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Influence of steaming on selected wood properties of four hardwood species

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Abstract Influence of steaming on various mechanical and physical properties of two European (*Robinia pseudoacacia* L., *Quercus robur* L.) and two tropical (*Intsia bijuga*, *Hymenolobium petraeum*) hardwood species were investigated. Each of these wood species requires adequate adhesive systems for the use as dimension stock because of their highly reactive surface chemistry. In order to optimize the gluing behaviour of the timbers involved, steaming processes with five different sets of parameters (steaming time and temperature) were carried out. In addition to the adhesive test, bending strength, hardness, and colour of the modified timbers were examined.

The result of steaming highly depends on the wood species. For black locust, steaming is a suitable method of colour homogenization and colourization. Short term, low temperature treatment improves the adhesion performance also, whereas the colour change value reaches its maximum in the case of long term, high temperature steaming. Hue shift of oak and sapupira was inconsiderable for any applied set of parameters, only a small L* decrease was observed at higher temperatures. The colour of merbau samples shifted slightly during the treatment. Bending strength and hardness of wood samples in all of the four wood species decreased during the treatment. However, steaming time is more important than temperature while aspect of colour change both time and temperature has the same significance.

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M. E. van der Zee SHR Hout Research, PO Box 497, 6700 AL Wageningen, The Netherlands Adhesive properties of sapupira can be greatly improved by hydrothermal treatment.

Einfluss der Dämpfung auf verschiedene Eigenschaften von vier Laubholzarten

Zusammenfassung Der Einfluss der Dämpfung auf verschiedene mechanische und physikalische Eigenschaften von zwei europäischen (Robinia pseudoacacia L., Quercus *robur* L.) und zwei tropischen (*Intsia bijuga*, *Hymenolobium petraeum*) Laubholzarten wurde untersucht. Wegen der stark reagierenden Oberflächenchemie ist für jede dieser Holzarten ein spezielles Klebstoffsystem erforderlich. Um das Verklebungsverhalten dieser Holzarten zu optimieren, wurden Dämpfungen mit fünf verschiedenen Parameterkombinationen (Dämpfungsdauer und Temperatur) durchgeführt. Neben den Klebstoffprüfungen wurden die Biegefestigkeit, die Härte und die Farbe der behandelten Hölzer untersucht. Das Ergebnis der Dämpfung hängt dabei stark von der Holzart ab. Für die Robinie ist die Dämpfung zur Farbhomogenisierung und Färbung gut geeignet. Das Klebeverhalten wird auch bei einer Dämpfung von kurzer Dauer und niedriger Temperatur verbessert, wohingegen Farbänderungen ihren Maximalwert bei einer Dämpfung von langer Dauer und hoher Temperatur erreichen. Bei Eiche und Sapupira ergaben sich bei allen Versuchsreihen keine nennenswerten Änderungen des Farbtons. Bei höheren Temperaturen war lediglich eine geringe L*-Abnahme zu beobachten. Die Farbe der Merbauproben veränderte sich im Verlauf der Dämpfung leicht. Während der Dämpfung nahmen bei allen vier Holzarten die Biegefestigkeit und die Härte ab. Dabei spielt die Dämpfungsdauer eine größere Rolle als die Temperatur, wohingegen bezüglich Farbänderung beiden die gleiche Bedeutung zukommt. Die Klebeeigenschaften von Sapupira können durch eine hydrothermische Behandlung stark verbessert werden.

1 Introduction

Improvement of wood quality via hydrothermal treatment has a tradition in excess of 60 years. Steam acts by dissolving or altering (oxidizing, decomposing) extractives and other chemical constituents of the wood. There are several scientific studies and papers related to changes of physical and mechanical properties of specific wood species, experiencing to solve particular technological problems. Numerous researches have been done on the subject of steam treatment of black locust (*Robinia pseudoacacia*). Most of these studies investigated the colour change and colour homogenization of this extremely inhomogeneous species in order to reach a more preferable wood texture for the market (Molnár et al. 1998, Tolvaj and Faix 1996, Tolvaj et al. 2000, Tolvaj et al. 2002). Several papers deal with the steaming of beech wood (Irmouli and Haluk 2005, Ledig et al. 2004), and a remarkable development project pointed out that aesthetic problems of beech core can be solved by steaming (Tolvaj et al. 2002, Tolvaj and Molnár 2006). Çolak and Çolakoğlu (1996) reported about the effect of steaming on some mechanical properties of Okumumè plywood. It is also well known that heat treatment, combined with light-irradiation, effects intensive changes in the chemical composition of wood, which result in a dynamic colour change (Mitsui et al. 2001, Mitsui 2004, Mitsui 2006).

