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EVALUATION OF SOME TECHNOLOGICAL PROPERTIES OF CHINESE PARASOL WOOD AFTER ECO-FRIENDLY SILICONE OIL HEAT TREATMENT

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Abstract:
This study evaluated the effect of eco-friendly silicone oil heat treatment temperatures (150°C, 180°C and 210°C) on the changes in the mechanical properties and color of Chinese parasol (Firmiana simplex L.) wood in comparison with the control wood. The hardness, modulus of rupture (MOR), modulus of elasticity (MOE), impact bending, compression strength and colour of the wood were investigated. Silicone oil thermal modification reduces the mechanical of Chinese parasol wood, and the reduction surges with increasing modification temperature. The color of silicone oil thermally modified wood show decrease in lightness (ΔL^*) *and increase in total color change* (ΔE^*)*, indicating that silicone oil was effective in the modification of Chinese parasol wood.*

Key words: *chinese parasol; color change; oil heat treatment; silicone oil; technological properties.*

INTRODUCTION

Wood is a natural, sustainable bio-material with good features and superior formability which qualifies it to be used as an engineering and construction material in both exterior and interior decoration work. Over the years, wood has been favoured as the best material for building and furniture making. This is because of its sterling unique properties such as balanced strength to mass ratio, lightweight, heat insulation ability, easy processability and better seismic performance compared other materials. Other green advantages of wood include: its renewability, sustainability, generate fewer greenhouse gases and provides a long-term repository for atmospheric carbon (Otto Rapp *et al.* 2007, Morrell 2008). However, despite its excellent advantages stated above, the use of untreated wood for structural application suffer some setback due to prolonged exposure to certain environmental conditions like biotic factors (fungi, bacteria, insects), climatic abiotic factors (temperature, humidity, rainfall, UV exposure and wind forces) and edaphic abiotic factors (moisture content, mineral/nutrient composition, pH level, aeration) (Marais *et al*. 2020), therefore shortening the service life of the wood.

Wood modification is the only way to make wood last longer in the environment. Previously, wood modification industry had depended on toxic wood preservation methods to modify the wood to make it effective against degradation for many years using various formulations and combination of chromium copper and arsenic, borates, pentachlorophenol in various formulations and creosote (Rowell 2020). Presently, a countless number of these wood preservatives have either been banned in many countries or only permit restricted use. This has led to another wood modification approach not based on toxicity.

Thermal modification of wood is an effective method to modify wood against some environmental and biological factors as well as improving the properties of wood and it is considered widely from different perspectives by researchers and wood industries (Vernois 2001, Hill 2007, Tjeerdsma *et al.* 1998, Sandberg *et al*. 2013, Militz 2015, Umar *et al.* 2016, Wentzel *et al.* 2019). For effective modification of the chemical, mechanical and physical properties of wood, thermal treatment is performed at different level of temperatures (150°C to 260°C) under different treatment conditions (steam, vacuum, nitrogen, oil) in oxygen-free atmosphere (open or closed systems) (Hill 2007). This is because, at treatment temperature below 150°C, no significant modification can occur in the wood components while on the other hand, at elevated treatment temperature above 300°C, the wood constituents are severely degraded. In addition to treatment

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temperature and time, other factors that influence the properties of thermally modified wood are the moisture content of the wood, the pressure conditions and the heat transfer media (Militz and Altgen 2014).

Different wood modification methods have been established particularly in Europe (Esteves and Pereira 2009, Militz and Altgen 2014). But this study is particularly interested in Oil Heat Treatment (OHT) method using silicone oil as a heating medium to improve heat flow into the wood. The advantages of silicone oil as heating medium in the thermal treatment of wood include the following: oil limits excessive oxygen from the wood during the treatment process, the boiling point of silicone oil is higher than the temperature required for the thermal treatment thus making it a suitable heating medium and it is non-toxic and environmentally friendly in composition.

