

IMPREGNATION ON POPLAR WOOD WITH VEGETABLE OILS: EFFECTS ON PHYSICAL MECHANICAL AND DIMENSIONAL STABILITY PROPERTIES

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ABSTRACT

In this study, vegetable oils were selected to modify poplar with vacuum-pressure impregnation technology and the optimum progress was studied. The weight percent gain (WPG), modulus of rupture (MOR), modulus of elasticity (MOE), water uptake rate (WUR), volume swelling rate (VSR) and volume shrinkage rate (VSR') were evaluated. The results showed that the dimensional stability, physical and mechanical properties of treated wood were significantly improved. Scanning electron microscopy (SEM) observations revealed that castor oil was inserted into the interior of the wood through the pores. Vegetable oil modification (castor oil) decreased the intensities of hydroxyl, cellulose and hemicellulose specific peak in the Fourier transform infrared spectroscopy (FT-IR) results. The Fourier transform infrared spectroscopy (FT-IR) results showed that castor oil treatment decreased the intensities of hydroxyl, cellulose and hemicellulose specific peak. Ultimately, the optimum process of castor oil treatment was impregnation pressure 1.8 MPa, time 1.0 h and temperature 85°C based on the range and variance analysis.

KEYWORDS: Poplar, vegetable oils, dimensional stability, physical and mechanical properties, range and significance analysis.

INTRODUCTION

With the rapid development of the global economy, the development of the timber industry has shown a trend of diversified investment, large industrial scale, and high industrial agglomeration, which has led to a substantial increase in the demand for timber (Köhl et al. 2021). With the prohibition of logging in natural forests, the lack of high-quality wood and the increasing demand for lumber in China, fast-growing timber has been entering the market because of its fast growth rate, short rotation cycle and high yield. The development and

utilization of timber resources have gradually moved away from natural forests to fast-growing plantations. Fast-growing species have gradually become the main raw material in the woodworking industry (Guan and Chen 2021). Compared with natural forest wood, fast-growing trees have many disadvantages (such as low density, soft texture, etc), which restrict the application of fast-growing wood and reduce its value (Han et al. 2020).

At present, researchers concentrate on wood modification with environmentally friendly modifier (plant extracts, wax, vegetable oil, tannin), which is an important research direction for non-formaldehyde modification. Vegetable oils are permeable and film-forming to wood for protection and coating finishing improving the durability of wooden utensils (Ozgenç et al. 2013). Castor oil is extracted from seeds and contains 35% -57%. Castor oil is a naturally occurring fatty acid triglyceride, 90% of which is ricinoleic acid (9-enyl-12-hydroxy octadecanoic acid) (Paes et al. 2010). Priadi et al. (2021) was reported that jabor wood preserved with boron solutions was treated with castor oil and heat treatment. The authors stated that the water absorption of wood and leaching of boron preservation was reduced by heating 160°C and castor oil treatment, and the decay resistance of treated wood was increased. Chang and Lu (2017) investigated that the film of castor oil-based wood coating had superior film hardness, gloss, and flexibility, which could be applied for wood finishing. Turkey red oil is obtained from castor oil by sulfonation, precipitation, neutralization and other reaction processes, which was applied for wood protective coating (Chen et al. 2020). Turpentine is a by-product from the production of rosin, which were widely used in industry and medicine. Several studies have explored that vegetable oils (tung oil, linseed oil, palm tung oil, and Litsea cubeba oil) impregnated wood (Humar and Lesar 2013, Temiz et al. 2013, Zhou et al. 2020, Chen et al. 2020), and less studies have been done with castor oil, turkey red oil and turpentine.

The objective of this study is to explore the method of deep modification based on the advantage of vegetable oil in wood surface modification. The effects of three different vegetable oils impregnating modified poplar wood were preliminarily studied, and the most effective vegetable oil was selected. Then, the influences of impregnation pressure, time and temperature on wood properties were further investigated.

