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INFLUENCE OF SILICONE OIL THERMAL MODIFICATION ON MECHANICAL PROPERTIES OF MASSON PINE WOOD

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ABSTRACT

In this study, the effect of silicone oil thermal modification at different treatment temperatures (150 ºC, 180 ºC and 210 ºC for 2 h and 4 h.) on the mechanical properties of Masson pine (*Pinus massoniana* L.) wood was investigated. The density, modulus of elasticity (MOE), modulus of rupture (MOR), impact bending, compressive strength, and hardness of silicone oil thermal treated samples were evaluated and compared with those of untreated samples. Results showed the mechanical properties of Masson pine wood reduces after silicone oil thermal modification. The higher the modification temperature, the lower the mechanical properties of Mason pine wood. At 210 ºC for 4 h, mechanical properties of the modified samples were two times lower than the mechanical properties of the untreated. Higher modification temperature and longer treatment time contributed to lower mechanical properties.

Keywords: Hardness, Masson pine, mechanical properties, silicone oil, thermal modification.

INTRODUCTION

Wood is one of the most regularly used materials in several engineering and structural applications because of its distinctive properties with its capacity to be easily shaped and fastened with adhesives, low thermal extension, good shock resistance and sufficiently high mechanical strength (Bal and Bektaş 2013, Bekhta and Niemz 2003, Tomak *et al*. 2011). Due to the desirable features of wood as a renewable raw material, its environmental adaptability, and excellent mechanical properties, it is one of the construction materials widely used globally for decoration, furniture, and other applications because of its extraordinary range of attractive properties (Bekhta and Niemz 2003). Despite the excellent performance of wood, it has some detrimental characteristics such as hygroscopicity, anisotropic, dimensional instability, and poor decay resistance which limit its usage.

According to Bal and Bektas (2013), heat treatment affects some properties of wood, and the nature and extent of these effects are determined by many factors. Previous investigations reported that wood exposed to high temperatures increases the dimensional stability (Giebeler 1983), inevitably reduces strength and

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toughness and modify the chemical components of wood with severity of thermal modification and the wood species used (Esteves and Pereira 2009, Okon *et al*. 2017). Degradation of hemicelluloses, which bond cellulose and lignin in the cell wall, reduces the strength properties of modified wood. Reduction in toughness, hardness, bending, compression, and tension strength as a result of heat treatment have been reported in several research works (Vernois 2001, Yildiz *et al*. 2006).

In recent years, increasing environmental concern has led to increased use of thermally modified wood in engineering and structural applications. Thermal modification of wood is regarded as an environmentally friendly method of wood modification presently adopted globally, though a high-energy input is needed for the process (Navi and Sandberg 2012) compared to chemical modification with additional wood preservative chemicals. Factors such as treatment time and temperature, treatment condition, open or closed system have given rise to different thermal modification processes. Temperature is the most important parameter in wood modification, it varies between 180 ºC and 280 ºC depending on the processing conditions, utilization of the end product, wood species, sample size and moisture content of the wood (Militz 2002, Okon *et al.* 2018). Earlier studies reported that mass loss of wood due to thermodegradation reaction is a good indicator of the effectiveness of the treatment intensity (Welbacher 2007).

Thermal modification can be conducted using different treating media (e.g air, nitrogen, water and oil) and different treatment medium has it effect on the properties of the treated wood. Modification of wood with silicone oil as the heating medium has been reported to be a sustainable, environmentally friendly and cost-effective approach to wood modification (Okon *et al*. 2017), since no toxic chemical is involved (Okon *et al*. 2018), its boiling point like other vegetable oils (linseed, soybean, rapeseed etc.) is higher than 260 ºC (Gunstone 2011) and the oil itself is readily available in the market for purchase at low cost. The specific gravity of silicone oil in comparison to water (1,00) is lower (0,97), this enables it to float in water like other vegetables oils. Silicone oil has a high viscosity (1,000 - 5,000 cs), when dispersed the small droplet generated has the capacity to recoalesce back into a large bubble (Barca *et al*. 2014).

Generally, Oil heat treatment (OHT) enhances effective and even transfer of heat in wood and has been put to use in Europe (Sidorova 2008). Oil do isolate oxygen from the wood during heat treatment thereby preventing oxidation. It is imperative to modify wood using silicone oil as the heating medium to improve its quality for multi-purpose application in the wood industries (Okon *et al.* 2018).

