhance their use for value added products in furniture

manufacturing as an alternative to expensive tropical species and to obtain different levels of accelerated aging for wooden samples [62].

3.1 Preparation of the Veneer Samples

Defect free veneer samples cut from commercially manufactured wood veneers of black alder (*Alnus glutinosa* L. Gaertn.), beech (*Fagus sylvatica* L.) and Hinoki cypress wood (*Chamaecyparis obtusa* Endl.) were used. Samples had the dimensions of 70 mm by 70mm by 0.5mm. A total of twenty samples were cut for each species. Prior to heat treatment all samples were conditioned for one week at 20°C and 60%RH. The moisture content of samples was 7.5%. Five groups, each of four specimens, were prepared. Out of them, one group was kept as reference while the others were subjected to heat treatment in a regular oven at 190°C with different time spans for 5, 10, 20 and 40 minutes, respectively [63].

3.2 Changes in Colour of the Veneer Samples

When wood is heated, its colour changes to yellow, brown, red or grey. Whatever the species, heat treated wood darkening depends on treatment schedule. Based on visual observations, the surface colour of heat-treated samples changed from lighter to darker with increasing the exposure time, but with different rates. In this study, a Chroma Meter Konika Minolta CR-400 device was used for colour measurements (Fig. 9). For each sample three color measurements were made in randomly selected zones, according to ISO 7724-2 standard [64]. CIELab System was used for colour evaluation [65]. L^* , a^* and b^* colour coordinates were determined both for control group and heat-treated veneers. Hereinafter, the colour differences (ΔL^* , Δa^* and Δb^*) and then the total colour change ΔE^* were calculated, according to the following relations:

$$\Delta L^* = L^*_E - L^*_i \quad (1)$$

$$\Delta a^* = a^*_E - a^*_i \quad (2)$$

$$\Delta b^* = b^*_E - b^*_i \quad (3)$$

$$\Delta E^* = \sqrt{\left((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2 \right)} \quad (4)$$

where: L^* is the degree of lightness; a^* – degree of redness and greenness; b^* – degree of yellowness and blueness, and the subscripts E and i indicate the values for the exposed samples and control references, respectively.



Fig. 9. Chroma Meter Konika Minolta CR 400 [63]

Surface colour changes under different heating durations for black alder and beech veneers are shown in Fig. 10. The surface colour changes of veneers due to heat treatment were investigated using the CIELab colour space. A decrease in lightness L^* with a similar trend for both species under study was noticed.

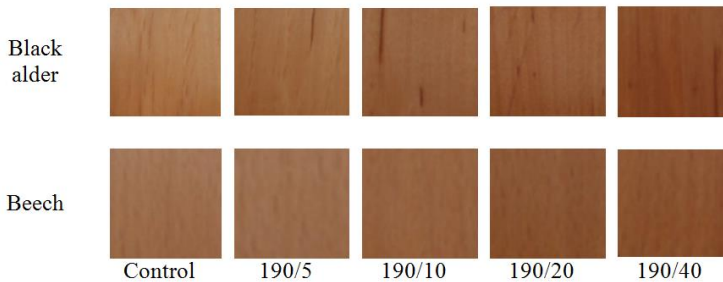


Fig. 10. Visual appearance of alder and beech veneers as function of heat treatment ($T^{\circ}\text{C}/\text{min}$) [63]

In particular, lightness L^* values monotonously decreased the first 10 minutes of heat exposure at 190°C . Afterwards the overall colour became darker as the heating time increased up to 40 minutes. The change in lightness was more pronounced for black alder than for beech veneers. Such darkening but at a slight rate was also reported for birch, alder and pine veneer specimens during their thermo-densification process for 4 minutes at temperature of 200°C and pressure ranging from 4 to 12 MPa, while almost no changes appeared for beech samples at the same parameters [54]. Oak veneers subjected to oven heat treatment at 200°C for 10, 20 and 30 minutes also showed a significant surface darkness when compared to press treatment [53]. Moreover, the feasibility of predicting some wood properties using lightness values, such as swelling, equilibrium moisture content, decay resistance and modulus of rupture (MOR),