MATERIALS AND METHODS

Materials

Wood samples of poplar wood (*Populus euramericana* cv. I-214) were prepared from plantations in Jiangsu province, China. The sapwood from specimens was sawed from log segment in the dimension of 53 mm × 53 mm × 310 mm (tangential × radial × longitudinal). Poplar samples were selected based on flat surfaces and no cracks (moisture content 8-10%, average absolute dry density 0.41 g·cm⁻³). Poplar wood samples (*Populus euramericana*) were impregnated with castor oil (0.955 g·cm⁻³); Turkey red oil (1.060 g·cm⁻³) and turpentine (0.840 g·cm⁻³). They were obtained from Guangzhou Changsheng Chemical Co.

Methods

Impregnation experiments

The specimens were placed into a vacuum-pressure impregnating tank and with a pumping vacuum, and the vacuum was maintained for 30 min at 0.098-0.090 MPa. Then, vegetable oil was pumped into the tank through a thin tube, and the pressure was maintained for a period of time. After impregnation, the excess vegetable oil on the wood surface was wiped off. Finally, the samples were placed in a ventilated place for 3-4 days, after which they were conditioned at 80°C for 24 h, 95°C for 24 h, and 103°C until reaching a constant weight (Wang et al. 2014a). The formula of weight percentage gain (WPG) is as follows (Emmerich et al. 2019):

$$\text{WPG} = \frac{M - M_0}{M_0} \times 100 \quad (\%) \quad (1)$$

where: M_0 is the mass of untreated wood (g) and M is the mass of treated wood (g).

The design parameters of orthogonal test $L_9 (3^3)$ are shown in Tab. 1. The properties of impregnated woods were investigated by the impregnation pressure (A), time (B) and temperature (C).

Tab. 1: Orthogonal experiment of castor oil treating wood.

Group	A. Impregnation pressure (MPa)	B. Impregnation time (h)	C. Impregnation temperature (°C)
1	1.4	1	25
2	1.4	1.5	55
3	1.4	2	85
4	1.6	1	55
5	1.6	1.5	85
6	1.6	2	25
7	1.8	1	85
8	1.8	1.5	25
9	1.8	2	55

Physical and mechanical properties

The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined by an universal testing machine (UTM-5000) according to Chinese national standard GB/T 17657-2013 and GB/T1936.1: 2009. Five replicates (20 mm × 20 mm × 300 mm) were prepared for each measurement.

Water uptake rate (WUR) and dimensional stability

Prior to testing, the samples were oven-dried, and their weight and dimensions were measured. The samples (20 mm × 20 mm × 20 mm) with 5 duplicate samples were completely immersed in the water for 5 cm and soaked in normal temperature water for 24 h and boiling water for 2 h, respectively. After immersion, the weight and dimensions of the samples were gauged. Then, the samples incubated at 80°C for 4 h and until reaching a constant weight at 103°C, and the mass and dimensions were recorded again. The tests were according to the Chinese national standards GB/T 1934-2009 and GB/T 1932-2009. The water uptake rate

(WUR), water repellency efficiency (WRE), anti-swelling efficiency (ASE) and anti-shrinkage efficiency (ASE') were calculated according to:

$$WUR = \frac{M_i - M}{M_i} \times 100 \quad (\%) \quad (2)$$

$$WRE = \frac{WUR_c - WUR_T}{WUR_c} \times 100 \quad (\%) \quad (3)$$

$$VSR = \frac{V_i - V_a}{V_0} \times 100 \quad (\%) \quad (4)$$

$$ASE = \frac{VSR_0 - VSR_i}{VSR_0} \times 100 \quad (\%) \quad (5)$$

$$ASR' = \frac{V_i - V_a}{V_c} \times 100 \quad (\%) \quad (6)$$

$$ASE' = \frac{VSR'_i - VSR'_a}{VSR'_0} \times 100 \quad (\%) \quad (7)$$

where: M_i is the mass of the samples after soaking (g), WUR_c and WUR_T are the WUR of control and treated wood (g). V_0 , V_i and V_a are wood samples in over-drying, volume after soaking, over-drying after wetting, respectively (cm^3). VSR_0 and VSR'_0 is VSR of control (%). VSR_i and VSR'_i are the VSR of treating samples (%).

Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FT-IR)

Scanning electron microscopy (Phenom XL) was used to observe the microstructure changes of the specimens before and after impregnation, with an acceleration voltage of 5 kV. Wood samples were cut along with three anatomical directions (transversal, radial and tangential).