In this study, the silicone oil thermal modification of Chinese parasol (*Firmiana simplex* L.) was carried out at different temperatures (150^{o}C, 180^{o}C and 210^{o}C) and time (2h and 4h). The mechanical properties (hardness, modulus of rupture-MOR, modulus of elasticity-MOE, impact bending, compression strength) and color were examined using a Universal Testing Machine and X-rite spectrophotometer.

MATERIALS AND METHODS

Chinese parasol (*Firmiana simplex* L.) is predominantly used in the manufacture of different thermally modified structural components like cladding, decking and doors, therefore it was of spectacular interest in this study. Sawn wood of the species was subjected to silicone oil thermal modification at a temperature of 150^{o}C, 180^{o}C and 210^{o}C for 2h and 4h. The modification process was performed in a laboratory-scale treatment oil bath based on modified Oil Heat Treatment (OHT) process, (Menz Holz) Germany (Hill 2007, Esteves and Pereira 2009). The samples were submerged in heated silicone oil according to the stated temperature above. Silicone oil served as a heat transfer medium shielding the wood from oxygen during the treatment process, thus preventing oxidation process. For each of the tested properties, ten samples were used for each treatment temperature and time. After the treatments, the specimens were then placed in conditioning chamber at temperature of 20ºC and 65% relative humidity.

Mechanical properties

Brinell hardness testing was performed using a Shimadzu universal testing machine with a 10kN load cell on the tangential, radial and longitudinal surfaces according to ISO 6506-1 (2014), while the dimensions of the samples measured were 50mmx50mmx70mm (tangential x radial x longitudinal), and ten samples were used for this experiment. A 10mm diameter indenter (tungsten carbide composite ball) at the end of the load application arm was forced into the surface of the test samples. The indenter was driven into the samples in three steps. It was slowly increased during the first 15s, then it was maintained for 25s and finally, the applied force was decreased to 40s. The load was removed after 20s, and the diameter of the indentation left on the surface by the composite ball was measured. The Brinell hardness was computed using equation (1).

$$HB\ (N/mm^2) = \frac{2 . F}{\pi . D\ (D - \sqrt{D^2 - d^2}\ )} \quad (1)$$

where: F = the force applied (N), d = the diameter of the indentation left by the composite ball on the surface of the test sample (mm) and D = the diameter of the composite ball (mm)

The bending test was determined using a Shimadzu universal testing machine (Japan) with a 10kN load cell according to ISO 13061-4 (2014). Three-point bending tests were performed on samples with dimensions 20mmx20mmx300mm (Tangential x radial x longitudinal) (Fig. 1). Ten replicate samples were tested for each treatment temperature and time with dimensions. The loading speed in bending was 4mm/min and the test period was 3min. The load-deformation were recorded and data obtained were computed to determine the modulus of elasticity (MOE) and modulus of rupture (MOR), equation (2 and 3).

$$MOE\ (N/mm^2) = PL^3/4bh^3 f \quad (2)$$

$$MOR\ (N/mm^2) = 3P_{max}L/2bh^2 \quad (3)$$

where: P = the load difference in elasticity (N), L = the supporting span (mm), b = the width of the samples (mm), h = the thickness of the samples (mm), f = deflection at the mid-length below the proportion deflection limit (mm) and P_{max} = the maximum load when the sample is broken (N).

Impact bending of the samples was determined according to the ISO 13061-10 (2014). The impact bending tests of the samples were performed in the tangential direction. The dimensions of the impact bending test samples were 20mmx20mmx300mm (Tangential x radial x longitudinal) (Fig. 1). The tests were performed on a universal testing machine. The impact was computed using equation (4).

$$IB\ (kgm/cm^2) = \frac{\beta}{b * h} \tag{4}$$

where: β = the absorbing energy (kgm), *b* = the width of the sample (cm) and *h* = the thickness (cm).

