1	Original Research Article
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3 4	Physical and Mechanical properties of heat treated <i>Daniella oliveri</i> (Africa Balsam Tree) Wood
5	
6	Abstract
7	Aims: This work investigated the effect of thermal modification on some of the physical properties and mechanical
8	properties of Daniella oliveri wood.
9	Study Design: The study design used for this experiment was 3 x4 Factorial experiment in Completely
10	Randomized Design.
11	Place and Duration of Study: The study was conducted at the Federal University of Technology,
12	Akure wood laboratory and the study lasted for 6 months.
13	Methodology: Wood samples were thermally treated at the temperature of 120, 140,160 and 180 °C,
14	for different durations of 1, 1.5 and 2 hours in a muffle furnace. The planks were air-dried to reduce the
15	moisture content and then machined into the required dimensions in the direction parallel to grain with a
16	circular saw. Thirty-nine defect-free samples of dimensions 20 mm × 20 mm × 60 mm were prepared for
17	dimensional stability and compression test, static bending tests and the hardness tests to make a total of
18	117 samples.
19	$\pmb{Results}$: The result showed that the average weight loss of the treated wood samples varied from 3.79 %
20	at 120 °C for 1 hour to 7.51 % at 180 °C for 2 hours. The treatment led to reduction in density from 528 to
21	459 kg/m ³ at 180 °C for 2 hours. The heat treatment also led to reduction in water absorption and
22	volumetric swelling of the treated samples. The mean value for MOE ranges from 2.17x10 ³ N/mm ² to 2.96
23	x 10 ³ N/mm ² for the treated samples while the untreated was 2.22x10 ³ N/mm ² . Heat treatment brought
24	about improvement in the maximum compressive strength and the Janka hardness parallel to the grain of
25	wood samples. The value of compressive strength increased from 26.58 N/mm² to 41.71 N/mm² and

hardness from 69.24 N to 75.5 N. It can be therefore be concluded that thermal modification greatly enhanced the dimensional stability and mechanical properties of wood samples.

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Keywords: Compression, Daniella oliveri, Heat treatment, Hardness, Modulus of Elasticity, Weight loss

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1.0 Introduction

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The heat treatment method for modifying wood enhances its dimensional stability and is more environment-friendly as compared to other methods like chemical treatments (Poncsak et al., 2006; Kocaefe et al. 2008; Gunduz et al., 2009; Garcia et al. 2012). Although, heat treatment results in significant changes in the wood properties, but it also causes undesirable reductions in the mechanical properties of wood such as the Modulus of Elasticity. Different species of wood react differently to heat treatment based on their individual compositional variations, so it is important to determine the optimal conditions (such as duration and temperature) for heat treatment to achieve the best balance of physical and mechanical properties. To achieve this, tests must be conducted to determine the value of the properties of wood samples that have been heat treated at different durations and temperatures. As a result of heat treatment, the chemical composition of wood is altered; the hemicelluloses are most affected, and cellulose is somewhat resistant to chemical alteration (Esteves & Pereira, 2009). Other changes that occur as a result of heat treatment include increased lignin content, increased dimensional stability due to cross-linking in the lignin, the destruction of some of the sorption sites, improved durability, decreased mechanical properties such as static and dynamic bending strength and tensile strength, lower equilibrium moisture content, and darker colour as reported in previous studies (Esteves & Pereira, 2009). Many studies have been conducted to determine the influence of heat treatment on physical properties of different wood species using a wide range of treatment conditions. The difference in the species and the different treatment schedules causes changes in the physical properties, such as mass loss, anti-swelling efficiency, and equilibrium moisture content, produce a wide range of values. Mass loss is a determinative factor of the result of heat treatment, that is, the greater the mass loss, the greater the effects on the physical and mechanical properties. Gunduz et al. (2009) reported that there is a significant relationship between mass loss and compression strength of wood and Esteves et al. (2007) also observed that there