was previously reported [16,51,57]. The process of discoloration is due to the changes which take place in extractives, lignin and hemicelluloses. The behavior is always accompanied by a reduction in lightness. Previous studies reported that decrease in lightness was caused by a decrease in hemicellulose contents, especially pentosan [27-29]. Extractives of each wood species make a specific colour which is expressed by two chromatic parameters, namely a^* and b^* . It is fact that the red component of wood is highly associated with the extractive content in wood [66,67]. Fig. 11 shows that the colour coordinates, a^* and b^* , respected almost the same pattern trend with the decrease in lightness for both species. The redness coordinate a^* increased gradually for each time span during the heat treatment for both species here investigated.

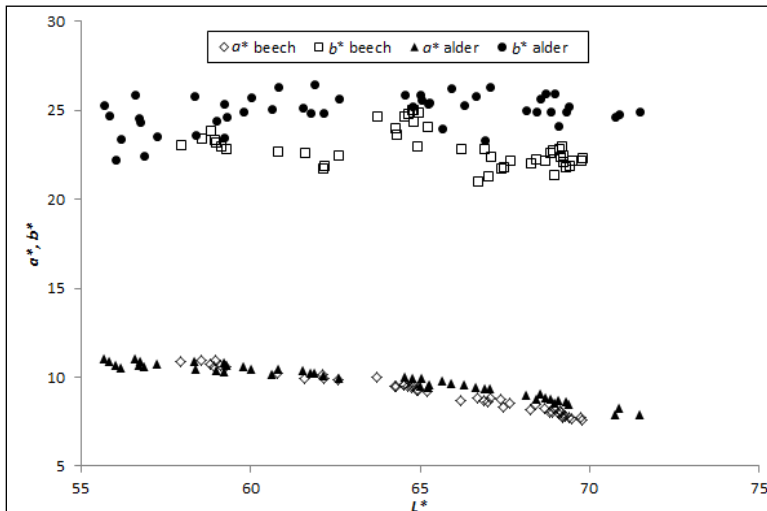
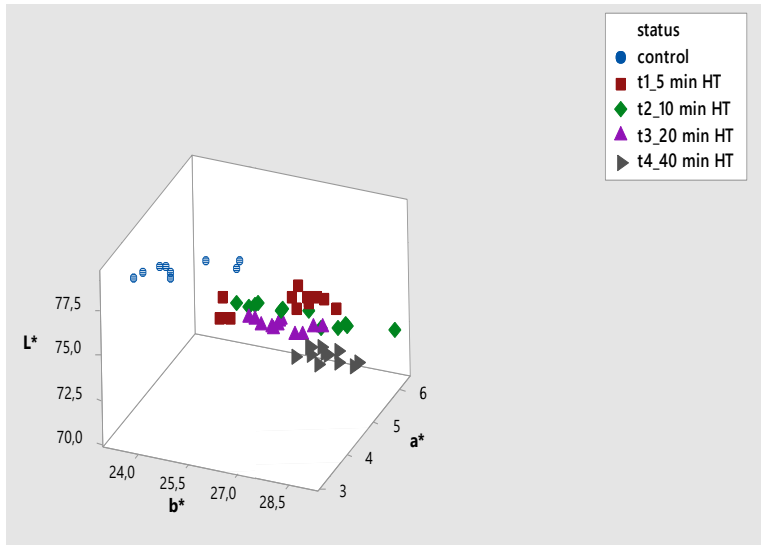


