

Oil Uptake Percentage in Oil-Heat-Treated Wood, its Determination by Soxhlet Extraction, and its Effects on Wood Compression Strength Parallel to the Grain

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Oil heat treatment can effectively improve the dimensional stability and bio-durability of wood. However, the characteristics of high oil uptake (50 percent or higher) and high susceptibility to leaching from wood have an adverse effect on subsequent manufacturing processes of wood product and production costs. A solvent extraction (100% ethanol) process was used to extract the surplus oil from the treated wood. Because the making of powder specimens from high oil uptake wood would result in experimental error, a new method of determination of oil uptake percentage was proposed by two step Soxhlet extraction with ethanol. The oil uptake percentage of oil-heat-treated poplar wood at 180 °C for 2 h with this method was determined to be 113.29%. Additionally, the average oil uptake percentage (OUP) of oil-treated poplar after the oil extraction process for 2 and 6 h was 54.82% and 29.11%, respectively. Moreover, the average oil extraction percentage (OEP) of specimens with the oil extraction process at the first step (wood sticks) in the Soxhlet extractor apparatus was larger than that of oil-heat-treated poplar specimens. Due to the combined effect of chemical changes in the wood at high temperatures and of oil uptake by the wood, the compression strength parallel to the grain changed at different OUP levels.

Keywords: Poplar; Soxhlet extraction; Oil uptake percentage; Oil extraction percentage; Compression strength parallel to grain; Chemical analysis

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INTRODUCTION

It has been noted that thermal treatment of wood changes its physical and chemical properties. As a result of such treatment, the wood's related defects, such as swelling due to increased moisture, are reduced (Bak and Németh 2012) and its biological durability enhanced (Yildiz *et al.* 2006). Since industrial vegetable oils are good heat transfer media and are potential carriers that can further enhance the benefits of the treatment, various hot oils have been used for thermal treatments. The dimensional stability and durability of wood, especially those of fast grown plantation timbers, can be improved by oil heat-treatment without application of any persistent toxic chemicals. In addition, the mechanical properties of oil-heat-treated wood are a little better than heat treatment of wood in different gaseous atmospheres (Rapp and Sailer 2001). The physical and chemical properties of wood would be altered permanently after heat treatment over 150 °C in gaseous atmospheres; at the same time, the mechanical properties would be decreased by heat (Mitchell 1988), and the wood becomes more brittle; bending and tensile strengths decrease by 10 to 30%.

The good mechanical properties of oil-heat-treated wood are related not only to the oxygen-free atmosphere (Stamm 1956) during the heat treatment but also to the high oil uptake of treated wood. However, high quantities of oil absorbed by wood during heat treatment (50 percent or higher) (Tjeerdsma *et al.* 2005) and the properties of high susceptibility to spill out of wood can have an adverse effect on further process and production costs, such as sawing and planing processes, ornament painting, and so on. How to extract surplus oil from oil-heat-treated wood and calculate accurately the oil uptake percentage will be important for the better utilization of oil heat treatment.

Solvent extraction, beginning in 1870, has been the primary means of extracting vegetable oil from oleaginous materials in Europe (Hron *et al.* 1994). The most widely used solvent for extraction of oil from oilseeds is hexane, because it has a high solubility for fats and oils, high chemical stability, and a suitable vapor pressure, allowing separation by flash and stripping operations (Wan *et al.* 1995; Setha *et al.* 2007). However, hexane is flammable, explosive, and harmful for air emissions and human contact, and therefore it is important to find a bio-renewable solvent alternative to hexane (Hron and Koltun 1984). Ethanol at or near its boiling point is a good solvent for vegetable oils (Sato *et al.* 1934), is obtainable from bio-renewable resources, and is one of four vegetable oil solvents (water, ethanol, butane, and propane) that are generally recognized as safe.