is a significant relationship between mass loss and equilibrium moisture content of wood. Brito $et\ al.$ (2006) in his studies determined the density and shrinkage behaviour of E.grandis wood where he showed that the thermal rectification process (only when a temperature of 200 °C was used) influenced shrinkage properties of wood significantly. Brito $et\ al.$ (2008) studied the changes in chemical composition that occurred when Eucalyptus and Pinus wood were subjected to heat treatment at 120, 140, 160, 180 °C, and the results showed that the arabinose, mannose, galactose, and xylose contents of the treated wood decreased significantly at 160 and 180 °C. Calonego $et\ al.$ (2011) reported the physical and mechanical properties of thermally-modified E.grandis wood, and the results showed decrease in mass, equilibrium moisture content, volumetric shrinkage to be 6.7, 21.5 and 23.2 % respectively, at a temperature of 180 °C and a duration of 2.5h. In previous studies, some properties of heat treated E.grandis wood were found to decrease as a result of heat treatment; these includes mass and equilibrium moisture content which gave different values (Garcia $et\ al.$, 2012).

Therefore, this study focused on the effect of heat treatment on *Daniella oliveri* wood at different temperatures and time durations on its dimensional stability (absorption and swelling), MC, the weight loss and mechanical properties of wood.

2.0 Materials and methods

2.1 Sample preparation

The samples for this study were obtained from a local sawmill in Akure, Ondo State, Nigeria and the planks were air-dried to reduce the moisture content and then machined into the required dimensions in the direction parallel to grain with a circular saw. Thirty-nine defect-free samples of dimensions 20 mm \times 20 mm \times 60 mm were prepared for dimensional stability and compression test. For the evaluation of static bending strength tests (modulus of rupture and modulus of elasticity), thirty-nine specimens of dimensions 20 mm \times 20 mm \times 300 mm were prepared. Another thirty-nine specimens of dimensions 30 mm \times 30 mm \times 25 mm were prepared for hardness test. In total, 117 specimens, both treated and untreated were prepared and correctly labelled. The specimens were oven dried at 103 ± 2 °C until constant weight was achieved. Thereafter, the weights and dimensions of the specimens were measured to determine the moisture content of the samples using;

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88 $MC(\%) = \frac{Wet \ weight-Oven \ dried \ weight}{Oven \ dried \ weight} \ X \ 100\%$ 89

2.2 Thermal modification process

The heat treatments were conducted in a Muffle furnace. The conditioned specimens were treated at temperatures of 120, 140,160 and 180 °C for 1, 1.5 and 2 hr. The wood samples were introduced into the furnace and then ramped to the temperature at which the actual heat treatment occurred. At the end of each treatment period, the samples were removed from the furnace and cooled in a desiccator containing silica gel. Thereafter weights and dimensions were determined to get the weight change. The weight loss (WL), was determined using equation (2):

2.2.1 Weight Loss

104 WL(%) =
$$\left(\frac{W_o - W_t}{W_o}\right) \times 100$$
 (2)

Where: WL (%) is the weight loss, W_o (g) is the oven-dry weight of specimens before the treatment and W_t (g) is the dry weight of specimens after the thermal treatment.

110 2.2.2 Dimensional stability tests

Treated and untreated wood samples were submerged in distilled water in a stainless steel container. A metal screen was placed over the samples to hold the samples approximately 2.5 cm below the surface. Water absorption and thickness swelling were assessed after 24, 48 and 72 hours of water soaking. From the measurement of the dimensions and weights of specimens, the properties measured include Density, Water Absorption (WA), and Volumetric Swelling (S) which were then calculated in accordance with ASTM standard for testing small clear samples. (ASTM, 2009).

2.2.3 Water absorption

121 WA (%) =
$$\left(\frac{W_{\text{Wet}} - W_{\text{dry}}}{W_{\text{dry}}}\right) \times 100$$
 (3)

- Where:
- WA (%) = Water absorption

2.2.4 Volumetric swelling

- W_{wet} = Weight of the samples after soaking in water
- V_{drv} = Weight of the oven dried samples.

