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EFFICACY OF LINSEED OIL-TREATED WOOD TO IMPROVE HYDROPHOBICITY, DIMENSIONAL STABILITY, AND THERMOSTABILITY

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ABSTRACT

In this work, linseed oil was impregnated into the wood at room temperature, under vacuum pressure. The properties of linseed oil-treated wood, including dimensional stability, wood moisture absorption, chemical structure, thermostability, and morphological characteristics, were evaluated. Linseed oil displayed good permeability in Ailanthus wood, with weight gains of 30.95% after impregnation. The swelling coefficients of treated wood in the tangential and radial directions decreased by 25.97 to 33.33%, indicating that impregnation improved the dimensional stability of wood. Moreover, linseed oil treatment significantly modified the wood structure, although the FTIR spectra generally remained unchanged. Observation by scanning electron microscopy showed, that linseed oil impregnated into the wood and occluded pits, thereby prevented moisture absorption. This technique can be used in a variety of wood products, such as buildings, furniture, and landscape architecture.

KEYWORDS: Linseed oil treatment, swelling coefficients, thermostability, wood dimensional stability.

INTRODUCTION

Wood is a complex lignocellulose material, its low dimensional stability and durability limits its widespread use. Previous research has led to improvements wood durability, dimensional stability, and hydrophobicity using steam-heat treatment (Wang et al. 2019, Saeed et al. 2016), acetylation and silylation (Ziegler et al. 2008), alkoxysilane modification (Broda et al. 2018), siloxane modification (Giudice et al. 2013), silicone oil heat treatment (He et al. 2019, Qian et al. 2019, Okon et al. 2017), and tricine and bicine modification (Popescu et al. 2020), among many others (Turkoglu et al. 2015). However, thermal treatments may darken the original wood color and attenuate its mechanical properties (Jiang et al. 2020, Sun et al. 2019, Lin et al. 2018, Li et al.

2017, Esteves et al. 2011, Esteves and Pereira 2009, Esteves et al. 2008), whereas chemical treatments may damage the environment. Moreover, many of these modifications are complex and consume large amounts of energy.

Some natural materials are widely used to improve the performance of wood due to their environmentally friendly properties, such as wax, shellac and some vegetable oils (Yang and Liu 2020, Yang et al. 2020, Liu et al. 2020a,b, Dubey et al. 2011, 2012a,b). Linseed oil is produced from the dried ripe seed of the flax plant (*Linum usitatissimum*), which is cultivated for oil and fiber (Yang and Luo 2013). Once exposed to air, the boiled linseed oil can form a siccative but transparent film on a wood surface (Schönemann and Edwards 2011, Lazzari and Chiantore 1999). Linseed oil is environmentally friendly and low-cost with excellent water repellent capacity (Eriksson et al. 2011, Fredriksson et al. 2010). It is commonly applied to conserve archaeological wood (Lucejko et al. 2018). At present, linseed oil is widely utilized for the production of inks, paints, soap, varnishes, and many other products (Arrieta et al. 2017); however, it has not been commonly utilized to enhance the hydrophobicity of wood products.

Following some studies of natural materials impregnated wood dimensional stability (Liu et al. 2020a,b, He et al. 2019), this paper examines the use of linseed oil as an environmentally friendly modifier to increase wood dimensional stability and hydrophobicity. The objectives of this research were to examine *(1)* the effects of linseed oil on wood chemical and physical structures, dimensional stability, and thermostability, and *(2)* the drying mechanisms of linseed oil.

MATERIALS AND METHODS

Sample preparation

Ailanthus (*Ailanthus Desf.*), a commonly cultivated white-colored wood with a similar grain to white oak, was obtained from Qian Heng Mo Zhong company, Sichuan, China. Sapwood specimens with dimensions of 20 × 20 × 20 mm (longitudinal section × radial section × cross-section) and a primal moisture content of 70 ± 5% (mean ± standard error) were prepared and used in the experiments (D4933, 2016). The wood specimens were heat-dried at 103°C before the experiments.

