# **Original Research Article**

# IMPACT OF THERMAL TREATMENT ON ANATOMICAL AND MECHANICAL PROPERTIES OF *Rhicinodendron heudelotii* WOOD

#### 5 6 ABSTRACT

7 The study examined the impact of thermal treatment on anatomical and mechanical properties 8 Rhicinodendronheudelotiiwood. Wood samples were oven dried at 105°C and cooled in a desiccator to a constant 9 weight before the thermal treatment. Heat treatment of wood was carried out in a Furnace at 120°C, 140°Cand 160°C 10 for 45 minutes and 90 minutes. The Density, Modulus of Elasticity (MOE), Modulus of Rupture (MOR), and the anatomical properties were assessed. The results for Density of Rhicinodendron heudelotii wood showed decrease 11 from 279 kg/m<sup>3</sup> (120°C at 45 minutes) to 256 kg/m<sup>3</sup> (160°C at 90 minutes) while that of control was 281 kg/m<sup>3</sup> 12 which was lower than the treated samples. The increase in temperature with time had effect of the color of wood as 13 14 it changed from creamy white to dark brown. Theimage of untreated and treated samples showed no significant 15 changes within and among treatment groups as there was no effect of treatment time and exposure on the samples.

The MOE of heat treated *Rhicinodendron heudelotii* increased from 2064.84 N/mm<sup>2</sup> (140°C for 45 minutes) to
2271.93 N/mm<sup>2</sup> (160°C for 90 minutes) while MORdecreased from 40.56 N/mm<sup>2</sup> (140°C for 90 minutes) to 33.53
N/mm<sup>2</sup> (160°C for 90 minutes). The study revealed that the wood could be used in a light furniture as unnecessarily
heavy wood is not important. Also, the study proved effective in improving the modulus of elasticity of the wood.
Keywords: Mechanical properties, Rhicinodendronheudelotii, Heat treatment, Colour, Density

#### 21 **1.0 Introduction**

Wood as a renewable natural resource has been used by man for thousands of years since his appearance on Earth and has contributed to his survival and to the development of civilization. For many reasons, the natural wood properties of some species do not give adequate performance which necessitate modification of the material (Ajuziogy*et al.*,2014). Wood is a complex polymeric material which consists mainly of cellulose, hemicellulose and lignin with a minor quantity of extractives. The exposure of wood to elevated temperatures causes thermal degradation of its structure such as degradation of hemicellulose often accompanied by loss of mass and thus modifying the properties of wood. Thermal degradation is utilized commercially to produce wood products with improved dimensional stability and reduced hygroscopicity (Akyildiz *et al.*, 2008).

Wood is a hygroscopic material that is capable of losing and gaining moisture due to the fact that the cell wall polymers contain hydroxyl groups. In an environment containing moisture, dry wood will absorb moisture until it reaches equilibrium with the surrounding atmosphere. Similarly, saturated wood when placed in an atmosphere of lower relative humidity (*H*) will lose moisture until equilibrium is attained. If the wood is placed in an environment where the relative humidity is stable, it will attain constant moisture content which is known as the equilibrium moisture content ( $M_{emc}$ ). At this point, the wood no longer loses or gain moisture. After treatment at 180°C or above, there is chemical changes in lignin and hemicelluloses of the treated wood making it to be less hygroscopic.

Wood modification can be defined as a process that improves the properties of wood by producing a new material that when disposed at the end of the product life cycle doesn't present an environmental hazard any greater than unmodified wood (Hills, 2006). However, the thermal modification of wood is defined as the application of heat to wood to cause a desired improvement in the properties of the material. Thermal modification is invariably performed between the temperature of 180°C and 260°Cwith temperature lower than 140°C resulting in only a slight change in the material properties. Temperature is one of the most important parameters responsible for the effect of modification in heat treated wood (Mitchell,1988, Korkut&Guller, 2008).

In hardwood, the cells that make up the anatomical organization are the vessels, parenchyma cells, fibres and the 45 46 wood rays, although, fibres are the main element that is responsible for the strength of the wood (Panshin&Zeeuw, 47 1980). Wood density is one of the most important wood property and indicator of strength for solid wood and fibre products (De Guth, 1980), It is usually affected by the cell wall thickness, the cell diameter, the earlywood to 48 49 latewood ratio and the chemical composition of the wood (Cave & Walker, 1994). Philips (1941) reported that wood density is a measure of the cell wall material per unit volume and as such gives a very good indication of the 50 51 strength properties and expected pulp yields of timber (Philips, 1941). In softwood, the cells that make up the 52 anatomical organization are the vessels, fibers, parenchyma cells and the wood rays. Fibers are the principal element that is responsible for the strength of the wood same as hardwood (Panshin&Zeeuw, 1980)Wood density is an important wood property for solid wood and fibre products, it is one of the most important properties influencing the guality of a timber (De Guth, 1980).

Variation in anatomical properties is therefore not observed only from one species to the other but also within the same species, within a tree, and also between the core wood of the tree and the outer wood (Albert *et al.*, 2004). According to Boonstra *et al.* (2006a) heat treatment affects the anatomical structure of wood but this effect depends on the wood species and the process parameters used. Softwood species are the most susceptible to tangential cracks in the latewood section, especially with narrow annual rings and/or an abrupt transition between earlywood and latewood.