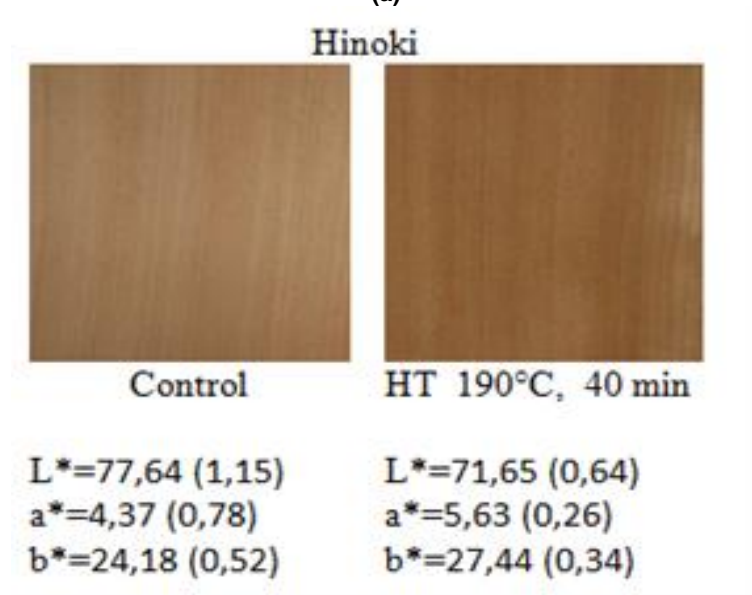
Fig. 11. Relation between a^* and b^* colour coordinates and L^* in heat treated veneers, plotted irrespective of exposure conditions [63]

It appeared that black alder veneers became redder than beech samples as the duration of heating process increased to 20 minutes. At the end of treatment both veneer species recorded almost same values of the red-green chromaticity ($a^*=10.67$ for black alder and $a^*=10.45$ for beech). Bekhta et al. determined that beech veneers showed a tendency to become redder while alder veneers became greener during the densification process at 200°C [54]. On the other hand, it is well-known that wood species of *Alnus* changes from a light-pinkish colour to a red/orange colour on exposure to air, which is caused by Maillard reactions between sugars and amino acids/proteins in wood [63]. Therefore, it was suggested that thermal modification of colour in red alder involves certain reactions which produce orange/red chromophoric groups in wood [63]. The yellow colour component of wood is influenced particularly by the photochemistry of lignin. Also, lignin derivatives, such as quinone and stilbenes generate the yellowing process [67]. Different variations of b^* values for the two wood species

under study were noticed. In case of black alder veneers, the direction of colour modification for b^* coordinate respected a decreased linear trend during the first 10 minutes of heat exposure, while for beech veneers an increase of yellow coordinate was noticed for the first 5 minutes of exposure. Next 5 minutes of heat exposure did not affect the yellow coordinate for beech veneers. The situation changed after that and for both species an increase of yellow coordinate was recorded at 20 minutes time span of exposure. At this point, the description of the colour evolution may be explained as an orange darkening considering the progress of colour chromaticity expressed by a^* and b^* coordinates. The same decreasing trend of yellow coordinate was observed during last time span of heat exposure for both veneer species. A similar evolution of the yellow colour component for thermo-densified veneers was mentioned by Bekhta et al. [54]. Such increase and decrease in colour coordinates a^* and b^* , for different heat treatment conditions, have been also reported for various wood species such as spruce, pine, oak, poplar, birch, sugi, keyaki, acacia [16,19,28,30,53]. The partial colour differences ΔL^* , Δa^* and Δb^* and the total colour differences ΔE^* depended on the heat treatment conditions. For both investigated wood veneers, the lightness difference ΔL^* trend decreased with increasing the heating time. The largest absolute value of lightness difference ΔL^* was observed at the longest heat exposure time of 40 minutes for black alder veneers ($\Delta L^* = -13.76$ for black alder and $\Delta L^* = -10.40$ for beech). Important chromatic differences for redness variation were noticed in the case of beech veneers ($\Delta a^* = 2.84$) when compared to black alder veneers ($\Delta a^* = 2.25$) for 40 minutes time span of heat exposure. A strong (Pearson) correlation of Δa^* with the lignin content was mentioned for heat treated beech while the smallest correlation between colour differences and chemical changes was found with cellulose [29]. The chromatic differences of yellowness recorded at 20 minutes time span of heat exposure presented for beech veneers the highest value among the two wood species ($\Delta b^* = 2.92$). The largest changes in total colour differences ΔE^* were observed at the end of the treatment, at 40 minutes time span of heat exposure for both veneer species. It was reported that the changes in L^* and ΔE^* of hinoki had similar degree to those of cellulose during heat treatment at 180°C. Carbonyl groups in the cellulose chains and the formation of low molecular substances (furan type) were estimated as being responsible for the colour changes [32]. Moreover, total changes of colour in thermally modified wood originates from chemical changes more in lignin than in polyssacharides, due to the darkening of lignin itself, which is associated with the generation of chromophoric groups [29]. The 3D scatterplot of the colour coordinates recorded for hinoki veneers before and after heat treatment and the partial and total colour changes are presented in Fig. 12 and Fig. 13, respectively [68]. The lightness (L^*) systematically and gradually decreased with increasing of exposure time while the redness (a^*) and yellowness (b^*) presented little variations. These results are in accordance with the specialty literature [69,70]. Similarities with other wood species under heat treatment were found [28,29,30,53,63].