Wood is a porous material having macropores, mesopores, and micropores (Zhao 2002). This system of pores provides natural channels for the extraction of oil. Anderson and Hermann (1950) and Delius and Becker (1961) separately used acetone and hexyl alcohol as solvents for the extraction of pine gum from pine, and the result showed that solvent extraction can decrease the content of pine gum effectively and there is no influence on the mechanical properties.

Based on the analysis given above, the solvent extraction process was employed to extract the superfluous oil from oil-heat-treated wood. A two-step Soxhlet extraction method, used to determine oil uptake percentages of oil-heat-treated poplar wood, was established. Furthermore, the compression strength parallel to the grain of oil-heat-treated wood at different oil uptake percentage levels was investigated; additionally, the corresponding changes in chemical components and the SEM analysis of different wood specimens were studied in this work.

EXPERIMENTAL

Oil Heat-treatment

Poplar (*Populus* spp.), about 10 years old, was obtained from a wood plantation in Ciyang, Jiangsu, China. The poplar was processed to create flat sawn lumber with a thickness of 22 mm and a width of 160 mm. The length of each specimen was approximately 260 mm. The specimens were then conditioned in a conditioning chamber to 10 to 12% moisture content.

In the oil-heat treatment, wood specimens were immersed with clamping in a reactor at 180 °C for 2 h with soybean oil. After treatment, the heating was turned off, specimens were not removed until the temperature decreased to 80 °C, and then the oil was drained. Following this, treated specimens were wiped and cooled down for the subsequent experiment.

Determination of Oil Uptake

Wood samples for solvent extraction were prepared from the oil-heat-treated wood. The oil in the treated wood was at a high level and easily expelled from the wood in the course of grinding, which would lead to oil loss of the treated wood. Therefore, wood slices were split by chopper from each wood sample to ensure the error was as minor as possible. Each slice, having dimensions of 30 mm (length) × 22 mm (width) × 2 mm (thickness), was then cut it into tiny sticks with a length of 30 mm, a width of 2 mm, and a thickness of 2 mm.

First step of Soxhlet extraction

The sticks, weighing 40 g, were wrapped up with chemical analytical filter paper and put into the Soxhlet extractor apparatus. The organic solvent extraction followed the procedures in ASTM standards (ASTM D1105-96). The solvent used was 100% ethanol. Wood sticks were then put into the extractor and extracted with ethanol for 6 h, or longer if necessary, until there was no excess oil loss in the subsequent grinding process. Extraction with 100% ethanol should be carried out at a rate of no less than 5 siphonings per hour. Then the extracted sticks were oven dried at 85 °C (boiling temperature of 100% ethanol is 78.3 °C) until free of ethanol; the weight of sticks in this way was recorded as m_0 (g). Meanwhile, the round-bottom flask was put into the water bath, and the flask was entirely surrounded by the water at 85 °C for 4 h. Finally, the flask without ethanol was oven dried at 103 °C, until there was no weight change. The total oil extracted in this way was recorded as m_1 (g).

Second step of Soxhlet extraction

The above-mentioned oven-dried sticks were ground in a plant mill to homogeneously mixed powders. Poplar powders were sieved down to 30 to 40 mesh in order to acquire a relatively uniform sample. Poplar powder samples weighing 6 g, accurate to 0.0001 g, were gained from above wood sticks and divided into two groups, which each were wrapped in analytical filter paper and then were placed in siphon tubes. Extraction with 100% ethanol was carried out at a rate of no less than 5 siphonings per hour. The round-bottom flask was taken out every 2 h, and at the same time, the new flask with new ethanol was fixed and extraction continued. The flask with oil was put into the water bath at a temperature setting of 85 °C; then the flask without ethanol was oven-dried at 103 °C, until there was no weight change. The process was continued until no oil could be found in the flask, and then the ending extraction time was determined. The total oil extracted in this process was recorded as m_2 (g). Finally, the extracted powders were oven-dried at 103 °C until there were no weight changes and recorded as m (g).