131 VS (%) =
$$\left(\frac{V_{\text{Wet}} - V_{\text{dry}}}{V_{\text{dry}}}\right) \times 100$$
 (4)

- Where:
- VS%= Volumetric swelling
- $V_{\text{wet}} = V_{\text{olume}}$ of the samples after soaking in water
- V_{dry} = Volume of the same sample after oven drying.

2.2.5 Density determination

This is the mass per unit volume of a material.

$$141 \quad \rho = \frac{M}{V} \tag{Kg/m}^3) \tag{5}$$

Where $\rho = Density$

M = Mass (kg)

 $V = Volume (m^3)$

2.3 Mechanical properties tests

2.3.1 Static bending

For evaluation of static bending strength, three points' flexural tests were performed on both untreated and treated samples in accordance with ASTM 143 (2009). The dimension of wood samples used for the test was 20 mm x 20 mm x 300 mm. Three replicates were tested for each treated wood sample on an Instron 5500R-1137 Universal Test Machine equipped with a 454 kg load cell. Data were collected and processed using SPSS in which MOE and MOR were calculated using the software.

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$$MOE = \frac{PL^3}{4ywh^3}$$
 (N/mm²) (6)

$$161 \quad MOR = \frac{_{3PL}}{_{2wh^2}} \qquad (N/mm^2) \tag{7}$$

163 Where;

P is the load, L is the length, y is the deflection, w is the width and h is the depth or thickness of the specimen.

2.3.2 Determination of maximum compressive strength (mcs) parallel to grain

The Maximum Compressive Strength (MCS) parallel to grain was determined using sample size of 20 mm x 20 mm x 60 mm in accordance with ASTM 143 standard (2009). This was carried out with the use of Instron 5500R-1137 Universal Testing Machine with a load applied at a rate of 280 N. The values obtained were used to calculate the compressive strength using the equation below.

$$\begin{array}{ccc}
175 & \partial_{c} &= \underline{P} \\
176 & \text{bd} & \text{N/mm}^{2}
\end{array} \tag{8}$$

- ∂_c = Maximum Compressive Strength in N/mm²
- b =width in mm
- d = depth in mm
- 181 P = Load in Newton



Figure 1: Maximum Compressive Strength (MCS) parallel to grain test on a Universal

Testing Machine

2.3.3 Hardness test

A Janka hardness tester with a modified, diameter 11.3 mm ball (projected area 1 cm²) was used to determine the hardness of specimens. One centralised penetration was made on the tangential and radial face by continuously applying the load at a rate of 6.6 mm/ min. The load at which the ball attained half its penetration was recorded as the hardness (N) of the wood specimen.



Figure 2: Determination of Hardness using Janka tester

3.0 Results and discussion

3.1 Physical properties of thermally modified Daniella oliveri

Table 1 and 2 shows the physical properties of thermally modified wood of *Daniella oliveri* at each treatment duration. The weight loss values were found to differ among the different treatment temperatures and time. The values of weight loss of the treated wood varied from 3.79% at 120 °C for 1 hour to 7.51% at 180 °C for 2 hours. The highest percentage weight loss was obtained for the treated samples at 180 °C for 2 hours. Similar results were reported by Brito *et al.* (2006) who found that the thermal treatment of *E.grandis* at 200 °C caused an increase of 25% in weight loss. It is obvious from the table that the wood weight loss increased as the temperature and time increased. Garcia *et al.* (2012) found that the lower the physical the greater the weight loss and mechanical properties of wood. The results of density decreased for treated