A Fourier infrared spectrometer (XDS Master Lab) was applied to analyze the chemical structure of each specimen before and after treatment. The specimens were sawn into wood chips and crushed into wood powder with a pulverizer (2000C), then filtered to 200 mesh. Before the test, the samples were dried at 80°C and tested by the KBr pellet method (spectrum range $400\text{-}4000 \text{ cm}^{-1}$, resolution 4 cm^{-1} and scanned 16 times) (Jebrane et al. 2015).

RESULTS AND DISCUSSION

Preliminary test results of poplar modified by three vegetable oils

The physical and mechanical properties of poplar modified by three vegetable oils are shown in Tab. 2.

Tab. 2: Physical and mechanical properties of poplar treated by three vegetable oils (preliminary results).

Group	WPG (%)	MOR (MPa)	MOE (MPa)
The control	0	68.7	6866.8
Turkey red oil	11.7	84.1	6917.2
Castor oil	18.7	86.7	7476.3
Turpentine	54.6	75.8	7328.9

Notes: Impregnation pressure 1.4 MPa, time 1.5 h and temperature 25°C .

The highest WPG of 54.6% was observed when wood was impregnated by turpentine, which can be interpreted by the high penetration of the impregnating agent into the wood because of turpentine lowest molecular weight (Lyu et al. 2021). Specimens treated by castor oil showed the highest MOR and MOE at 86.7 MPa and 7476.3 MPa, respectively. The cost of turpentine is more than the other two oils in the Chinese market. In addition, a pungent smell can be detected in samples modified with turpentine, and its application is restricted (Chen et al. 2021). The lowest WPG was observed when the samples were treated by Turkey red oil. Furthermore, the MOR and MOE of samples by Turkey red oils samples (Chen et al. 2020) were lower than those of castor oil. To summary, the best impregnation influences were obtained by modifying the specimens with castor oil. Therefore, castor oil was utilized for the further orthogonal experiments.

Orthogonal test of poplar treated by castor oil

Weight percent gain (WPG) and mechanical property

Fig. 1 displays the WPG and density, MOR and MOE of the control and treated specimens. There was obviously some difference between WPG of all treatment specimens in Fig. 1a. The rang of WPG in all groups was 10.4% to 21.4% under different process conditions, respectively. The samples had different size holes. The density of the fourth group of specimens was 0.38 g cm^{-3} , which was the lowest. The lower the density of the specimen, the higher the porosity, so oil was embedded into the interior of the wood relatively easily. The castor oil in poplar is mainly distributed in the vessel, and the loss of castor oil during curing and drying is also an important reason (Li et al. 2020). By comparing different processes and WPG, it was found that the WPG of modified samples can be controlled.

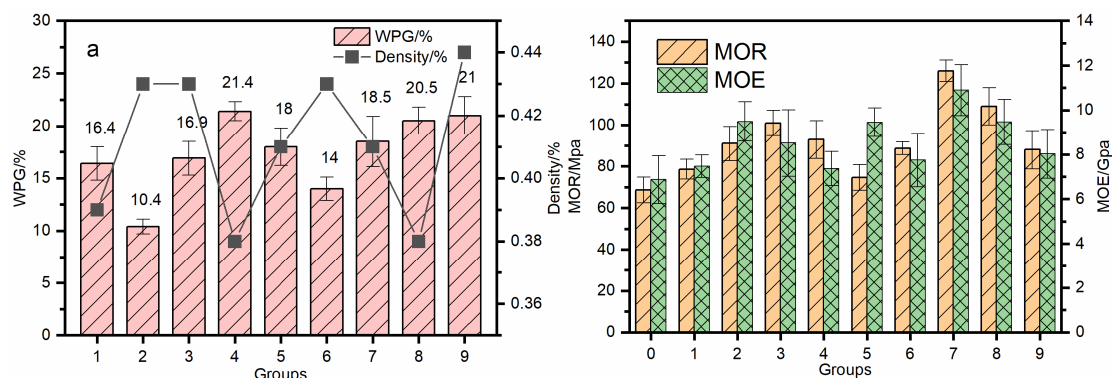


Fig. 1: a) WPG and density, b) MOR and MOE.