Impregnating the wood with linseed oil

Linseed oil was obtained from Fabryo Company (Brasov, Romania). The impregnation was conducted in a vacuum chamber produced by Shanghai Laboratory Instrumental Works Co., Ltd. (Shanghai, China). Twenty dry specimens were weighed, submerged into linseed oil, and then placed in the vacuum chamber under a pressure of 0.01 MPa and 20°C for 1.5 h. Subsequently, the pressure was allowed to equilibrate to atmospheric level for 1.5 h. The wood specimens were impregnated with linseed oil by vacuuming and reversing the process three times. The wood specimens that were not impregnated with linseed oil were regarded as the control group. Following the treatment, these specimens were naturally dried for 7 days until the linseed oil was cured, dried at 103°C, and then weighed.

Weight percentage gain (WPG)

The specimens were weighed immediately before and after the impregnation. The WPG was determined based on the weight and was calculated by Eq. 1:

$$WPG=\frac{w_w-w_0}{w_0}\times 100\ \% \tag{1}$$

where: w_0 represents the dry weight of specimens before the impregnation (g), and w_w represents the dry weight after the impregnation (g).

Treatment means were compared with a t-test at P = 0.05 in SAS (v. 9.4, SAS Institute, Cary, NC).

Estimation of wood dimensional stability

The swelling was measured following the ASTM D4933-2016 standard. All wood specimens were oven-dried and then placed in a climate chamber at 20°C with 65 % humidity to achieve the equilibrium moisture content (EMC). The dimensions and weights were determined before and following the conditioning. The swelling coefficient was computed with Eq. 2:

$$a=\frac{l_w-l_0}{l_0}\times 100\ \% \tag{2}$$

where: a denotes the swelling coefficient for assessing the longitudinal, tangential, and radial parameters of the specimens, l_w denotes the dimension after conditioning, and l_0 represents the initial dimension of the specimens.

Treatments were compared with t-tests in SAS at p = 0.05.

Moisture absorption

Moisture absorption (MA) experiments were performed following the ASTM D4933: 2016 standard. The wood specimens were placed in a climate chamber under a constant temperature of 20°C with 65% relative humidity to reach the EMC. After conditioning in the climate chamber, the MA was calculated using Eq. 3:

$$MA=\frac{w_a-w_b}{w_b}\times 100\ \% \tag{3}$$

where: w_b and w_a denote the weights before and after the conditioning (g), respectively.

Treatments were compared with t-tests in SAS at p = 0.05.

Chemical structure analysis using FTIR spectroscopy

Attenuated total reflectance infrared (ATR-IR) spectra of the control group and linseed oil-treated wood specimens were collected using a standard FTIR spectrometer (Tensor 27, Bruker, Germany) via direct transmittance at a resolution of 4 cm^{-1} for 32 scans at 500 – 4000

cm^{-1}. The background spectra and the light equipment were aligned immediately before the measurements. The spectra were averaged over six measurements for each treatment before data analysis.

Thermogravimetric analysis

A thermogravimetric analyzer (Netzsch STA449F3, Germany) was employed to measure the degradation characteristics of wood specimens. The untreated control and linseed oil-treated wood powder were placed in a chamber filled with nitrogen and heated at a rate of $10°C \cdot min^{-1}$ until reaching an ultimate temperature of 800°C.

Morphological characteristics

To investigate the potential variations in the physical structures, an environmental scanning electron microscope (SEM) (Quanta 200, FEI Company, the Netherlands) was employed to observe the surface shapes of wood specimens. Increase in electrical conductivity of a sample is the single most common requirement for SEM. In this study, a sputter gold coating (2 nm) was applied on the wood specimens using a Gold Palladium SEM Annular Sputtering target 2" ID × 3" OD × 0.1mm Anatech (SC502-314; Quorum Technologies, Ltd., Watford, UK). Bombarding voltage used for SEM was 20.00 kV.