It was found that there is a reduction of all of the strength properties of timbers during steaming (Fábián 1976). Generally, the decrease of strength values is proportional to the density decrement. Hydrothermal treatment causes the most significant changes in impact bending strength, but during a treatment above $100\degree C$, all of the strength properties decrease considerably. Therefore, it is necessary to gather information on changes of strength properties caused by steaming, especially for wood species being used in a structural capacity.

The simultaneous effect of heat and moisture generates radical changes in the wood structure. Oxidation and decomposition of wood constituents have a strong influence on wettability, which may be of interest to further processing. Surface free energy data can be used for understanding the wetting processes between timber surface and different liquid materials, such as dyes and adhesives (de Meijer 1999). Contact angle and surface free energy data allows us to calculate the adhesion work at the interface between wood and other matters (de Meijer and Militz 1998), or they can be used to estimate the change of chemical constituents of wood surface after diverse treatments (Pecina and Paprzycki 1990, Casilla et al. 1981), and ageing processes (Kainins and Knaebe 1992, Nussbaum 1995). Recently, Lifshitz–van der Waals (γ^{LW}) and (Lewis) acid-base components are being used to determine surface free energy (Gardner 1996, Shen et al. 1998). Methods developed to define surface free energy of wood are generally based upon the measuring of static contact angle of the fixed drops or the measuring of dynamic contact angle (Gardner et al. 1991). Wood surface has low surface free energy independently of the measuring techniques (de Meijer 1999). Values are typically between 30 and 50 mJ m⁻². It was found that the sessile drop method is the most applicable contact angle measuring method for wood. Although it depends on grain orientation, additional measurements are unnecessary.

Colour is imparted to wood by extraneous materials (tannins, etc.) called extractives. These chemical compounds make it possible to artificially change and homogenize natural wood colour by application of chemicals (Miller et al. 1985, Mitsui and Tolvaj 2005), water, or heattreatment (Mitsui 2006, Ayadi et al. 2003, Tolvaj and Faix 1996). European beach is often steamed to darken and homogenize its colour and to make it more desirable for the furniture market (Tolvaj et al. 2001, Tolvaj et al. 2002, Molnár and Tolvaj 2004, Tolvaj and Molnár 2006). Tolvaj et al. (2000) reported that steaming below $100\degree$ C can reduce the heterogeneity of the natural colour of black locust (*Robinia pseudoacacia*).

This paper deals with responses of two European (*Robinia pseudoacacia*, *Quercus robur*) and two tropical (*Intsia bijuga*, *Hymenolobium petraeum*) hardwood species to hydrothermal treatment performed with different combinations of process parameters (time and temperature). Each of these wood species especially black locust and sapupira requires adequate adhesive systems because of their highly reactive surface chemistry. This study is aimed at optimization of gluing behaviour of the timbers involved by steaming as well as measuring other important wood properties such as bending strength, hardness, contact angle and colour that may alter during thermal treatment. Surface free energy was calculated from the measured contact angle values. Adhesion performance was characterized by means of shear strength measurement of glued joints. Colour change caused by steam treatment was given in the CIEL*a*b* system.

2 Materials and methods

2.1 Materials

This study examined the heartwood of black locust (*Robinia pseudoacacia*), oak (*Quercus robur*), mearbau (*Intsia bi-* *juga*), and sapupira (*Hymenolobium petraeum*). Before processing, raw materials were stored at normal climate (20 ◦C, 65% RH) for at least two weeks. Six sets of test materials per wood species were prepared, one of them considered as control series. Table 1 shows the size and number of specimens of each series.

2.2 Hydrothermal treatment

After shaping samples, indirect steam treatment was carried out in a cylindrical pressure chamber. Net steaming time values for black locust, merbau, and sapupira are as follows: 3 and 20 hours at 108 ◦C, 7.5 hours at 115 ◦C, 3 and 20 hours at 122 °C. Steaming temperatures of oak had to be reduced to 92 ◦C, 100 ◦C, and 108 ◦C because of frequent ray checks.