(a)



(b)

Fig. 12. 3D Scatterplot of the colour coordinates of hinoki veneers before and after heat treatment (a) and the veneers appearance (b) [68]

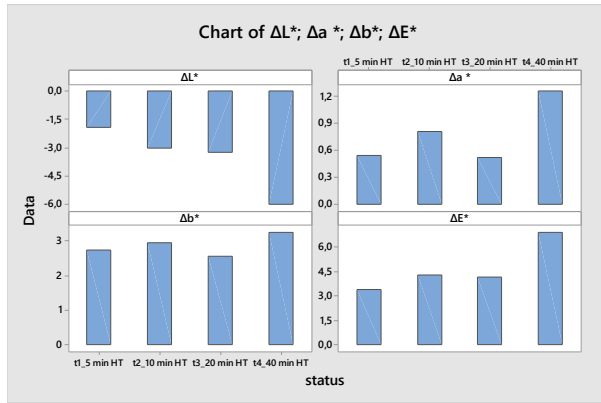


Fig. 13. Partial and total colour changes of hinoki veneers after heat treatment [68]

3.3 NIR Spectra Analysis of the Veneer Samples

Apart of CIELab colour measurement which was shown to give significant correlations with milled wood, NIR spectroscopy offers multiple advantages of allowing predictions for wood properties, like moisture content, density and stiffness [56]. NIR spectrum of the heat-treated wood gave information on the process extent and also on the wood relevant properties and uses. Therefore, NIR technique is a powerful tool used to determine chemical composition and to detect physical, mechanical and anatomical properties of wood materials [60].

Near infrared (NIR) spectroscopy has been widely used for measuring various properties of wood [60]. Most of absorption band assignments for wood appeared in NIR region is summarized in the review [59]. Esteves and Pereira have demonstrated that NIR could predict several parameters including CIELab parameters and dimensional stability of heat-treated pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood [56]. Gonzalez and Hale mentioned that physical properties of small specimens of heat-treated wood can be predicted with CIELab color space measurement [57]. According to Sandak et al., cellulose and lignin content of archeological oak wood were successfully predicted by NIR spectra with aid of partial least squares regression (PLSR) analysis [58]. It was found that such methods may be integrated in the production line in wood industry and also for heat treated wood with a view to assess the wood quality.

In this study, the diffuse reflectance NIR spectra were achieved from the surface of veneers by using a Fourier Transform Near Infrared (FT-NIR) spectrometer MatrixF-Bruker Optics (Fig. 14). The measured NIR spectra ranged between 10,000-4,000 cm^{-1} with 3.85 cm^{-1} interval. The spectral resolution was set to 8 cm^{-1} . FT-NIR measurements were performed three times on each sample in randomly selected zones in a climatic chamber. Each spectrum was achieved as an average of 32 scans. Bands were assigned as indicated by Schwanninger et

al. [59]. The second derivative was computed by gap-segment derivative (segment=7, gap=3 point) in order to eliminate the baseline drift and separate the overlap of absorption peaks. PLSR was then applied to build the predictive models for $L^*a^*b^*$ values. Each variable was mean centered and the maximum number of latent variable (LV) was set to five. Four third of data sets were randomly selected as calibration set and remaining data were used for test set validation. The optimum number of LV was determined where the minimum standard error of validation (SEV) was obtained. Data were processed by Matlab R2014a.