RESULTS AND DISCUSSION

Weight percentage gain (WPG)

WPG value indicates the net weight of the wood penetrated by linseed oil, as shown in Tab. 1. This depends on the permeability of linseed oil inside the Ailanthus wood. The dry weight of the untreated wood specimens measured 4.497± 0.233 g; however, after impregnating with linseed oil, the dry weight and WPG measured 5.889 ± 0.199 g and 30.95%, respectively.

Wood dimensional stability

Wood dimensional stability is an important factor affecting its utilization and quality, but it depends on tree species, tree age, and age of the wood, among other factors. The radial and tangential swelling coefficients are critical factors to assess the wood dimensional stability because they are more sensitive than those in the longitudinal direction. The linseed oil impregnating treatment significantly improved wood dimensional stability ($P < 0.0001$) in tangential and radial directions but not in a longitudinal direction ($P = 0.1112$) (Fig. 1). The swelling coefficient in the tangential direction of the untreated control measured 3.08%, while the linseed oil-treated wood measured 2.17%.

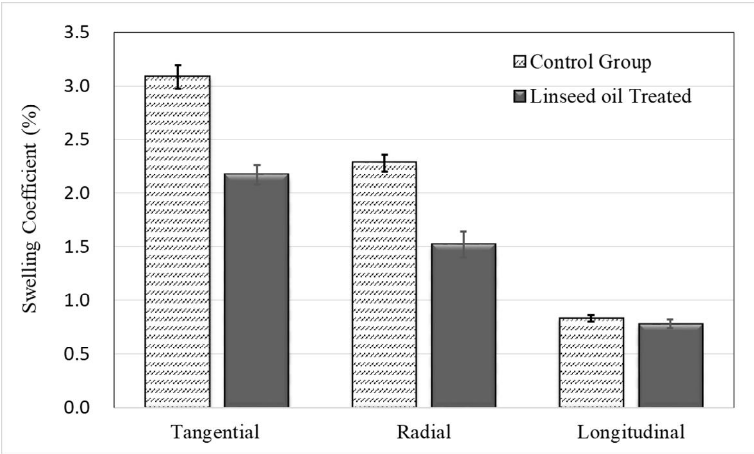

Fig. 1: Swelling coefficients in different directions.

After the treatment with linseed oil, the swelling coefficient in tangential direction decreased by 25.97% compared with the control group. The swelling coefficient of the control group in the radial direction measured 2.28%, while the treated group measured 1.52%. The swelling coefficient of the linseed oil-treated specimens was 33.33 % lower compared with the control group. The control and linseed oil-treated specimens exhibited similar longitudinal swelling coefficients and measured 0.83% and 0.78%, respectively. These observations indicated that the linseed oil treatment can significantly decrease wood swelling coefficients in the radial and tangential directions but not in the longitudinal direction. This is logical as wood tends to shrink and swell in the radial and tangential directions, but not in the longitudinal direction. The increased wood dimensional stability could be attributed to the excellent water repellent property of linseed oil (Humar and Lesar 2013).

Moisture absorption

When the moisture content of wood is under the fiber saturation point (FSP), it can significantly affect wood stability (Yang and Liu 2018). To further elucidate the impact of linseed oil impregnation on wood dimensional stability, the hydrophilicity of wood was evaluated by moisture absorption (MA). The specimens' weights before and following the conditioning in the climate chamber are shown in Fig. 2. After conditioning in the climate chamber, the weights of the control group varied between 4.497 g and 5.033 g before and after conditioning, and the MA was 11.92%. The treated specimens' weights changed and varied from 5.889 g to 6.213 g, and the MA was 5.50%. Linseed oil treatment can reduce water MA Ailanthus wood by 53.86%.