wood samples as compared to control samples and also the values of density decreased from 528 Kg/m³ at 120 °C for 1 hour to 459 Kg/m³ at 180 °C for 2 hours while the control was 693 Kg/m³. Table 2 shows the water absorption and volume swelling at different immersion time. Moisture uptake reduced for treated wood samples than for control samples (Table 2). This might be attributed to the chemical decomposition of carbohydrates occurring at treatment temperature which are responsible for the wood-water interactions. The reduction effect of the thermal treatment on the water absorption was more remarkable compared to the long immersion time as shown in Table 2. It should be noted that the water absorption values of heat-treated wood were higher than those of untreated wood samples for each of the three immersion period. The result shows that thermally treated samples are more effective in moisture uptake reduction in short duration exposure to water, but loses its relative influence with prolonged time, which can be attributed to a lowered fibre saturation point of thermally treated wood. At the early stage of water soaking, water is located within the cell wall until the fibre saturation point is reached. After this point, water is located in the cell lumen as free water. The water absorption values increased with increasing temperature of thermal treatment in accordance with the work of Dundar et al., 2012. The study demonstrates that the thermally modified D. oliveri wood showed reductions of 13.38% at 160 °C for 1.5 hours to 5.41% at 180 °C for 2 hours in volumetric swelling when soaked in water for 24 hours compared with the values found for untreated wood (29.32 %). Similar results were presented by Brito et al. (2006), who found that the thermal treatment of E. grandis for 200 °C caused a decrease of 25% in volumetric shrinkage, and by Calonego et al. (2012), who concluded that wood of E. grandis thermally modified at 220 °C presented improvement in the volumetric swelling. The effects of thermal treatment on the physical

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properties of *D.oliveri* wood showed that temperatures up to 180 °C for 2 hours led to improvement in dimensional stability without losses of material. The results of this study agreed with those reported by Bhuiyan *et al.* (2001), Calonego *et al.* (2012), Metsä-Kortelainen *et al.* (2005) for thermally modified wood of other species. Also, Korkut and Guller (2008) reported a reduction in some physical properties, oven-dry density, air-dry density and swelling of wood. Kocaefe *et al.*, (2007) also reported that wood subjected to high temperature loses its capacity to reabsorb water in contrast to the hydrophilic behavior of the conventionally dried wood.

Table 1: Mean values of Physical properties of thermally modified *Daniella oliveri*

Transfer ant (OC)	Time	Dangita	M C(0/)	Waight Lagg (0/)
Treatment(°C)	Time	Density	M.C(%)	Weight Loss (%)
	(min)	(Kg/m³)		
Control	-	693±118.56	12.83 ± 6.04	-
120	60	528±95.98	8.77±1.65	3.79 ± 2.01
120	90	521±44.91	6.61±0.58	4.91 ± 0.86
120	120	496±23.84	6.56 ± 0.70	5.55 ± 0.25
140	60	509±56.69	8.43 ± 3.35	3.88 ± 2.60
140	90	514±40.81	9.36 ± 5.30	5.63 ± 1.01
140	120	489±33.07	6.18 ± 0.33	5.44 ± 0.44
160	60	500±23.38	6.51 ± 0.89	5.98 ± 0.57
160	90	463±22.22	6.59 ± 0.33	6.56 ± 0.37
160	120	492±2.18	7.00 ± 1.33	6.35 ± 0.33
180	60	516±23.04	6.75 ± 1.68	7.45 ± 0.61
180	90	474±28.40	6.85 ± 0.86	7.13 ± 0.49
180	120	459±13.43	5.90 ± 0.28	7.51 ± 0.22

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Table 2: Mean values of Physical properties of thermally modified Daniella oliveri