Fig. 1b illustrates that the MOR and MOE of all treated groups were higher than the control, which was similarly found in other researchers (Temiz et al. 2013). Compared with the control group, the MOR of the treated specimens were improved, and the increased rate of MOR was between 7.4% and 59.2%, with a maximum of 10.93 GPa. The increase rate of MOE was between 8.6% and 83.7%, with a maximum of 126.34 MPa. The MOR and MOE of treated samples were enhanced by 37.4% and 26.8%, respectively. The following factors can account for

this phenomenon. First, the modifier was filled into wood vessels and pores (Abolghasem Khazaeian et al. 2014). In addition, wood components could be cross-linked to it. The ability of wood to resist external pressure was improved. Besides, the internal tissue, parenchyma and cell walls of wood were covered with impregnating agent, which led to an increase in the wood density (Temiz et al. 2013, Zhang et al. 2020). Hence, physical and mechanical properties improved. In general, the MOR and MOE of wood can be improved by castor oil treatment.

Water uptake rate (WUR) and dimensional stability

Vegetable oil is endowed with excellent resistance to water. When oil is sprayed on the surface of a wooden utensil, it is oxidized and polymerized on the surface to form a protective film to prevent water uptake (Tang et al. 2019). The problem of hygroscopic expansion cannot be fundamentally solved after modification, but the amounts of hydrophilic groups and wettability in wood can be reduced to slow the wood water uptake, which further improves the dimensional stability of wood (Fredriksson et al. 2010, Ahmed et al. 2017).

The WUR and WRE, VSR and VSR' of untreated and castor oil treated specimens can be observed in Fig. 2. As shown in Fig. 2a, the WUR values of samples at normal temperature for 24 h were visibly below the control. Compared with the other groups, the WUR of the Group 3 specimens was the lowest at 67.8%. The WRE of the castor oil treated woods was between 11.1% and 36.8%, which distinctly indicated that the water repellency of untreated woods was improved.

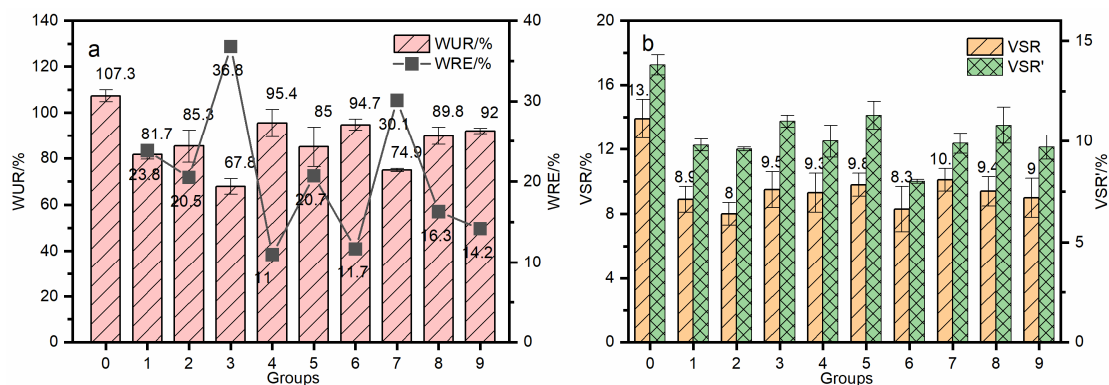


Fig. 2: Specimens in normal temperature water for 24 h: a) WUR and WRE, b) VSR and VSR'.

The VSR and VSR' of the control and treated specimen groups treated in normal temperature water for 24 h are shown in Fig. 2b. There are significant differences between VSR and VSR' in all groups, and the VSR and VSR' were lower than those of the control when the specimens soaked in room temperature. The minimum VSR (8.04%) was in group 2, and the minimum VSR' (7.95%) was in the group 7. which indicated distinctly improvement over untreated wood. It was indicated that the moisture absorption of wood can be effectively reduced at room temperature.