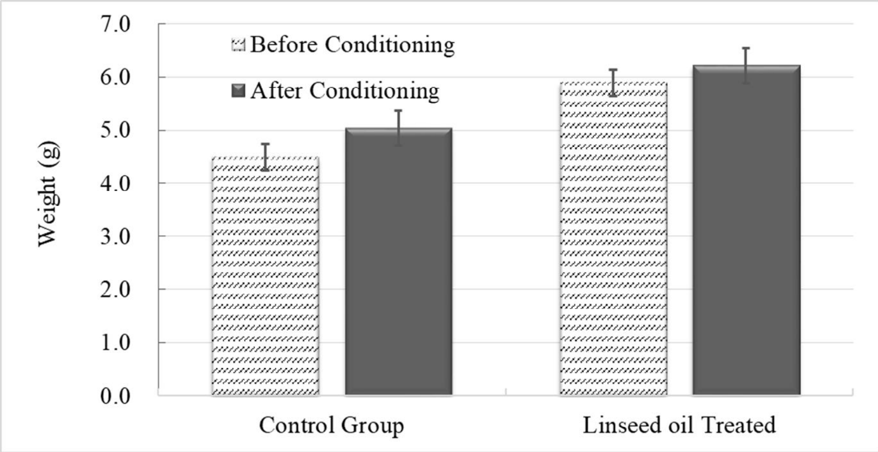

Fig. 2: Weights of the specimens before and after conditioning.

Chemical structure analysis using FTIR spectroscopy

Wood dimensional stability is related to its hydrophilic chemical composition, which generally contains hydroxyl groups and other chemical components. The reduction of these components can improve stability (Jiang et al. 2015). FTIR-ATR spectrometry has been widely employed to reveal chemical changes associated with various treatments. Infrared spectra are sensitive indicators of chemical changes, as demonstrated by previous research in the fields of wood treatments (Basso et al. 2017).

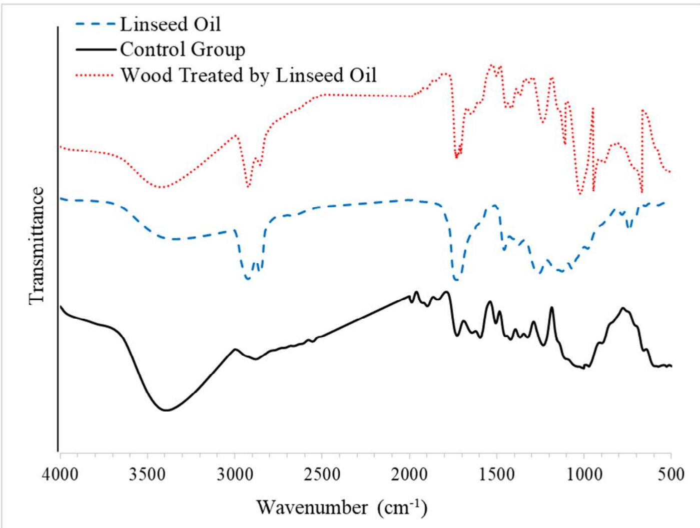

Fig. 3: FTIR spectra of the samples treated by linseed oil.

Fig. 3 presents the FTIR spectra results in the untreated control and the linseed oil-treated specimens. Although the spectra were largely unchanged, the linseed oil treatment modified the chemical structures of the wood specimens. The significant decrease of band intensities at ~3400 cm^{-1} corresponded to −OH stretching. Since linseed oil treatment reduces the relative number of hydroxyl groups (He et al. 2019), it improves the dimensional stability of the wood. Moreover, the band intensity at 1384 cm^{-1} and 2900 cm^{-1} (−CH stretching), and the bands at 1729 cm^{-1} (C=O) and 1604 cm^{-1} (aromatic carbon skeleton stretching vibrations) changed (Liu et al.

2020a,b, He et al. 2019, Esteves et al. 2011). After being treated with linseed oil, another chemical groups did not change. Thus, linseed oil treatment changed some of the chemical groups in the wood, but the composition of the wood remained the same.