2.3 Colour measurements

The colour of the surface of freshly cut and planed specimens was measured with a Minolta CM 2600d spectrophotometer. The sensorhead was 10 mm in diameter. Measurements were made using a D65 illuminant and a 10◦ standard observer. The spectrophotometer directly indicates the lightness (L^*) and hue co-ordinates $(a^*$ and $b^*)$ so there is no need for additional calculation.

2.4 Measuring of strength properties

Abr

The bending strength of samples was measured with 3 point loading parallel to the grain, according to DIN 52186. For hardness measurements, the Janka method was applied. Sample materials for the adhesion test were modified in one piece. After steaming, samples were shaped according to EN 205 (1998). Two 5 mm thick panels were glued with ordinary PVAC adhesive and then pressed. After hardening, a set of samples was stored under normal climate for one week and another one in cold water for 24 hours. Adhesion performance of untreated and steamed samples was then evaluated by calculating the shear strength of glued joints:

$$
\tau = F/A \tag{1}
$$

where *F* indicates the shear force, and *A* indicates the glued area.

2.5 Surface free energy measurements

Surface free energy of untreated and steam treated timber surfaces was calculated using their contact angle data measured on freshly cut, planed surfaces with the sessile drop method. Components of surface free energy were calculated using the contact angle of diiodomethane, formamide, and water (Table 2) based on the following equation (Good 1993):

$$
0.5\gamma_1(1+\cos\Theta) = \sqrt{\left(\gamma_s^{\text{LW}}\gamma_1^{\text{LW}}\right)} + \sqrt{\left(\gamma_s^-\gamma_1^+\right)} + \sqrt{\left(\gamma_s^+\gamma_1^-\right)}.
$$
\n(2)

Lifshitz–van der Waals component of surface free energy (γ_s^{LW}) can be obtained from the contact angle of diiodomethane (subscript ,,1"):

$$
\gamma_s^{\text{LW}} = 0.25 \gamma_1^{\text{LW}} (1 + \cos \Theta)^2. \tag{3}
$$

Combined acid-base component (γ_s^{AB}) of surface free energy is as follows:

$$
\gamma_{\rm s}^{\rm AB} = 2\sqrt{\gamma_{\rm s}^+} \sqrt{\gamma_{\rm s}^-} \,. \tag{4}
$$

Complete surface free energy of wood (γ_s) :

$$
\gamma_{\rm s} = \gamma_{\rm s}^{\rm LW} + \gamma_{\rm s}^{\rm AB} \,. \tag{5}
$$

 $\mathbf{1}$

After Janka-hardness test, samples were cut and planed before measuring of colour and contact angle ² Samples for shear strength test were shaped after steaming

Table 2 Physical properties and surface free energy values of liquids at 20 ℃ applied by contact angle measurements (Van Oss 1994) Tabelle 2 Physikalische Eigenschaften und freie Oberflächenenergie der Flüssigkeiten bei 20 ℃ mittels Kontaktwinkelmessung (Van Oss 1994)

Liquid	Density kg/m ³	Viscosity mPa s	νL $\mathrm{mJ\,m^{-2}}$	LW $\mathrm{mJ}\,\mathrm{m}^{-2}$	$mJ m^{-2}$	$\mathrm{mJ\,m^{-2}}$	λ . AB $mJ m^{-2}$
Diiodomethane	3325	2.8	50.8	50.8			
Water	1000	.00	72.8	21.8	25.5	25.5	51.0
Formamide	799	.02	58	39	2.28	39.6	19

3 Results and discussion

3.1 Colour change caused by steam treatment

The colour change of samples was highly dependent upon the wood species. Table 3 presents the influence of treatment parameters (temperature and time) on the colour of wood samples. A major shift of L^* and b^* occurred in the case of black locust (Figs. 1 and 3) and merbau. These species contain numerous extraneous water soluble materials (Harborne and Baxter 1999, Imamura et al. 1974) that affect their natural colour, as well as their behaviour, during heat treatment. A large quantity of robinetin can be found in these species. Therefore, it is thought that this extractive is responsible for the considerable yellow hue shift of black locust and merbau. Yellow hue of black locust depends both upon steaming time and steaming temperature, whereas the b* co-ordinate of merbau shifted in the same degree independently of treatment parameters. The incrusted robinetin that is visible to the naked eye on the surface is thought to be much easier dissolvable than in the case of black locust. Therefore, yellow colour diminishes even at lower steaming temperature. The L* co-ordinate of black locust and merbau is influenced both by treatment temperature and time. The higher the treatment temperature and/or the longer the treatment, the darker colour can be reached. Steaming at $122\degree C$ for 20 hours results in a very intensive lightness change: black locust becomes darker than the untreated merbau.