Few studies have explored the use of silicone oil to improve wood physical properties (Okon *et al*. 2017, Okon *et al*. 2018), but no study has used silicone oil thermal modification to investigate the mechanical properties of wood. This study aimed at investigating the effect of silicone oil thermal modification at different treatment temperature and time on mechanical properties such as modulus of elasticity (MOE), modulus of rupture (MOR), impact bending and compressive strength as well as density of Masson pine wood.

MATERIALS AND METHODS

Masson pine (*Pinus massoniana* Lamb.) wood was obtained from the People's Republic of China. Samples with dimension 20 mm x 20 mm x 300 mm (tangential x radial x longitudinal) along the grain direction were prepared. Samples were condition in a relative humidity chamber at 65 % ± 3 % and a temperature of 20 ºC ± 2 °C to obtain 12 % moisture content. Silicone oil was purchased from Chemicals Regent Company Limited, Beijing, People's Republic of China.

Silicone oil thermal modification

After conditioning in the humidifier, the prepared samples were divided into two groups: unmodified (untreated) and silicone oil thermally modified (150 °C, 180 °C and 210 °C). Silicone oil thermal modification was performed in an oil bath with samples immersed into the heated oil at temperature (150 °C, 180 °C and 210 °C) at modification time of 2 h and 8 h. The modified samples were removed from the oil bath after the target temperature and time has elapsed. The specific gravity and viscosity of silicone oil used in this study were 0,97 and 2500 cs.

Wood density

The density of the samples was determined by measuring the mass (M) and volume (V) (i.e length, width and thickness) of the untreated and silicone oil thermally modified samples using Equation 1.

$$\delta\left(\frac{g}{cm^3}\right)=\frac{M}{V} \quad (1)$$

Where is the oven - dry density; M is oven - dry mass of each sample and V is Oven - dry volume of sample.

Mechanical properties

Modulus of elasticity (MOE) and modulus of rupture (MOR) was determined according to GB/T 1936.1 and GB/T 1936.2 (2009). Compression strength parallel to grain and impact bending were determined according to GB/T 1935 and GB/T 1940 (2009). Brinell hardness was performed on the tangential and radial direction using a universal testing machine by driving a steel ball with a diameter of 11, 2 mm into test the samples (GB/T 1941 2009).

Statistical analysis

Density and mechanical properties data were analyzed using analysis of variance (ANOVA) to test each treatment for significant differences and mean separation was carried out using Dunnett test to compare each treatment with the untreated. They were considered to be significantly different when the $P < 0,05$ and all analyses were performed using R version 3.1.1 statistical software (R Development Core Team 2014).

RESULTS AND DISCUSSION

Figure 1 shows the wood density of untreated and silicone oil thermal modified Masson pine wood. The results show that the treatment temperature had an effect on the density of the wood. The density of the modified wood increased compared to the untreated. The mean density of the wood was 0,55 g/cm^3 for the untreated and varied between 0,78 g/cm^3 to 0,69 g/cm^3 for a treatment time of 2 h and 0,76 g/cm^3 to 0,66 g/cm^3 for a treatment time of 8 h between treatment temperature (150 ºC to 210 ºC) of the modified wood and was significantly different compared with the untreated ($F = 20,39$, $p < 0,001$). Silicone oil thermal modification increased the density of the wood to levels higher than that of the untreated (Figure 1). The highest mean density value was obtained in the samples treated for 2 h at 150 ºC and the lowest was found in samples treated for 8 h at 210 ºC. However, the wood density gradually reduced with an increase in treatment temperature. Similar results were reported by (Boonstra 2008, Esteves *et al*. 2008, Esteves and Pereira 2009) in their study, they asserted that changes in the chemical composition/structure, reduce hygroscopic and equilibrium moisture content and evaporation of extractives were the possible causes of the decrease in density of thermally modified wood. According to Dubey (2010) wood mass loss at a high temperature is the cause of a slight decrease in the density of the wood. Mass loss of wood is one of the key indicators of the degree of thermal modification and is directly associated with the decrease in density of wood after silicone oil thermal modification.