Thermostability

The thermal characteristics of wood specimens treated with linseed oil were evaluated using the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves (Fig. 4).

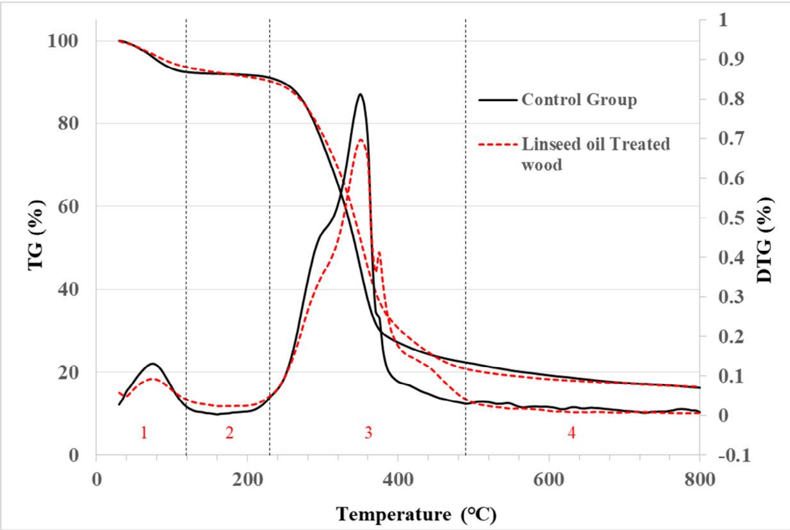

Fig. 4: TG and DTG curves of the wood samples treated.

The TG curve in the first region (marked by "1") represents depolymerization and dehydration. A 6–8% weight reduction was noted in the range of 30 to 120°C in all cases. At this stage, the wood composition does not degrade significantly (He et al. 2019), although the wood's moisture evaporates and some small molecules are degraded. In the range of 120°C to 230°C (marked by "2"), some wood components, such as hemicellulose, may degrade (Esteves and Pereira 2009, Li et al. 2017, 2018) while the chemical bonds of other complex wood components may break. The most mass loss occurred at the temperature of 230°C – 490°C (marked by "3"), accounting for about 70% of the total mass. At this stage, all the structural components of the wood sample underwent thermal degradation. Also, in this region, linseed oil-treated samples reacted differently, with two peaks appearing at 355°C and 375°C. By contrast, at 355°C, the DTG curve of the control samples is more common and has a peak. This spike can be attributed to the breakdown of cellulose (Giuntoli et al. 2009). In region "4," the mass loss of the sample decreases gradually. At this stage, compared with the control group (16.30 wt%), the residue content after linseed oil treatment decreased by 16.59%. The reduction in residue may be due to the degradation of most linseed oil and the inability of some wood components to decompose.

Morphology and mechanism

To evaluate physical structures changes, the SEM micrographs of the treated samples and control group are illustrated in Fig. 5. Results showed that linseed oil treatment significantly affected the tissue structure of the samples. Pits are the main moisture channels in the wood interior. After linseed oil treatment, some of the pits were occluded, and prevented MA under the environmental conditions, thus improving the dimensional stability of wood. Also, some areas show cured linseed oil (marked by a circle) attached to the internal surface of the wood. Thus, when linseed oil is coated on the inner surface of the wood, it prevents MA and improves the wood dimensional stability.

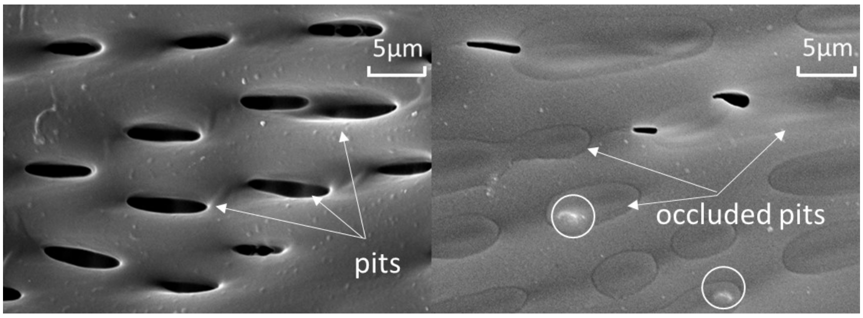

Fig. 5: SEM micrographs of the control group and l treated group.