Treatment(°C)	Time	WA (%)	WA (%)	WA (%)	VS (%)	VS (%)	VS (%)
	(min)	(24h)	(48h)	(72h)	(24h)	(48h)	(72h)
Control	0	73.9±36.79	88.2±39.24	95.3±43.7	29.3±12.40	32.40±1.93	37.14±4.08
120	60	98.2±14.64	110.0±15.2	113±14.49	11.14±6.03	13.40±3.34	25.07±10.63
120	90	99.6±8.72	111.0±5.36	119±7.16	10.02±5.73	13.39±6.08	15.60±6.43
120	120	104.0±3.49	118.0±2.32	123±1.33	8.09±2.62	9.53±3.05	14.15±3.19
140	60	101.0±3.41	114.0±6.83	118±7.19	9.43±1.96	11.47±4.18	15.16±3.79
140	90	74.9±28.08	90.4±28.27	97.3±28.37	7.91±3.17	13.44±6.93	14.03±2.87
140	120	94.1±6.29	111.0±28.2	116±7.96	9.57±3.32	11.72±1.68	16.37±1.38
160	60	93.0±33.06	106.0±29.3	116±30.97	12.27±2.65	9.48±2.02	14.09±4.12
160	90	98.02±9.53	113.0±6.5	120±9.65	12.70±1.80	9.26±2.08	14.06±0.38
160	120	88.03±7.87	103.8±7.9	111±4.34	13.38±0.05	9.79±0.61	16.17±0.91
180	60	75.21±24.1	92.7±21.28	100±21.38	12.74±2.28	16.54±1.67	16.11±1.56
180	90	74.67±8.6	93.89±6.06	103±6.13	8.39±5.68	12.13±5.69	14.34±3.15
180	120	66.3±10.2	85.89±12.8	95.3±15.5	5.41±0.98	9.03±1.61	9.86±1.58
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251 Mechanical Properties of thermally treated *Daniella oliveri*

Table 3: Mean values of Mechanical properties of thermally modified *Daniella oliveri*

Time (min)	MOR(N/mm ²)	MOE(N/mm ²)	Compression test(N/mm ²)	JankaHardness test(N)
0	46.43±7.10	2220±675.7	24.92±7.69	68.34
60	42.75±19.1	2440±956.6	26.58±8.84	69.24
90	43.90±6.96	2630±184.3	36.60±4.11	69.28
120	51.91±3.77	2960±244.7	35.93±3.65	71.8
60	49.95±2.79	2760±93.29	29.89±1.24	71.95
	y			
90	48.65±2.67	2490±154.0	32.09±0.91	73.37
120	53.79±2.22	2490±35.10	40.46±9.10	74.62
60	40.64±0.65	2870±383.8	41.71±7.68	72.05
90	43.7±11.18	2650±687.6	31.92±1.88	72.85
120	33.89±10.2	2170±706.1	29.50±6.37	70.25
60	45.56±3.34	2790±136.1	35.20±2.25	75.5
90	35.55±23.7	2430±902.8	25.39±11.03	73.97
120	39.34±5.96	2350±454.9	29.4±36.94	74.07
	(min) 0 60 90 120 60 90 120 60 90 120 60 90	(min) 2) 0 46.43±7.10 60 42.75±19.1 90 43.90±6.96 120 51.91±3.77 60 49.95±2.79 90 48.65±2.67 120 53.79±2.22 60 40.64±0.65 90 43.7±11.18 120 33.89±10.2 60 45.56±3.34 90 35.55±23.7	(min) 2) 0 46.43±7.10 2220±675.7 60 42.75±19.1 2440±956.6 90 43.90±6.96 2630±184.3 120 51.91±3.77 2960±244.7 60 49.95±2.79 2760±93.29 90 48.65±2.67 2490±154.0 120 53.79±2.22 2490±35.10 60 40.64±0.65 2870±383.8 90 43.7±11.18 2650±687.6 120 33.89±10.2 2170±706.1 60 45.56±3.34 2790±136.1 90 35.55±23.7 2430±902.8	(min) 2) test(N/mm²) 0 46.43±7.10 2220±675.7 24.92±7.69 60 42.75±19.1 2440±956.6 26.58±8.84 90 43.90±6.96 2630±184.3 36.60±4.11 120 51.91±3.77 2960±244.7 35.93±3.65 60 49.95±2.79 2760±93.29 29.89±1.24 90 48.65±2.67 2490±154.0 32.09±0.91 120 53.79±2.22 2490±35.10 40.46±9.10 60 40.64±0.65 2870±383.8 41.71±7.68 90 43.7±11.18 2650±687.6 31.92±1.88 120 33.89±10.2 2170±706.1 29.50±6.37 60 45.56±3.34 2790±136.1 35.20±2.25 90 35.55±23.7 2430±902.8 25.39±11.03