Compressive strength (CS) parallel to the grain of the samples was determined according to the method described by ISO 13061-17 (2017) using a universal testing machine. The crosshead speed was 1.2 mm/min. The dimension of compressive strength test samples was 20mmx20mmx60mm (Tangential x radial x longitudinal). The CS value was computed using equation (5).

$$CS\ (N/mm^2) = \frac{P_{max}}{b * h} \tag{5}$$

where: P_{max} *= the maximum load applied to the samples (N), b* = the width of the samples (mm), *h* = the thickness of the samples (mm). Strength values were adjusted to 12% moisture content using equation (6).

$$\propto_{12} = \propto_m [1 + \propto (m - 12)] \tag{6}$$

where: $\propto_{12}$ = the strength at 12% moisture content (N/mm_2), $\propto_m$= the strength at moisture content deviated from 12% (N/mm_2), $\propto$ = the constant value indicating the relationship between strength and moisture content and *m* = the percentage moisture content in the test samples (%).

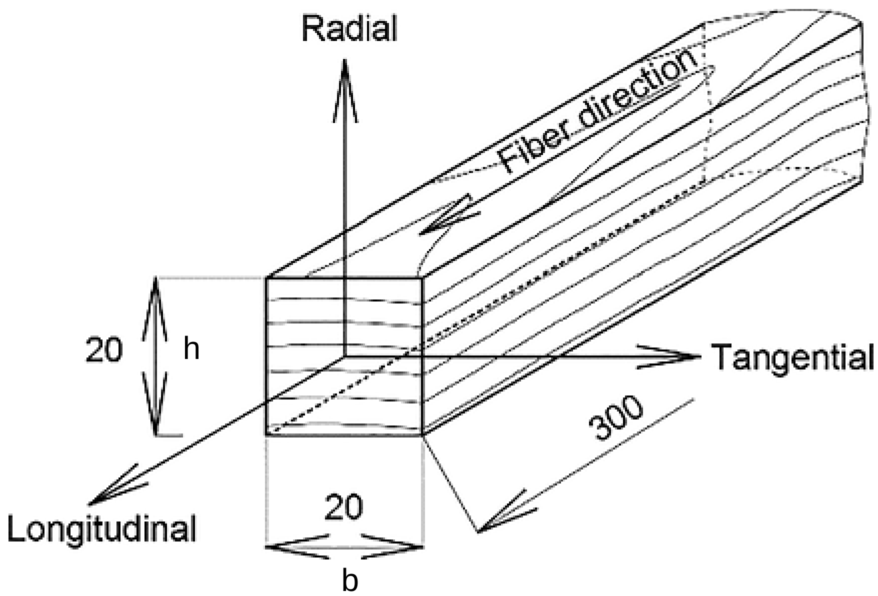

Fig. 1.
Shape and dimensions of the samples.

Surface properties

Color of the control and eco-friendly silicone oil treated samples were measured on the surface of the samples before and after treatment by using an X-rite spectrophotometer (Japan) (Fig. 2). Measurements were taken at the center position and each sample was measured five times, while a total of ten samples were measured for each treatment to obtain a mean value. With reference to ASTM D 2244-2 (2011), the CIELAB color system coordinate was characterized by three-dimensional parameters L*, a* and b*, which represent color coordinates of the individual wood sample before treatment. Where the L* axis denotes lightness and varies from 100 (white) to 0 (black), +a* denotes red, -a* denotes green, +b* denotes yellow and -b* denotes blue chromatic coordinates respectively (Fig. 3). Total color change (ΔE^*) was computed according to equation (7).

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{7}$$

where: ΔL^*, Δa^* and Δb^* are the differences in color before and after eco-friendly silicone oil treatment.