Linseed oil is mainly composed of linoleic acid (14 – 19%) and linolenic acid (48 – 60%) (Lazzari and Chiantore 1999). The typical mechanism of the linseed oil drying process is hydrocarbon oxidation, which is promoted by the active hydrogen atom at the vinyl position of the molecule (Fig. 6). In linoleic acid and linolenic acid components, one or two methylene groups are located between two unbound double bonds and the formation of hydrogen peroxide is easier to achieve. Then W-type pentadienyl can react with oxygen at both ends to form a hydroperoxide mixture with trans and cis conjugation. Also, the reversibility of oxygen addition leads to the isomerization of W radicals, which can be converted into trans products. The alkoxy radicals produced by the decomposition of hydroperoxides may lead to the formation of oxygen-containing structures, such as alcohols and different carbonyls, or crosslinked products. These radicals are polymerized with other linseed oil molecules. Finally, the crosslinking density of linseed oil increases and a curing film is formed on the inner surface of the wood. As such, the water transport pathway is blocked, resulting in improved wood dimensional stability (Liu et al. 2020a,b, He et al. 2018).

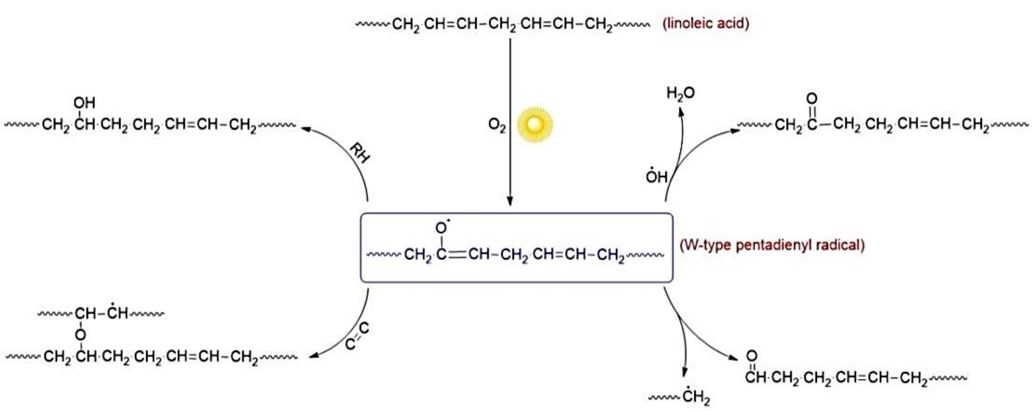

Fig. 6: The drying mechanism of linseed oil (Lazzari and Chiantore 1999).

CONCLUSIONS

Linseed oil impregnation treatment is an effective technique to improve wood stability. Linseed oil has good permeability in the wood and the WPG reached to 30.95% after impregnation. After the treatment with linseed oil, the swelling coefficients of wood in the tangential direction and radial direction decreased by 25.97% and 33.33%, respectively, resulting in improved wood dimensional stability. The moisture absorption of linseed oil-treated wood was reduced by 53.86% under the condition of 20°C and 65% relative humidity. Also, linseed oil treatment changed the structure of the wood to some extent, but the spectrum remained unchanged. In TG analysis the DTG curve of the control samples was more common with a steep peak at 355°C. The samples treated with linseed oil had two decomposition peaks around 355°C and 375°C. Linseed oil impregnates into the wood, occludes pits, and solidifies at the internal wood surface, thereby preventing moisture absorption. This technique can be used in a variety of wood products, such as buildings, furniture, and landscape architecture.

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