The oil uptake percentage (OUP) and the oil extraction percentage (OEP) of specimens were determined from the following equations,

$$OUP = \frac{m_1 \times \frac{6}{m_0} + m_2}{m} \times 100\% \quad (1)$$

$$OEP_1 = \frac{m_1 \times \frac{6}{m_0}}{m_1 \times \frac{6}{m_0} + m_2} \times 100\% \quad OEP_2 = \frac{m_2}{m_1 \times \frac{6}{m_0} + m_2} \times 100\% \quad (2, 3)$$

where m_0 (g) is the weight of sticks after the first Soxhlet extraction, m_1 (g) is the total weight of oil extracted with sticks in first Soxhlet extraction, m_2 (g) is the total weight of oil extracted with wood powders in the second Soxhlet extraction, and m (g) is the weight of the extracted powders. The values for OUP and OEP in this study were the average of 2 replicates.

Oil Extraction Process

The oil extraction experiment was carried out in a 10-L laboratory stainless steel treated reactor. Each wood sample was 20 mm (tangential) \times 20 mm (radial) \times 30 mm (longitudinal).

A vacuum of 0.01 MPa and pressure of 0.9 MPa were employed for all the oil extraction trials. Firstly, the sample was under a vacuum for 30 min, and then the ethanol was pumped to the treated reactor at a pressure of 0.9 MPa for 30 min. Secondly, pressure was released, and the temperature was kept constant at 85 °C for 2 h; 6 h, during which the ethanol was continuously recirculated to ensure the operating temperature within one degree of the set temperature. Subsequently, the ethanol was drained and the sample was kept under a vacuum for 30 min to remove the remaining oil on the wood surface. Finally, wood specimens were taken out and oven-dried at 85 °C until free of ethanol.

Compression Strength Parallel to Grain

Prior to the test, the dimensions were measured to the nearest 0.001 mm, and their weights were recorded to the nearest 0.01 g. The samples were then conditioned in a conditioning chamber at 25 °C and 65% relative humidity. The values for compression strength parallel to the grain were recorded using a Universal Mechanical Test Machine according to the Standard Methods of Testing Small Clear Specimens of Timber-ASTM D 143.

Chemical Analysis of Poplar Components

Before chemical analysis of the specimens, including oil-treated poplar wood, the treated wood after oil extraction for 2 and 6 h were chopped to a length of 1 to 2 cm and ground in a plant mill to a homogeneous meal. The method for the quantitative determination of lignin was based on Klason's technique, involving hydrolysis with 72% sulfuric acid. The solution was then placed in an autoclave for 1 h at 121 °C. The lignin content was determined as acid-insoluble Klason lignin and acid-soluble lignin by the NREL/TP-510-42618 method. Cellulose and hemicellulose content were calculated by the recovered sugar that was determined by HPLC analysis.

RESULTS AND DISCUSSION

Measurement and Calculation of Oil Uptake

Table 1 lists the oil extraction percentage (OEP) in two-step extraction based on Soxhlet extraction of oil treated poplar wood (including OEP at 2 h intervals for the second step extraction).

Table 1. Oil Extraction Percentage (OEP) In Two Step Soxhlet Extraction

| Specimens | Extraction time (h) | Extracted oil (g) | Oil extraction percentage (%) |
|---|---------------------|-------------------|-------------------------------|
| Poplar sticks (the first step extraction) | 6 | 3.2423 | 71.31 |
| Poplar powders (the second step extraction) | 2 | 0.6290 | 13.83 |
| | 4 | 0.3932 | 8.65 |
| | 6 | 0.1607 | 3.53 |
| | 8 | 0.0848 | 1.87 |
| | 10 | 0.0271 | 0.60 |
| | 12 | 0.0086 | 0.19 |
| | 14 | 0.0011 | 0.02 |

It was found that the oil could be extracted mostly during the first Soxhlet extraction step with poplar wood sticks using 100% ethanol. In the second Soxhlet extraction step, the oil extraction percentage decreased gradually at 2 h intervals, and there was no more oil extracted by ethanol at an extraction time up to 14 h. Thus, the Soxhlet extraction time used to oil heat-treated poplar wood in the second step extraction was 14 h.