The MOR, MOE, Compression test and Janka hardness tests are shown in the Table 3. It was observed that the modulus of rupture values of the treated samples ranged from 53,79 N/mm² at 140 °C for 120 mins to 33.89 N/mm² at 160 ℃ for 120 mins and later increased to 45.56 N/mm² at 180 ℃ for 60 mins compared to untreated sample which had a value of 46.43 N/mm². The decrease in strength properties of wood after thermal treatment can be explained by the rate of thermal degradation and losses of substances after treatment. The decrease in strength is mainly due to the depolymerisation reactions of wood polymers (Wikberg and Maunu, 2004). Lowest values were recorded for Modulus of Rupture at the treatment of 160 °C for 2h. The Janka hardness parallel to the grain increased from 69.24 N to 75.5 N after heat treatment and the maximum hardness value was obtained as 75.5 N at 180 °C for 1 h. These results can be explained with loss in cell wall material and hemicelluloses degradation at high treatment temperature after heat treatments. Decrease in strength is mainly due to the de-polymerization reactions of wood polymers (Kotilainen, 2000). Furthermore, the wood density plays a key role on the mechanical properties of the wood, similar results for heat treated wood about reductions in mechanical strength properties were reported by Santos (2000) and Poncsak et al. (2006). The effect of heat treatment on the elastic properties of wood is minimal, although there was an increase in the value of the MOE during the bending test. Degradation of the hemicelluloses, disrupting the loadsharing capacity of the lignin-hemicelluloses matrix, and increase of the relative amount of crystalline cellulose could contribute to the increase of the MOE. The Modulus of Elasticity of the treated samples range from 2.17x 10³ N/mm² to 2.96 x 10³ N/mm² compared to the control (2.22 x 10³ N/mm²). The increased cross linking of the lignin network probably affects the MOE, since it is expected that an increased cross linking improves the rigid structure around the cellulose microfibrils and the strength characteristics of the middle lamella. Furthermore, heat treated wood is less hygroscopic than untreated wood (it contains less bound water in the cell wall), which affects the MOE making wood less pliable (Kubojima, 2000). The maximum compressive strength of wood samples parallel to grain increased from 26.58 N/mm² to 41.71 N/mm² and decreased after heat treatment. The increase of the compressive strength in longitudinal direction might be due to a lower amount of bound water in heat treated wood, however it is

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expected that the amount of bound water must be higher to affect the strength properties. Generally, the values of the different strength tests increased after heat treatment. Suleyman and Ayhan (2015) reported similar result for wild cherry and reported that the Compression strength values of wild cherry wood samples were decreased with increasing treatment time. It is clear from Table 3 that there is slight increase in the hardness number of wood with a decrease in the moisture content as the temperature increased with time (Ohsawa and Miyajima, 1959).

4.0 Conclusion

This study revealed that water absorption and volumetric swelling of the wood species studied decreased with increased heat treatment intensity. Thermal treatment resulted in reduction in swelling and water absorption of the samples to great extent.

The weight loss increased and density decreased with increased heat treatment at a temperature of 180 °C for duration of 2 hours. The density and weight loss decreased for the milder treatments, while as the duration increases this decrease tends to be slower and more gradual, which indicates that treatment duration of 2 hours to reduce the water re-absorbing capacity and hygroscopic properties of *Daniella oliveri* wood. However, temperature of 180 °C for 2 hours could be applied to wood where physical properties are preferred. The Modulus of Elasticity and Modulus of Rupture of the wood species decreased with heat treatment. The Maximum Compression strength parallel to grain decreased with heat treatment. The hardness increased with increased in heat treatment. Therefore, heat treated wood can be utilized with appropriate heat treatment time and temperature without any loss in strength values in areas, where woodwork such as parquet flooring and decorative purpose, stability are important.

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394	Competing Interest

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Authors have declared that no competing interest exists.