a* shift of black locust (Fig. 2) is considered as important as yellow change because the natural colour is yellowish rather than reddish. During the short treatment at low temperature, red hue did not change significantly, however, if

Fig. 1 Mean lightness value of untreated and steamed black locust samples

Abb. 1 Mittlerer Helligkeitswert von unbehandelten und gedämpften Robinienproben

the temperature is higher and/or the treatment time is longer, a* co-ordinate can be more than twice as high as that of untreated sample independently of process parameters. Red hue of merbau decreased equally at any combination of steaming parameters. Analogously to b* shift, explanation is thought to be the same.

The red- and yellow hue shift of sapupira and oak was inconsiderable at any applied steaming parameter. It is assumed that extractives of these species are hard to dissolve with water. However, the L*-co-ordinate changes significantly at certain parameters. In case of sapupira, a long treatment at high

Table 3 CIELab colour co-ordinates of untreated and steam treated wood specimens measured on freshly cut and planed surfaces (Lower temperature values apply to oak; $SD = standard deviation$ **Tabelle 3** CIELab Farbkoordinaten von frisch eingeschnittenen und gehobelten Oberflächen von unbehandelten und gedämpften Proben (die niedrigeren Temperaturen gelten für Eiche; $SD =$ Standardabweichung)

Fig. 2 Mean red hue value of untreated and steamed black locust samples

Abb. 2 Mittlerer Rotwert von unbehandelten und gedämpften Robinienproben

Fig. 3 Mean yellow hue value of untreated and steamed black locust samples

Abb. 3 Mittlerer Gelbwert von unbehandelten und gedämpften Robinienproben

temperature darkens the wood colour, whereas lightness of oak is affected by steaming temperature. Thermal processes are less intensive under 100◦C since there is no overpressure.

3.2 Changes in strength properties

Bending strength and hardness of each investigated wood species decreased during steaming. Table 4 shows the ratio of the bending strength value of treated and untreated samples for each species. Black locust and oak seem to be more sensitive to hydrothermal treatment than the tropical species

Table 4 Ratio of bending strength values determined before and after treatment (Lower temperature values apply to oak)

Tabelle 4 Verhältnis der Biegefestigkeit vor und nach Behandlung (die niedrigeren Temperaturen gelten für Eiche)

Steaming parameters	Black locust Merbau		Sapupira	Oak
$108/92$ °C, 20 h	59%	83%	81%	63%
$122/108$ °C, 20 h	60%	75%	78%	59%

but the bending strength of treated black locust is still higher than that of untreated oak (Fig. 4). Bending strength diminishing of oak was remarkable even at low temperature (Fig. 5), if a long treatment was implemented. It is interest-

Fig. 4 Mean bending strength value of untreated and steamed black locust samples

Abb. 4 Mittlere Biegefestigkeit von unbehandelten und gedämpften Robinienproben

Fig. 5 Mean bending strength value of untreated and steamed oak samples

Abb. 5 Mittlere Biegefestigkeit von unbehandelten und gedämpften Eichenproben

ing that bending strength value of treated samples is influenced by steaming time rather than temperature. This fact is especially obvious in the case of black locust and oak (Figs. 4 and 5).

The hardness of treated samples depends on the steaming time and temperature (Fig. 6). Generally, the higher the temperature and/or the longer the treatment the lower is the hardness value. Black locust is the most sensitive among the investigated wood species: mean hardness of samples steamed at 122 ℃ for 20 hours is half of the initial value.

3.3 Shear strength of glued joints

A description of the adhesion performance of untreated and steam treated wood surfaces was given through the shear strength of glued joints. Table 5 contains the calculated mean shear strength values of glued joints made of untreated and steamed wood samples. Standard deviation values are also displayed. The adhesion performance of black locust can be improved by steaming with proper process parameters. Treatment at high temperature, however, caused diminishing of shear strength. Supposedly, this is because of the extreme change of the other strength properties mentioned above. Steaming may optimize the surface chemistry, but at the same time, strength properties decrease with increasing treatment time as has been demonstrated in Fig. 4. This also applies to sapupira. Here, the best adhesion performance was reached through a steam treatment at 115 ◦C.