SEM Analysis

SEM photographs can establish the microstructure of a material. Images of wood specimens with the oil-heat treatment and the oil extraction process are shown in Figs. 1 and 2, respectively.

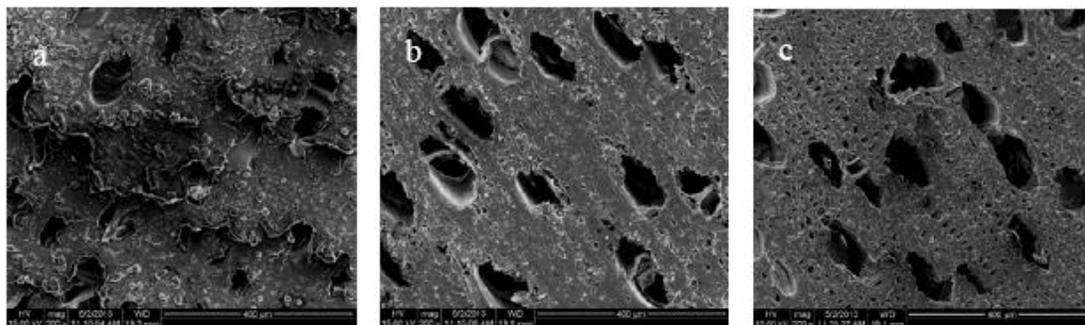


Fig. 1. SEM images of oil-heat-treated poplar (a), treated poplar with oil extraction process for 2 h (b), and for 6 h (c) (cross section)

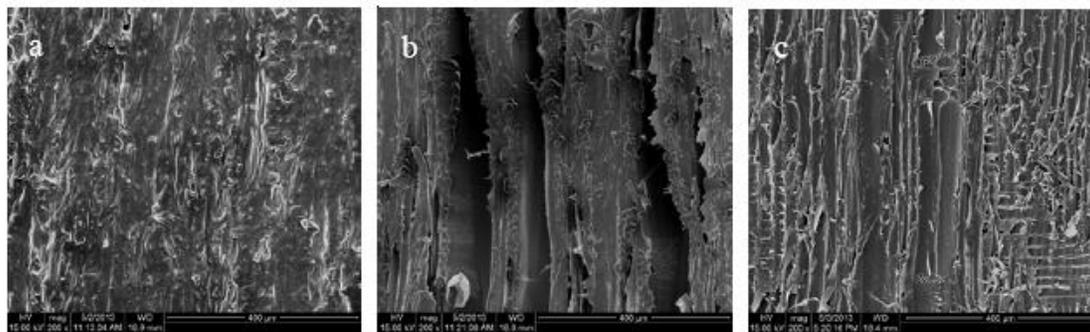


Fig. 2. SEM images of oil-heat-treated poplar (a), treated poplar with oil extraction process for 2 h (b), and for 6 h (c) (radial section)

For oil-heat-treated poplar wood, the wood specimen surfaces were sealed with thick soya bean oil, as can be seen in Figs. 1a and 2a. The microstructure of the oil-treated poplar wood cannot be seen because of the oil accumulated on the wood surface. In the case of oil-heat-treated poplar wood with high oil uptake percentage, when the wood was placed in contact with pure solvent, the first thing that happened was dissolution of the soya bean oil on the surface. Secondly, the oil in wood lumens began to be extracted by solvent as the surface oil dissolved. In the case of wood specimens with oil extraction process for 2 h, the wood lumens exposed after the oil extraction process are shown in Figs. 1b and 2b. There was not much oil on the surface of wood specimens following the oil extraction process compared to after the oil treatment. Meanwhile, small pores on the cell wall can be seen (Figs. 1c and 2c.) after an oil extraction process for 6 h.