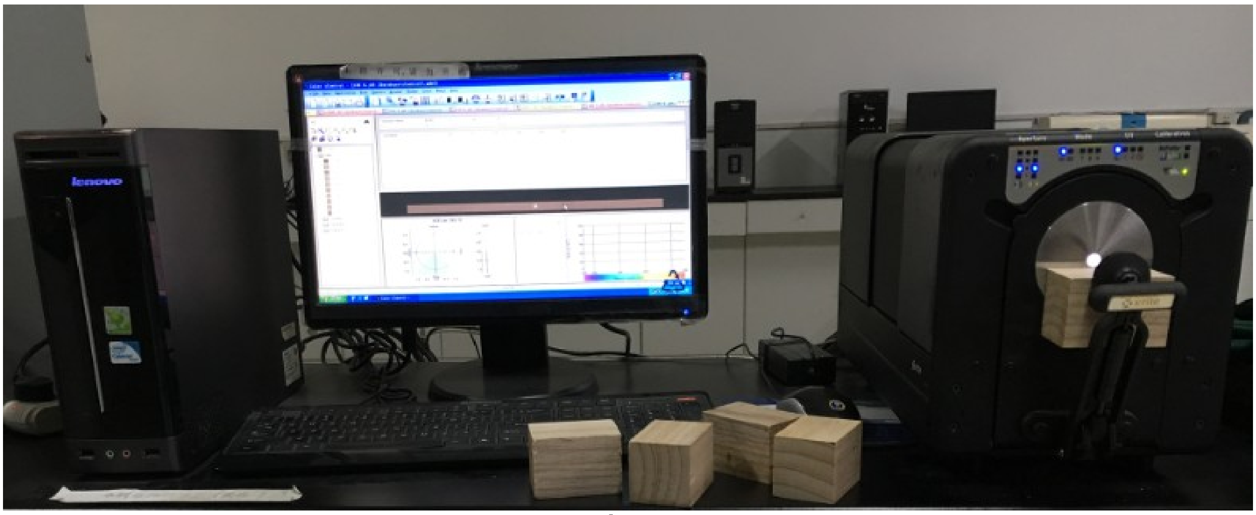

Fig. 2.
X-rite spectrophotometer used to measure color.

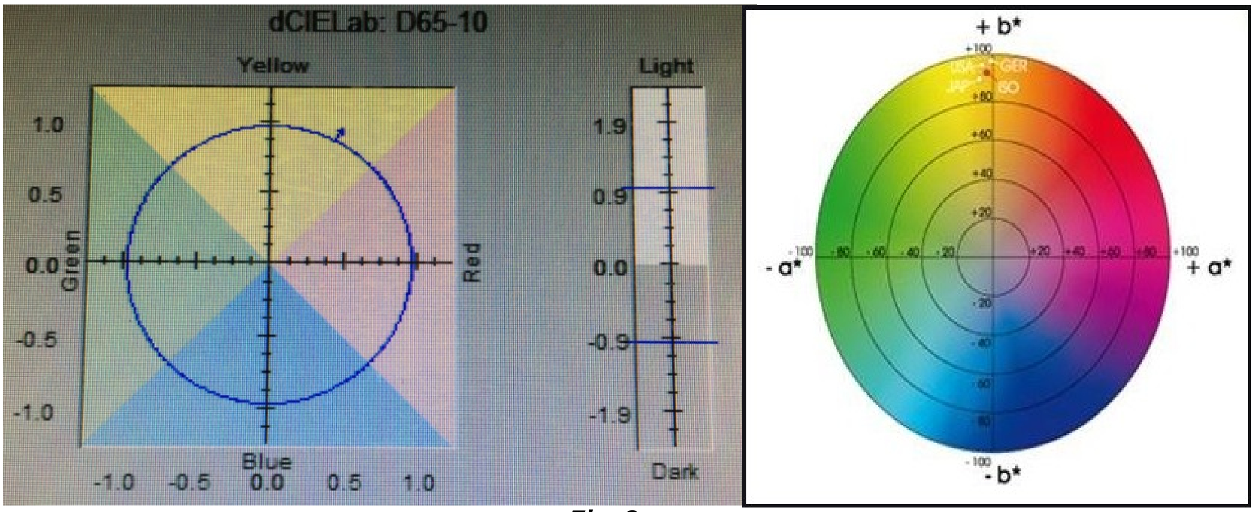

Fig. 3.
CIELab Chromaticity Color chart. L^* represent lightness, which varies from 0 (dark) to 100 (light), a^* varies from –a (green) to +a (red) and b^* varies from –b (blue) to +b (yellow).