Shear strength of glued joints made of merbau did not change significantly. Calculated values of treated samples are the same or slightly lower than that of untreated samples. For oak, steaming at any applied set of parameters was disadvantageous from the view point of adhesion performance.

3.3.1 Contact angle and surface free energy

Modification of wood surface chemistry caused by steam treatment effects the change of contact angle (Table 6). The contact angle between wood and water may be of impor-

Fig. 6 Mean hardness value of untreated and steamed samples Abb. 6 Mittlere Härte von unbehandelten und gedämpften Proben

Table 5 Effect of steaming parameters on the shearing strength of glued joints made of the investigated wood species (Lower temperature values apply to oak; $SD =$ standard deviation)

Tabelle 5 Einfluss der Dampfungsparameter auf die Scherfestigkeit geklebter Verbindungen der untersuchten Holzarten (die niedrigeren Tem- ¨ peraturen gelten für Eiche; $SD = Standardabweichung$)

Table 6 General changes of contact angle between wood surfaces and different test liquids after hydrothermal treatment (↓: contact angle decrease, ↑: contact angle increase, –: no change)

Tabelle 6 Tendenzielle Veränderungen des Kontaktwinkels zwischen Holzoberfläche und verschiedenen Flüssigkeiten nach hydrothermischer Behandlung (↓: Kontaktwinkelabnahme, ↑: Kontaktwinkelzunahme, -: keine Anderung)

Wood species	Diiodomethan	Water	Formamide
Black locust	◡		
Merbau	◡		
Sapupira	◡		
Oak			

tance to practice (water soluble systems). During a long term, high temperature treatment, black locust and merbau surfaces became strongly hydrophobic. However, contact angle between oak and water decreased by all of the applied steaming parameters (Fig. 7) which ensures a better wettability of the wood surface.

The calculated surface free energy values are also influenced by the measured proper contact angle of diiodomethane and formamide. The surface free energy of black locust and merbau slightly decreased during hydrothermal treatment but there were no obvious relationships between the process parameters and the final energy value. It is thought that other parameters, such as trachea diameter or the number of tracheas per unit area, that cause undesirable capillary penetration of test liquids, have a strong influence on contact angle and, therefore, on surface free energy value. After a long treatment at 122 ℃, surface free energy of sapupira increased significantly (Fig. 8). The calculated energy value of treated oak surfaces were higher than those of untreated samples.

Fig. 8 Surface free energy of untreated and treated sapupira samples Abb. 8 Freie Oberflächenenergie bei unbehandelten und behandelten Sapupiraproben

4 Conclusions

Dueto the modified chemical structure of wood caused by the simultaneous effect of heat and moisture, all of the investigated mechanical and physical properties of the examined wood species have been changed. Dissolving, oxidizing, and decomposing of extractives are thought to be the main reason for colour change and modification in surface chemistry. Black locust and merbau timbers contain a large quantity of water soluble extractives therefore the colour change of these species was the most intensive. The a* and b* co-ordinates of merbau decreased independently of process parameters, whereas L^* and b^* diminishing of black locust and L^* di-

minishing of merbau are greatly influenced by steaming time and temperature. The red hue of black locust shifted independently of treatment parameters at higher temperatures. The a* and b* shift of sapupira and oak were inconsiderable at any applied process parameters, however, lightness decrease during a long treatment at 122 ◦C was significant.

Generally, mechanical properties decayed with higher steaming temperature and/or longer treatment time. Bending strength of oak and black locust was influenced by steaming time rather than temperature.

Shear strength of glued joints was affected by two processes: changing of wood surface chemistry and decreasing of mechanical properties. Changes in surface chemistry caused an improvement or deterioration of wettability depending on wood species. It was found that measurements of contact angle with sessile drop method can hardly be used in order to describe the changes during a steam treatment process because results are strongly influenced by other factors such as anatomical structure that causes unwished penetration of test liquids. It is a fact, however, that adhesion performance of black locust and sapupira can be improved by proper steam treatment. It was found that contact angles between oak and water rose significantly during steam treatment and it also caused an increase in surface free energy, resulting in an improved wettability. A drastic decrease of strength properties induced, however, a significant fall of shear strength of glued joints made of oak timber.

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