OUP and OEP of Different Specimens

As can be seen in Table 2, the average oil uptake percentage of oil-heat-treated poplar with Soxhlet extraction was 113.29%. The remaining oil in oil-treated poplar after an oil extraction process of 2 h was 54.82%. And that of oil-treated poplar after an oil extraction process of 6 h was 29.11%. Figure 3 shows the effect of the specimens before and after the oil extraction process on the oil extraction percentage at different phases with Soxhlet extraction. The average oil extraction percentage of wood specimens at the first step (wood sticks) was 72.53%, while the second step (wood powders) was 27.47%. Wood is a porous material; the pores in the wood were open, supplying sufficient channels to allow oil to be extracted from the wood. Meanwhile, Hietala *et al.* (2002) showed that mild thermal temperatures (115 °C) did not change the size or distribution of cell wall micropores, and temperatures in excess of 180 °C could significantly increase both pores size and broadened the microporosity distribution, which supplied natural channels for extraction of oil from wood. In addition, the vacuum and pressure employed in the oil extraction process enhanced solvent impregnation.

Table 2. Oil Uptake Percentage (OUP) Of Different Treatments and Corresponding Compression Strength Parallel to Grain

| Treatments | Powders oven-dried weight (g) | Extracted oil (g) | Oil uptake percentage (%) | Compression strength (MPa) |
|-------------------------------|-------------------------------|-----------------------|---------------------------|------------------------------|
| Control | | | | 42.83 (3.58) _(SD) |
| Oil heat treatment | 4.3806 | 3.6492 ⁽¹⁾ | 112.98 | 45.2 (3.66) |
| | | 1.2999 ⁽²⁾ | | |
| | | 4.9491 ^(T) | | |
| Replicate | 4.0028 | 3.2432 ⁽¹⁾ | 113.59 | |
| | | 1.3063 ⁽²⁾ | | |
| | | 4.5468 ^(T) | | |
| Average | | | 113.29 | |
| Oil extraction process for 2h | 5.7085 | 3.0754 ⁽¹⁾ | 54.58 | 45.79 (2.93) |
| | | 0.0405 ⁽²⁾ | | |
| | | 3.1159 ^(T) | | |
| Replicate | 5.6854 | 3.0886 ⁽¹⁾ | 55.07 | |
| | | 0.0421 ⁽²⁾ | | |
| | | 3.1307 ^(T) | | |
| Average | | | 54.82 | |
| Oil extraction process for 6h | 5.7319 | 1.6309 ⁽¹⁾ | 28.83 | 43.6 (3.14) |
| | | 0.0215 ⁽²⁾ | | |
| | | 1.6524 ^(T) | | |
| Replicate | 5.7211 | 1.6574 ⁽¹⁾ | 29.4 | |
| | | 0.0247 ⁽²⁾ | | |
| | | 1.6821 ^(T) | | |
| Average | | | 29.11 | |

¹:The oil extracted from the first step Soxhlet extraction; ²: The oil extracted from the second step Soxhlet extraction; ^T: total oil; ^{SD}: Standard deviation

The average oil extraction percentage of wood specimens after the oil extraction process for 2 h and 6 h at the first step (wood sticks) was 98.68% and 98.62%, respectively. It is suggested that the wood sticks can be used to determine the oil uptake percentage of oil-heat-treated wood with the oil extraction process, and the second step extraction could be omitted in industrial applications of this approach.