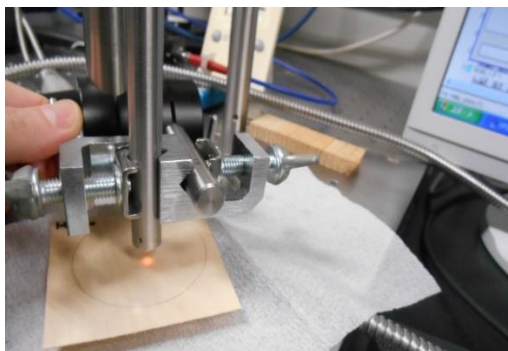


Fig. 14. Matrix-F Bruker Optics Spectrometer [63]

Heat treated black alder and beech veneers after different time spans of heat exposure at 190°C have been compared to control specimens. Changes of absorption bands were analysed in relation with the increase of heat exposure. Chemical changes due to heat treatment occurred for both wood species under study. Fig. 15 presents the FT-NIR second derivative spectra of black alder veneers for various time spans of heat exposure. Hemicelluloses (band 1725nm) were mostly affected by heat treatment as the heat exposure increased. It represents the first overtone of C-H stretching vibrations and is related to the degradation of hemicelluloses [71]. The effect of thermal exposure on hardwoods is associated to their higher acetyl and hemicellulose content [3]. Crystalline cellulose (1552nm and 1589nm) was slightly affected when compared to amorphous regions of cellulose (1428nm). A higher resistance of cellulose when compared to hemicelluloses under heat exposure was noticed. Esteves and Pereira reported that the glucose rate in hydrolysates increased under heating due to hemicellulose degradation [6]. Some shifts and changes in the case of lignin were noticed (band 1673nm). Deciduous lignin having guaiacyl and syringyl units is less stable than coniferous one which contains mainly guaiacyl units [58]. The lignin content increased with the increasing of heat exposure. Such increase in apparent lignin content related to the effect of polycondensation reactions with other components in the cell wall, which generated further cross-linking [3]. For both wood species here investigated changes in absorbance were observed for hemicellulose (1350nm, 1725nm, 2139nm), cellulose (1368nm, 1428nm, 1589nm, 1788nm, 2081nm) and lignin (1673nm, 1702nm, 2272nm).

Regarding the correlation between spectra and heat exposure and CIELab coordinates it was noticed the largest spectral variation at absorptions due to water (1919nm) and hemicellulose (2139nm). PLSR analysis was used to predict and express the colour coordinates by linear combination of absorbance at each wavenumber. A summary of PLSR model is presented in Table 2. Prediction models obtained for lightness L^* and redness a^* showed proper accuracy while the yellowness could not be predicted. Better coefficients were achieved for black alder veneers (Table 3). In case of yellowness prediction, samples were separated into two groups, namely: control and heat treated. This indicates there is great difference between untreated and heat-treated spectra while there is no great difference among heat treatment exposures. Fig. 16 shows the first 3 loadings for lightness L^* prediction model. As shown in Fig. 4, higher loading coefficient appeared at the wavelength range longer than 1800 nm. This may be due to the higher S/N ratio in longer wavelength region having stronger absorption. For both wood species, black alder and beech, the highest absolute value of LV1 and LV2 loading coefficient was observed at 1928nm and 1916nm, respectively. These regions are close to water absorption. This might be due to the indirect relationship between moisture content and lightness L^* . Based on hygroscopic properties of wood, the equilibrium moisture content decreased with the increase of thermal treatment exposure. There are many additional wavelengths related to lignin which contributed to lightness L^* prediction [63]. For both wood species, LV3 loading at 2272nm, which is assigned to lignin, showed almost the same negative value. In addition, in case of black alder, LV2 loading at 1723nm, which is assigned to hemicellulose, showed relatively high value in shorter wavelength region, while that for beech did not show any significant value. Effects of cellulose changes due to heat treatment on lightness L^* may differ between wood species. The results may help to clarify the relationship between colour appearance and changes of chemical components. Heat treatment applied to veneers from common or under-utilized wood species could therefore enlarge their use for high value product [63].