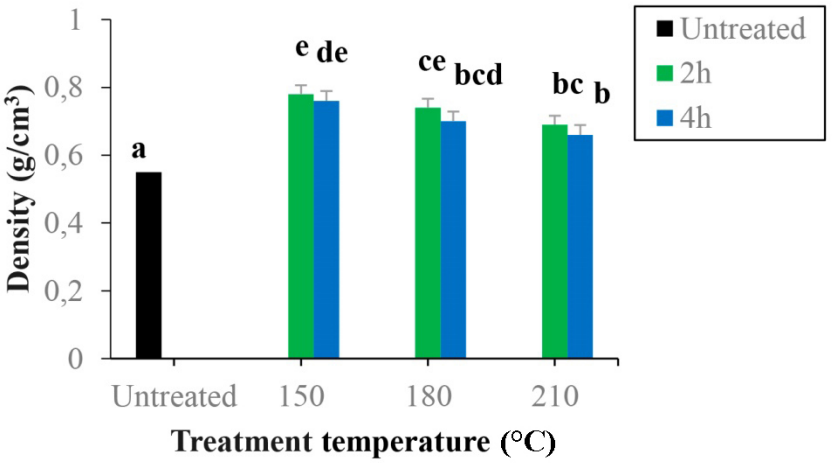

Figure 1: Density of untreated and silicone oil thermal modified Masson pine wood.

Table 1 shows the effect of silicone oil thermal modification on the mechanical properties of Masson pine wood. The mean mechanical properties values of the untreated samples 12088,02 MPa for MOE, 101,90 MPa for MOR, 5,59 kJ·m^{-2} for impact bending and, 55,46 MPa for compression strength were higher than that of the silicone oil modified samples.

The mean MOE of silicone oil modified samples ranged between 11553,18 MPa for 2 h and 10596,94 for 8 h at 150 ºC and 7869,62 MPa for 2 h and 6693,89 MPa for 8 h at 210 ºC (Table 1). There were no significant differences (F = 1,201 p = 0,318) in MOE among modified samples at different temperature and time. This is because MOE does not significantly change as much as other mechanical properties like MOR. It was observed that the MOE of silicone oil thermally modified Masson pine generally decreased with increase in treatment temperature. This is an indication that silicone oil modification applied in this study affects the MOE values of the samples. It should be noted that the degradation of chemical components of wood (hemicellulose) at high thermal temperature has an effect on the mechanical properties of the wood and evaporation of extractive at high thermal temperature caused mass loss as well.

The mean MOR of silicone oil modified samples ranged between 86,59 MPa for 2 h and 77,17 MPa for 8 h at 150 ºC and 54,12 MPa for 2 h and 37,85 MPa for 8 h at 210 ºC (F = 27,11, p = 1,0200). There were significant differences among silicone oil modified samples at different treatment temperature and time (Table 1). Silicone oil thermal modification decreased the MOR of Masson pine wood. This affirms the reports of Hofmann *et al.* (2013), Lahtela and Kärki (2016) that thermal modification invariably decreased MOR values of pine, beech and ash wood with increasing temperature. Reduction in MOR of thermally modified wood was attributed to the degradation of hemicelluloses (Boonstra *et al*. 2007), as degradation of the chemical structures of thermally modified wood has a resultant effect on the mechanical properties of the wood. Earlier works have reported increase brittleness and decrease bending strength of thermally modified wood (Boonstra *et al.* 2007, Kocaefe *et al*. 2008, Korkut *et al*. 2008).

There were significant differences in impact bending among silicone oil modified samples at different treatment temperature and time (F = 6,301 p = 3,1505) compared to the untreated. At 150 ºC, the mean impact bending value ranged between 7,33 kJ·m^{-2} for 2 h to 6,36 kJ·m^{-2} for 8 h and at 210 ºC, the mean value ranged between 4,50 kJ·m^{-2} for 2 h to 3,14 kJ·m^{-2} for 8 h respectively (Table 1). It was observed in this study that the impact bending of silicone oil thermally treated samples slightly increased at 150 ºC for 2 h and 8 h. This could be as a result of net oil uptake at low temperature (Dubey 2010, Okon *et al.* 2018) and increased crosslinking of lignin polymer during the oil thermal treatment, before gradually decreasing at 180 ºC and 210 ºC. At 210 ºC for 8 h, the highest decrease in impact bending values were observed. The crystallization of amorphous cellulose may be the major factor for the loss of mechanical strength, affecting especially bending and tensile strength when treatment temperature is higher (Zhao *et al.* 2015).

The compression strength at different treatment temperature and time varied significantly among treatments (F= 7,607 p = 3,67006). At 150 ºC, the mean compression strength value ranged between 50,38 MPa for 2 h to 46,81 MPa for 8 h and at 210 ºC, the mean value ranged between 33,98 MPa for 2 h to 31,47 MPa for 8 h respectively (Table 1). Compression strength of silicone oil thermally modified was generally decreased. Previous works also reported a decrease in compression strength of some thermally modified wood particularly at high temperature (Korkut and Aytin 2015, Percin and Altunok 2017, Pelit *et al.* 2018). Pelit *et al.* (2018) reported that compression strength values of thermally modified fir, linden and poplar decreased by 12 %, 13 %, and 5 % respectively compared to untreated samples. Rupture of bonds between molecules of materials, mass loss and degradation of hemicellulose are some of the causes of the decrease in strength of thermally modified wood (Yildiz *et al*. 2006).