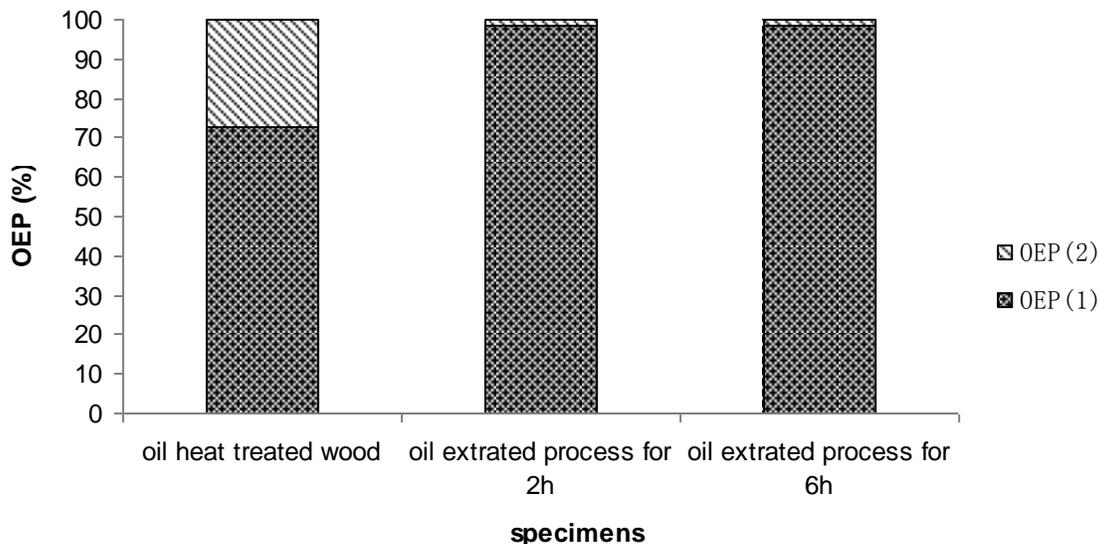


Fig. 3. Oil extraction percentage of different treatments at different extraction phase

Analysis of the Compression Strength Parallel to Grain of Wood

As can be seen from Table 2, the value of compression strength parallel to the grain was 45.20 MPa (OUP was 113.29%), 45.79 MPa (OUP was 54.82%), and 43.60 MPa (OUP was 29.11%), respectively. The value of the compression strength parallel to the grain of treated poplar wood increased compared to the control group, and the value was found to be a little bit higher than untreated wood when the oil extraction process was applied to treated poplar wood for 2 h. The weight of specimens increased due to the high oil uptake (113.29%) compared to the loss of substance after treatment, and high oil uptake in the specimens might have filled the lumens and coated the walls; such filling would be expected to thicken the fibers greatly, increasing their lengthwise strength. Compared to the control, the value of the compression strength parallel to grain of oil-heat-treated poplar was increased slightly. The oil uptake percentage decreased from 113.29% to 54.82%, and there were no more differences in terms of the compression strength parallel to grain between specimens with oil treatment and oil extraction processes. This is mainly because most of the oil extracted from wood was present in wood lumens, and this part of the oil has no influence on wood properties, as is the case for free water in wood not affecting the mechanical properties. Nevertheless, in the same oil extraction process for 6 h, the compression strength parallel to the grain decreased compared to the oil-treated poplar and oil extraction process for 2 h, and the oil uptake percentage decreased from 54.82% to 29.11%, as can be seen from Table 2. For heat treatment in a gaseous atmosphere, changes in the chemistry of wood at high temperature are considered to be the principal reason for the alteration of wood properties (Yildiz *et al.* 2006). However, for oil-heat-treated wood, the combined effect of chemical changes in the wood at high temperature and the effect of oil uptake by wood during the heat-treatment should be considered. The diminution in the strength properties at this level of oil uptake percentage (29.11%) was mainly related to the rate of thermal degradation, and the depolymerization reaction of poplar chemical component played a more important role than oil uptake with respect to the wood chemical properties.

Chemical Analysis of Poplar Wood Components

The results of the chemical analysis of poplar wood components with oil heat treatment and with oil extraction process are given in Table 3.