The WUR and WRE, VSR and VSR' of the untreated and treated specimens in boiling water for 2 h are presented in Fig. 3. Fig. 3a displays that the WUR values of samples in boiling water were universally higher than those of the control. Only the WRE of the third and ninth groups

were slightly higher than zero baseline, respectively 11.0% and 26.8%. It is clear that the water repellency of treated wood has not been effectively elevated. The VSR of treated wood exhibited similar values the control in Fig. 3b. However, the VSR' of samples was observably lower than that of the control. The VSR' of the modified specimens was improved by 39.05% to 51.90%. In summary, under high temperature and high humidity, the dimensional stability of the treated samples was slightly better than that of the untreated samples. These results implied that the treated specimens were not fittingly utilized under a high temperature and humidity environment.

The surface of the wood cell wall was covered with oil, which reduced the hygroscopicity and shrinkage of the wood (Chen et al. 2020). The improvement of vegetable oil waterproofing can be summarized by the following two points. First, the pit inside the wood was filled by a modifier, which obstructed the movement of water in the internal passage of the wood, reducing water uptake and wood size change (Chau et al. 2017, Dmitrenkov et al. 2021). Second, hydrophobic film can be formed by vegetable oil that can cross link with the internal hydroxyl groups of the wood, and the interaction between wood components and water was hindered (Wang et al. 2014).

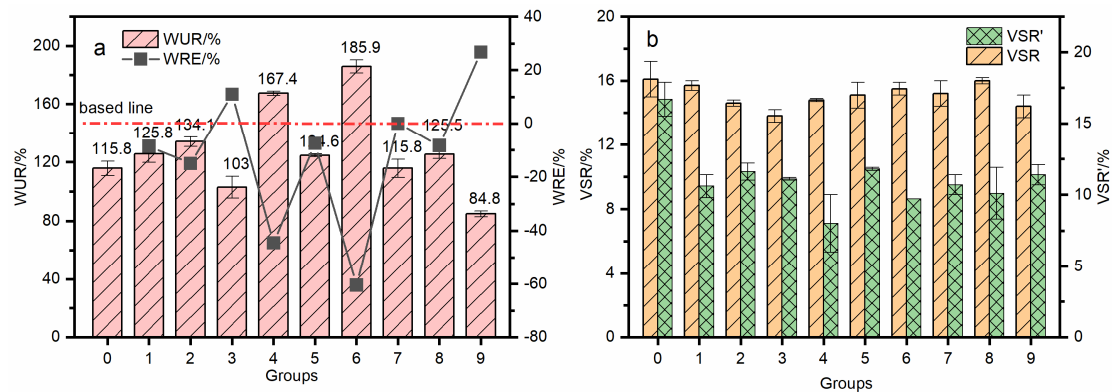


Fig. 3: Specimens in boiling water for 2 h: a) WUR and WRE, b) VSR and VSR'.

Scanning electron microscopy (SEM)

There was no significant difference between untreated wood and castor oil treated wood. The microscopic investigation of them is shown in Fig. 4. The cell lumens, pits, and ray cells were empty and visible in the untreated wood in Figs. 4a,b and c.