Table 1: Average mechanical properties of untreated and silicone oil thermally modified Masson pine wood.

Treatment temperature (ºC)	Time (h)	MOE (MPa)	MOR (MPa)	Impact bending ($kJ \cdot m^{-2}$)	Compression strength // to grain (MPa)
Untreated		12088,01a ± 2906,53	101,9e ± 13,34	5,59bc ± 1,78	55,46c ± 5,14
150	2	11553,18a ± 4024,07	86,59de ± 11,38	7,33c ± 2,13	50,38c ± 14,52
	8	10596,94a ± 1154,24	77,17cd ± 8,91	6,36bc ± 1,21	46,81bc ± 5,29
180	2	9674,71a ± 2663,54	79,26cd ± 16,52	5,51bc ± 1,85	45,92bc ± 10,74
	8	8415,57a ± 2561,11	60,35bc ± 14,89	4,86ab ± 2,18	43,58ac ± 3,92
210	2	7869,62a ± 725,77	54,12ab ± 14,34	4,50ab ± 1,61	33,98ab ± 16,49
	8	6693,89a ± 719,74	37,85a ± 10,03	3,14a ± 0,56	31,47a ± 3,01

The Mean values in percentage ± standard deviation was determined in each treatment.
Subscript within each column, followed by different letters of the alphabets are significantly different from the untreated at $p < 0,05$.

There were significant differences in silicone oil modified samples at different treatments temperature and time in the tangential direction (F = 28,39 p = 3,6400) and radial direction (F = 22,85 p = 4,0600). The mean hardness values in the tangential and radial directions were highest in the untreated while the lowest tangential and radial hardness values were obtained in the silicone oil thermally modified samples at 210 ºC for 2 h and 8 h respectively (Figure 2). An earlier study had revealed that hardness of wood is hinged on the density of the wood (Kollmann 1951). According to Pelit and Yorulmaz (2019) decrease in hardness may be caused by density losses and thermal decomposition in the wood samples as a result of thermal treatment, particularly at high temperature. In this study, silicone oil thermal modification reduced density and this may be the cause of the decrease in hardness after thermal modification. Furthermore, previous work asserted the causes of a decrease in hardness of wood to include: rupture deformation and fragmentation in the cell wall of wood at high temperature (Budakçi *et al*. 2016). Contrarily, earlier work reported that hardness of thermally modified wood increased at short treatment durations particularly at high temperatures, while on the other hand, longer treatment durations decreased the hardness of thermally modified wood (Sundqvist *et al*. 2006). High silicone oil modification temperature as applied in this study deed decrease hardness of the wood. This is in line with the report of a previous study (Scheiding *et al*. 2005).

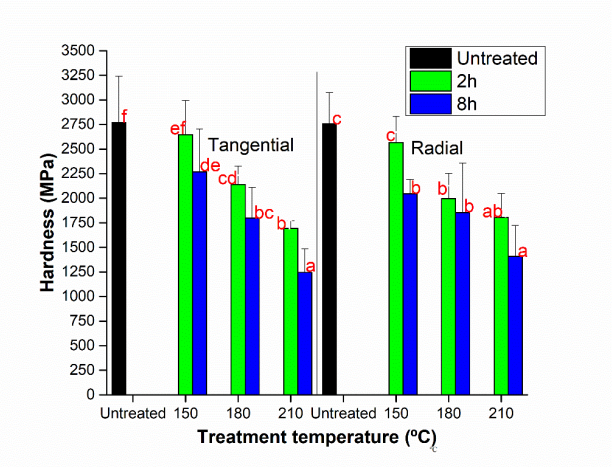

Figure 2: Shows Brinell hardness of untreated and silicone oil thermally modified Masson pine wood.

CONCLUSIONS

This study has demonstrated that silicone oil thermal modification had significant impact on the physical and mechanical properties of Masson pine wood. Modification temperature and time had important influence on the modulus of elasticity (MOE), modulus of rupture (MOR), impact bending and compressive strength and density resulting to the reduction in the mechanical properties of the treated wood. Significant mechanical properties reduction was observed in wood samples modified at temperature of 210 ºC for 8 h. Higher the modification temperature, the lower the mechanical properties of the wood.

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