Table 3. Chemical Composition of Oil-Heat-Treated Poplar Wood and Poplar Wood with Oil Extraction

| Chemical Composition | Control | Oil heat treatment | Oil extraction for 2 h | Oil extraction for 6 h |
|----------------------|---------|--------------------|------------------------|------------------------|
| Glucan | 48.86 | 49.42 | 49.15 | 49.45 |
| Xylan | 24.35 | 19.93 | 19.51 | 20.84 |
| Klason lignin | 23.47 | 25.31 | 22.29 | 21.75 |
| Acid-soluble lignin | 0.62 | 0.54 | 0.61 | 0.67 |

Hemicelluloses have lower thermal stability, are highly disordered, and are relatively easy to hydrolyze compared to celluloses (Yang *et al.* 2007). Xylan (pentosan) is the most reactive wood hemicellulose and, in general, pentosans are very sensitive to degradation and dehydration reactions (Manninen *et al.* 2002; Kotilainen 2000). The xylan content was observed to be 19.93%, and glucan content increased slightly to 49.42% after oil-heat treatment. Klason lignin content increased to 25.31% after oil-heat treatment, which has also been reported in the literature (Nuopponen *et al.* 2004). This is mainly caused by degradation of the hemicellulose and partial degradation of cellulose during heat treatment.

Glucan and xylan contents exhibited no significant change after the oil extraction process for 2 h and 6 h compared to oil-heat treatment. However, the Klason lignin content was decreased to 22.29% and 21.75% separately after oil extraction process for 2 h and 6 h, which means that the Klason lignin content was decreased compared to untreated poplar wood, as can be seen from Table 3. In the heat treatment of wood, carboxylic acid, mainly acetic acid, was formed by the cleavage of the acetyl groups of the hemicellulose in wood (Dietrichs *et al.* 1978; Bourgois and Guyonnet 1988). An explanation for this is that high-temperature treatment promoted delignification in an acidic environment, and long oil extraction time resulted in the formation of small fragments of the lignin. The fragments were dissolved in ethanol and unable to be recovered, leading to low lignin yield. There was no substantial change in the Klason lignin after oil extraction process from 2 h to 6 h. The hydrogen ion concentration (lower pH value) was characteristic to the oil-heat-treated poplar wood, and the corresponding small fragments of the lignin were determined. The oil extraction time for 2 h was able to achieve good lignin solubility. Further increase in the extraction time did not result in any decrease of the lignin content. In summary, the oil extraction process did not significantly change the cell-wall constituents, which indicates that the transformations of the chemical wood structure were caused by oil heat treatment.

The diminution in the strength properties at the level of oil uptake percentage (29.11%) was mainly related to the rate of thermal degradation. Hemicellulose was considered as an interfacial coupling agent between the highly polar surface of the microfibrils and the much less polar lignin matrix. Hemicellulose degradation results in wood becoming brittle and rigid, indicating the important role that it has in imparting

viscoelastic properties to wood. Lignin provides stiffness to the secondary cell wall and cohesion between cells, and consequently to wood tissue (Buchanan *et al.* 2000), and it has a strong influence on mechanical properties of wood (Nakajima *et al.* 2009). The chemical degradation of wood occurs in the hemicellulose and lignin components during heat treatment, which means that the bonding substances present in the fibrils decreases. Consequently, the compression strength parallel to grain decreases with reduced content of bonding substances.

CONCLUSIONS

- (1) To address problems related to high oil uptake in oil-heat-treated wood, ethanol was used as a solvent to extract the surplus oil from the wood. A new method for determination of the wood's oil uptake percentage (OUP) was proposed and demonstrated; the two-step Soxhlet extraction procedure involved the wood sticks being extracted with ethanol for 6 h and wood powders being extracted with ethanol for 14 h as a means of determining the oil uptake.
- (2) The oil extraction process was found to effectively extract the oil from the oil-heat-treated wood. The compression strength parallel to the grain was correspondingly changed at different OUP level as a result of the combined effect of chemical changes in the wood at high temperature and the effect of oil uptake by wood.
- (3) The average oil extraction percentage (OEP) of oil-heat-treated poplar was 72.53% during the first step of Soxhlet extraction. In the case of oil-heat-treated poplar, most of its oil (98% or so) could be extracted by 100% ethanol during the first step of extraction. Thus, for industrial purposes, the oil uptake percentage (OUP) could be determined quickly only by extraction with wood sticks (the first steps of Soxhlet extraction).

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