Statistical analysis

Data of Brinell hardness, MOR, MOE, impact bending, compressive strength and color were analyzed using the R statistics version 3.3.1 (2014) and are presented as mean ± standard deviation. Data were checked for normality and homogeneity of variance using Newman-Keuls (SNK) and Benjamini-Hochbery. Means were tested for significant differences by one-way analysis of variance (ANOVA) followed by a Tukey post hoc test, at $p < 0.05$ significance level.

RESULTS AND DISCUSSION

The results of the hardness test showed that silicone oil heat treatment modified the wood samples (Table 1) and there were significant differences among treatments at different temperatures compared to the control (Table 2). Silicone oil thermal modification affected hardness of the wood causing a reduction in wood hardness along the tangential, radial and transverse surfaces with the least hardness (1682.26N/mm^2) reduction obtained along the transverse surface; when treated at 210^{o}C for 4h. The effect of the treatment was highest on the radial hardness, followed by tangential hardness and then transverse hardness. The mean radial hardness of the treated wood was 2095.69N/mm^2 compared to control (2265.18N/mm^2) when the wood was treated at 150^{o}C for 2h. Radial hardness (997.62N/mm^2) decreased significantly at a treatment temperature of 210^{o}C for 4h compared to the control (2265.18N/mm^2). Similar results were reported by earlier researchers (Korkut *et al.* 2008, Percin *et al.* 2016). According to Yildiz *et al*. (2006), the high decrease in hardness values was obtained when two different wood species were treated at 180ºC for 10h. On the other hand, other studies reported contrary results, either a decrease or an increase in hardness after thermal modification of wood, depending on the wood species and the heat treatment method adopted (Percin *et al.* 2016). Hardness is primarily influenced by density, the hardness of the surface layer, modification temperature, moisture content, measurement techniques and conditions (i.e load level and loading time) (Holmberg 2000, Gašparík *et al.* 2016, Cao *et al.* 2020). Furthermore, high temperature as well as long treatment time directly affects (reduction in) hardness of the wood, as a result of the degradation of hemicellulose and lignin (Fang *et al*. 2012, Salca and Hiziroglu 2014).

Table 1

Mean Brinell hardness after eco-friendly silicone oil heat treatment

Treatment	**Surface**	**Control**	**150^{o}C**		**180^{o}C**		**210^{o}C**	
Time (h)			2	4	2	4	2	4
Hardness (N/mm^2)	Tangential	2390.53^{e} (192.92)	2101.47^{de} (271.89)	2093.57^{de} (93.36)	1735.21^{bc} (98.33)	1889.06^{cd} (342.92)	1420.32^{b} (404.87)	961.66^{a} (140.52)
	Radial	2265.18^{d} (411.17)	2095.69^{cd} (295.95)	1990.15^{cd} (122.70)	1791.31^{bc} (149.39)	1640.16^{b} (224.76)	1125.93^{a} (206.85)	997.62^{a} (97.31)
	Transversal	3889.84^{d} (399.85)	3780.77^{d} (447.24)	3214.31^{c} (405.46)	3009.76^{bc} (397.49)	2513.27^{b} (333.28)	1911.41^{a} (364.70)	1682.69^{a} (367.98)

The values represent mean and numbers in bracket denote standard deviation of ten replicates. Means within a column, followed by the same superscript are not significantly different by Student-Newman-Keuls (SNK) and Benjamini-Hochbery tests at $p < 0.05$.