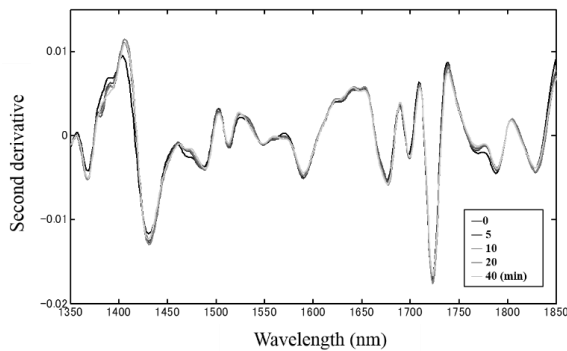


Fig. 15. Second Derivative Spectra of Alder Veneers as Function of Heat exposure – detail [63]

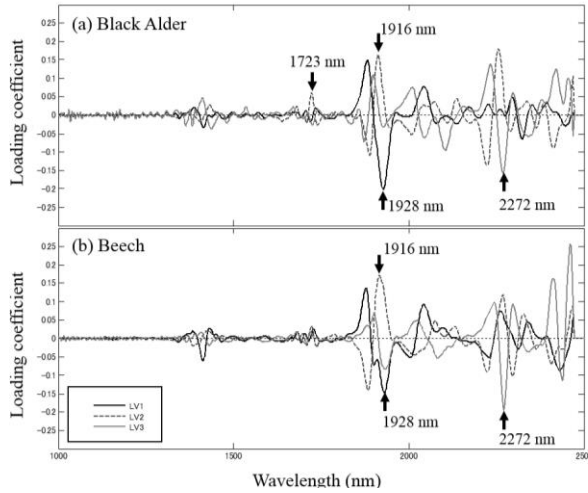


Fig. 16. Loadings for first 3 components of PLSR for L^* prediction models (a) black alder (b) beech [63]

Table 3. Summary of PLSR model [63]

Wood species	Objective	LV	R_c^2	R_v^2	SEC	SEV	RPD
Black alder	L^*	5	0.94	0.79	1.30	2.02	2.34
	a^*	5	0.90	0.68	0.30	0.43	1.92
	b^*	2	0.22	0.22	0.88	0.85	0.98
Beech	L^*	5	0.85	0.71	1.35	2.00	2.01
	a^*	5	0.82	0.75	0.42	0.51	2.15
	b^*	2	0.22	0.47	0.88	0.74	1.48

LV= Number of latent variables; R_c^2 =Determinant coefficient for calibration; R_v^2 =Determinant coefficient for test set validation; SEC=Standard error for calibration; SEV=Standard error for test set validation; RPD=ratio of performance (SD/SEV)

4. CONCLUSIONS

Heat treatment resulted in adverse effect on hardness characteristics of the solid wood samples. Hardness values of the specimens also decreased with increasing time and temperature treatments which can be related to deterioration of the cell wall structure after the heat treatment. It appears that strength losses can be limited through alternative modified heat treatment techniques. On the other hand, surface quality of the wood samples from all species was enhanced as a result of heat treatment. The progress of discoloration depended on each wood species. The changes in colour were caused mostly by the reduction in lightness which related to the degradation of hemicelluloses during heat treatment in wood species. It was found that black alder discoloured much more

than beech veneers under same treatment conditions. Moreover, prediction models by NIR for lightness and redness colour coordinates showed prediction accuracy. It seems that wood species having less commercial value can also be utilized effectively, following heat treatment, in areas where they previously had little potential. Therefore, such heat treatment would be considered to improve surface quality of the sample for furniture applications where smooth surfaces are ideal. The results in this study revealed that heat treatment can add potential value on wood material to be used more effectively in restoration works and furniture manufacturing [72,73].

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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