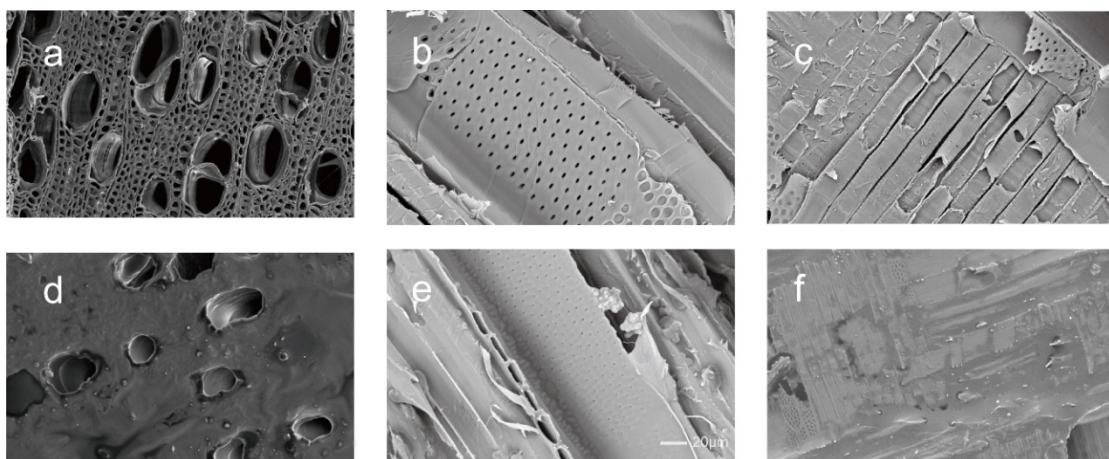


Fig. 4: SEM observations on cross section, tangential section and radial section of untreated wood (a, b and c), castor oil treated wood (d, e and f).

In contrast, as shown in Fig. 4d, the cell lumina are partially filled after impregnation with the castor oil, where there were a lot of polymers. Moreover, the pits and ray cells are covered or clogged by castor oil film Figs. 4e, f.

These results demonstrated that castor oil can be conveyed by intercellular space, pit, vessel. The excellent adhesion was shown in wood and castor oil. The interaction between wood and free water may be weakened by oil or oil film. Hence, the hydrophobicity of the wood or dimensional stability was enhanced.

Chemical structure analysis using FTIR

FTIR of untreated and treated wood are depicted in Fig. 5. The absorption intensity of peaks (3395 , 2916 , 1739 , 1652 , 1253 cm^{-1}) is significantly changed after castor oil treatment. Assignments of absorption IR spectra bands in wood are shown in Tab. 3.

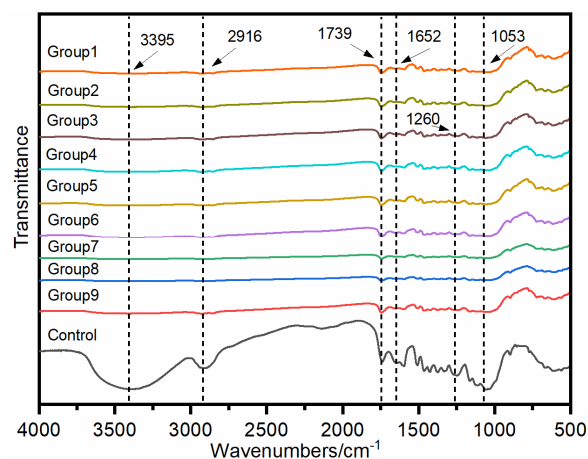


Fig. 5: FT-IR of the control and treated wood.

Tab. 3: Assignments of absorption IR spectra bands in wood.

Fr (cm^{-1})	Group and class	Assignments and remarks
1720 - 1740	C=O in unconjugated ketones, aldehydes and carboxy	C=O stretching
1645 - 1640	C=O in para-OH substituted aryl ketones, quinines	C=O stretching
1600	C=C in aromatic ring in lignin	Aromatic skeletal vibrations
1510	C=C in aromatic ring in lignin	Stretching vibrations of the aromatic rings present in lignin
1267	CO in lignin and hemicellulose	Guaiacyl ring breathing with CO-stretching
1162 - 1086	C-O-C in cellulose	Bridge oxygen stretching C-O-C asymmetric stretching vibration

The absorption peak at 3395 cm^{-1} is stretching vibrations of intermolecular associated hydroxyl groups (3500 - 3200 cm^{-1}) and the peak at 2916 cm^{-1} is C-H vibration (antisymmetric vibration peak of methylene), whose intensity decreased significantly because of oil treatments (Temiz et al. 2007). It confirmed that the number of hydroxyl group in the treated wood reduced obviously.

The absorption band (1710 to 1740 cm^{-1}) is characteristic of for non-conjugated carbonyl group, due to C=O stretch (xylan in hemicelluloses). The change in the band of it is due to the degradation of hemicelluloses (acetyl side chain breaks in hemicellulose) (Anderson et al. 1991, Fabiyi et al. 2008, 2011). Moreover, all castor oil treated groups were effectively found than untreated control. The result could indicate that oil molecules were contacted with hemicellulose, which led to the decrease of intensity.

Some researcher reported that the decrease of double bond at 1652 cm^{-1} because of the heated oil or curing heat treatment. The double bond can be reacted with unsaturated monomers by radical or cationic copolymerization (Larock et al. 2001, Li and Larock 2003).