Table 2

Analysis of Variance of Brinell hardness after eco-friendly silicone oil heat treatment

	Factor	df	Sum of square	Mean square	F-value	Significant Level, p
Tangential hardness	Treatment	6	44624135	7437356	49.03	***
	Error	63	9556122	151684		
Radial hardness	Treatment	6	13949871	2324978	41.02	***
	Error	63	3571155	56685		
Transversal hardness	Treatment	6	13848892	2308149	37.51	***
	Error	63	3877087	61541		

***** - significant, P < 0.05**

The results of modulus of rupture (MOR), modulus of elasticity (MOE), impact bending and compression strength are shown in Table 3, highlighting the effect of eco-friendly silicone oil heat treatment on the mechanical properties of the wood. Table 4 shows there were significant differences in mechanical properties after eco-friendly silicone oil modification. All the evaluated mechanical properties generally decreased after silicone oil thermal modification (Table 3) with increasing treatment temperature and time. The least MOR, MOE, impact bending and compression strength were obtained in the samples treated at 210^{o}C for 4h (20.12N/mm^2, 6084.59N/mm^2, 2.26N/mm^2, 20.95N/mm^2). Similar results were reported previously by some researchers (Korkut and Hiziroglu 2009, Candelier *et al*. 2013, Kačíková *et al.* 2013, Esteves *et al.* 2014). This results indicated that silicone oil heat treatment at a high temperature significantly affected the mechanical properties of the wood due to degradation of cell wall constituents (hemicellulose) and loss of wood mass, thus making the wood to become brittle after treatment. The brittleness of wood fibers owing to the silicone oil thermal modification process leads to a reduction in the strength of wood.

Previous studies have reported a decrease in the MOR of some selected wood species after thermal modification (Teoh *et al*. 2011, Cao *et al.* 2020). It was reported that the MOR of European aspen and silver birch wood decreased after thermo-mechanical timber modification by 10% and 16%, respectively (Marttila *et al.* 2017). This may probably be related to the degradation of amorphous cellulose in the wood polysaccharides, as hemicelluloses are degraded at high temperature by the loss of acetyl groups that become acetic acid (Cademartori *et al*. 2013).

The influence of thermal treatment on MOE according to Mitchell (2007) is dependent on treatment temperature and reaction time, wood species, initial moisture content and surrounding atmosphere and the influence could be positive or negative (Gong *et al.* 2010, Fang *et al*. 2012). Furthermore, the decrease in the compression strength of silicone oil thermal treated Chinese parasol wood could be attributed to a decrease in the wood density with increasing thermal modification temperature which in turn reduces the mechanical properties. In conclusion, it was observed that temperature had a significant effect on the MOR, MOE, impact bending and compression strength due to carbonization of fibers, leading to the reduction in mechanical strength at high temperature.

Table 3

Mean mechanical properties after eco-friendly silicone oil heat treatment

Treatment	**Control**	**150°C**		**180°C**		**210°C**	
Time (h)		2	4	2	4	2	4
MOR (N/mm^2)	70.6^{d} (9.34)	62.16^{d} (16.22)	61.22^{d} (8.94)	43.85^{c} (6.87)	38.36^{bc} (14.29)	27.62^{ab} (4.52)	20.12^{a} (2.55)
MOE (N/mm^2)	8097.27^{b} (1487.38)	1091.14^{ab} (1091.73)	7681.24^{ab} (1325.85)	7134.39^{ab} (738.11)	6813.29^{ab} (1913.24)	6261.03^{a} (968.69)	6084.59^{a} (1412.43)
Impact bending (kgm/cm^2)	4.69^{f} (0.15)	4.44^{ef} (0.74)	3.92^{de} (0.77)	3.40^{cd} (0.43)	2.94^{bc} (0.28)	2.34^{ab} (0.25)	2.26^{a} (0.07)
Compression (N/mm^2)	28.90^{ab} (10.69)	45.74^{c} (4.90)	47.71^{c} (9.60)	32.60^{b} (5.75)	31.03^{ab} (2.941)	21.63^{a} (7.68)	20.95^{a} (7.76)

The values represent mean and numbers in bracket denote standard deviation of ten replicates. Means within a column, followed by the same superscript are not significantly different by Student-Newman-Keuls (SNK) and Benjamini-Hochbery tests at $p < 0.05$.