The results of FTIR demonstrated that the castor oil was successfully impregnated into the interior of wood. Besides, the interaction between wood and water was reduced owing to castor oil, increasing the effectiveness of wood resistance to external deterioration factors, which are in accord with the results of Tab. 2.

Range and significance analysis

Because ASE and ASE' were measured the ability of wood with anti-swelling and anti-shrinkage efficiency. The value of ASE and ASE' is higher, the properties of wood were better. Thence, WPG, MOR, MOE, ASE and ASE' were applied to range and significance analysis (Li et al. 2020).

Moreover, range analysis was utilized to elaborate impregnation pressure, time and temperature on the physical and mechanical properties of castor oil treated wood (Tab. 4).

Tab. 4: Range analysis and variance analysis of castor oil treated wood.

Indicators		A. Pressure (MPa)	B. Time (h)	C. Temperature ($^{\circ}\text{C}$)
WPG (%)		5.4* (0.96)	2.5 (0.2)	0.8 (0.02)
MOR (MPa)		22.5 (0.94)	7.6 (0.12)	10.1 (0.2)
MOE (GPa)		1.3 (1.33)	1.4 (1.42)	1.4 (1.83 ^d)
Temperature water	ASE (%)	5.0 (1.28)	3.6 (0.7)	7.4 (3.39 ^c)
	ASE' (%)	2.7 (0.08)	7.3 (0.46)	8.7 (0.72)
Boiling water	ASE (%)	3.1 (4.63 ^b)	4.1 (9.3 ^a)	7.0 (24.72 ^a)
	ASE' (%)	7.6 (0.71)	8.4 (0.86)	6.4 (0.54)
$F_{0.01}(3,12) = 5.59$, $F_{0.05}(3,12) = 3.49$, $F_{0.10}(3,12) = 2.61$, $F_{0.25}(3,12) = 1.56$				

Notes: * values is R; value in parenthesis is F, ^{a-d} represents significant difference at 0.01, 0.05, 0.10 and 0.25 level, respectively.

The range value (R) is the difference between the maximal and minimal average values of a property among three levels of a certain factor. The higher R-value, the more important factor (Wang et al. 2014c). Overall, the R value of pressure on WPG and MOR was largest, followed by temperature and time, which illustrated the importance of impregnation pressure for castor oil treated wood. Whereas the ASE and ASE' of samples in temperature water and ASE in boiling water, the impregnation temperature is the most important factor.

The results of variance analysis expatiate the influence of three factors (pressure, time and temperature) on wood properties in Tab. 4. For the WPG and MOR of samples, impregnation

pressure is a significant element, which is consistent with the results of range analysis. The highly significant ($P < 0.01$) was found in ASE in temperature water, for the effect of impregnation time and temperature, which also has great effect on MOE, ASE and ASE' in temperature water. In general, impregnation pressure and temperature had more important influence on castor oil wood than time in this study. Finally, the optimum factor and level might be pressure of 1.8 MPa, time of 1.0h, temperature of 85°C.

CONCLUSION

(1) Through pre-test, the MOR and MOE of castor oil treated wood were higher than turpentine and Turkey red oils impregnated wood. The castor oil was utilized for the orthogonal experiments. (2) The results of SEM confirmed that castor oil can penetrate the wood through cell lumens, pits, and ray cells. (3) FTIR results showed that castor oil treatment decreased the intensity of hydroxyl and hemicellulose peaks (3395cm^{-1} and $1710\text{-}1740\text{ cm}^{-1}$), and the absorption changes at 2916 cm^{-1} and 1652 cm^{-1} . (4) Mechanical properties and dimensional stability of poplar were increased after treatment. However, the castor oil treated woods were not fittingly utilized under a high temperature and humidity environment by the results of VSR and VSR' in boiling water. An optimum process of castor oil treated poplar was obtained in this research based on orthogonal experiments and range analysis. Impregnation pressure and temperature influenced the wood properties more significant than time. Finally, the recommended combination of castor oil treated poplar was impregnation pressure 1.8 MPa, time 1.0 h and temperature 85°C.

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