Table 4

Analysis of Variance of mechanical properties after eco-friendly silicone oil heat treatment

	Factor	df	Sum of square	Mean square	F-value	Significant level, p
Modulus of rupture (MOR)	Treatment	6	21424	3571	34.83	***
	Error	63	6459	103		
Modulus of elasticity (MOE)	Treatment	6	37092520	6182087	3.518	**
	Error	63	110705983	1757238		
Impact bending	Treatment	6	56.36	9.393	44.26	***
	Error	3	13.37	0.212		
Compression	Treatment	6	6730	1121.7	20.06	***
	Error	63	3522	55.9		

***** - significant, P < 0.05**

Table 5 shows the colour of the wood as a function of silicone oil heat treatment. The color change after eco-friendly silicone oil treatment of the wood was significant, as the mean change in lightness (ΔL^*) decreased from 3.07 (light) in control samples to -46.79 (dark) in silicone oil heat-treated samples at 210°C for 4h, which denote the darkening of the wood after treatment. The changes in chromatic color coordinate Δa^* and Δb^* were also observed to decrease with the increase of silicone oil heat treatment temperature from 150°C to 210°C and treatment time 2h to 4h. The decrease in the chromatic color coordinates ΔL^*, Δa^* and Δb^* with increasing temperature can be attributed to changes in the chemical composition of the wood during silicone oil heat modification of the wood.

The total color change (ΔE^*) increased as the treatment temperature was increasing with the highest value obtained when the wood was treated at 210°C for 4h. The ΔE^* increased from 3.53 in the control to 48.71 at 210°C for 4h. Based on the findings from this study, the color of the wood darkened, with increasing silicone oil heat treatment temperature and time. This result is in agreement with earlier findings, that the color change after the heat was due to the oxidative and hydrolytic reactions during the heat treatment and that dark coloration occurred in proportion to increasing temperature and time in the heat treatment (Korkut and Kocaefe 2009, Ayata *et al.* 2018).

Table 5

Mean color change after eco-friendly silicone oil heat treatment

Treatment		**Control**	**150°C**		**180°C**		**210°C**	
Time (h)			2	4	2	4	2	4
Color	ΔL^*	3.07	-23.45	-25.68	-37.78	-38.15	-39.59	-46.79
	Δa^*	0.88	3.94	4.70	3.69	-2.38	-1.73	-2.77
	Δb^*	0.90	4.53	6.36	-3.00	-9.42	-10.69	-13.11
	ΔE^*	3.53	24.35	27.06	38.15	39.49	41.06	48.71

CONCLUSION

As observed from the results of this study, the mechanical properties of Chinese parasol wood were modified after eco-friendly silicone oil heat treatment. The mean Brinell hardness (tangential, radial and transverse sections), MOR, MOE, impact bending and compression strength was severely declined with increased silicone oil heat treatment temperature and the highest decline were obtained in samples treated at 210°C for 4h.

The color variation of silicone oil heat treated Chinese parasol wood was impressively modified. Silicone oil heat treatment improved the color aesthetic of the wood as it acquired color similar to those of the tropical woods. The color of the wood darkened the more particularly at higher treatment temperature compared to the control samples. The significant color changes (dark coloration) of the wood surface indicates the effectiveness of the treatment since the wood was light in color before subjected to treatment.

Silicone oil heat-treated wood materials can be used in various applications by modifying some of the properties of the wood. Chinese parasol wood has a huge potential to be used for diverse applications than its current used, if proper treatment parameters are selected during the modification process, the wood can become more competitive in the wood market.

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