#### 62 **2.0 Materials and Methods**

#### 63 **2.1 Sample Size and Collection of Samples**

*Rhicinodendronheudelotii* belongs to the Euphorbiaceae family. It grows in tropical Africa from Guinea to Angola,
East Africa and Madagascar in Cameroon.*Rhicinodendron heudelotii*log was obtained from a local sawmill in
Akure, Ondo State, Nigeria. The lumbers were air-dried to approximately 20% moisture content and cut along and
across the grain with a Circular sawing machine to get the heat treatment specimens. Twenty-eight defect-free
specimens of 20×20×10 mm were prepared for the evaluation of Density and weight loss according to ASTM
standards.

#### 70 2.2 Material Preparation

#### 71 2.2.1 Sample Preparation

For the evaluation of static bending strength tests (Modulus of rupture and Modulus of elasticity), twenty-eight specimens of 300 mm  $\times$  20 mm  $\times$  20 mm were prepared. A total of 56 specimens of both treated and untreated were prepared and properly labelled for the study. The specimens were oven dried at  $103\pm 2^{\circ}$ C until constant weight was achieved and then cooled in a desiccator containing silica gel to obtain the final weight. The wet weight and dry weight was estimated with a weighing balance of model H-1117 in the laboratory.

For anatomical samples, a radial sample of 1cm thickness was cut from the sample and then spitted using a bluntknife along the radial and tangential planes to make blocks in anatomical orientation, this orientation facilitates

79 microtome sectioning. The radial sample was softened by immersion in water for hours (marked with a soft pencil) 80 until it sinks under its own weight. Thin section was then taken from the three oriented planes: tangential, transverse 81 and radial section using a microtone sliding machine. It was placed in a petri-dish containing methylated spirit for 82 moistening and also to prevent rolling and flaking character of the wood section. Staining was done by adding drops 83 of safranin solution to the sections in the petri-dish and left for few minutes.

#### 84 2.2.2 Slide Preparation

After the microtome sample has been stained with safranin solution for few minutes, it was rinsed with water and alcohol until the excess stain was removed. It was dehydrated using ethanol and then placed on slide using forceps while the rough edges were also trimmed. Improvised Canada balsam was added to ensure clarity and was covered with coverslip (microscopic coverslip). Heat was finally applied to expel air bubbles from the prepared samples. Slide was then observed under a microscope.

#### 90 2.2.3 Maceration Process

Each wood samples was cut to slivers about 1mm thick and 1-2 cm in length and then subjected to maceration process. The slivers of each specimen was placed in a test tube and immersed with a mixture of acetic acid and hydrogen peroxide. The tubes and its contents were placed in an oven at 60°C for 48 hours, removed after 48 hours from the oven and washed thoroughly with water to remove acids from the process. The treated slivers were subjected to shaking to obtain more individual fibers. The macerated fibers were washed with distilled water and individual fibers were separated on slides, it was then measured under a microscope. All fibers were determined by Leica Image Analyzer System to get the *Fibre Length*, *Fibre diameter* and *Fibre lumen*.

#### 98 **2.3 Thermal treatment process**

99 The heat treatment was conducted in a furnace with a temperature-controlled heating unit. The conditioned 100 specimens (at constant M.C. of 12 %) were thermally treated at temperatures of 120, 140 and 160°C for the duration 101 of 45 and 90 minutes each. The temperature of the furnace was ramped to the temperature at which the actual heat 102 treatment occurred before introducing the wood samples. At the end of each treatment period, the samples were 103 removed from the furnace followed by weight and dimension estimation after cooling in a desiccator with a silica 104 gel to account for the weight change.

#### 105 2.3.1 Moisture Content Determination

Therefore, the weight and dimension of all specimens were measured to determine the moisturecontent (MC) using:

(1)

108 MC (%) = 
$$\frac{wet weight-oven dry weight}{oven dry weight} \times 100$$

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110 Where  $W_o(g)$  = the oven-dry weight of specimens before the treatment and

- 111  $W_t(g)$  = the oven-dry weight of specimens after the treatment.
- 112
- 113 The percentage weight loss (PWL) was determined using:

114 WL (%) = 
$$\left(\frac{Wo - Wt}{W_0}\right) x \ 100$$
 (2)

115 Where: *Wo*(g) is the oven dry weight of specimens before the treatment;

116 *Wt* is the dry weight of specimens after treated with heat

#### 117 2.3.2 Density Determination

118 Density is defined as the mass per unit volume of a material. Samples of 20 mm x 20 mm x 60 mm was cut for 119 density determination. After cutting, the wood samples were weighed on the weighing balance to estimate the green 120 weight; the samples were oven-dried at  $103\pm2^{\circ}$ C for 24 hours until the weight remained constant. The density  $\rho$  of 121 the wood species was determined using;

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$$\rho = \frac{M}{V} (kg/m^3)$$
(3)

## 123 Where: M = Mass (kg) and V = Volume (m<sup>3</sup>)

#### 124 **2.3.3 Mechanical Properties**

The Modulus of elasticity (MOE) and Modulus of rupture (MOR) was evaluated by subjecting samples of 20 mm x 20 mm x 300 mm to a force/load on the Universal Testing machine. All samples were supported by metal bearing plates to prevent damage to the beam at the point of contact between specimen and reaction support. At the point of failure, the force exerted on the specimen that caused the failure was recorded, the MOE and MOR was calculated using;

#### 130 **2.3.3.1 Moduli of elasticity and rupture**

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$$MOE = \frac{PL^3}{4bd^3D}$$
 (4)  
132  $MOR = \frac{3PL}{2bd^2} (N/mm^2)$  (5)

Where: L= Length of specimen between the machine supports (mm); b= Width of the specimen (mm); d= thickness
of the specimen (mm); P= Ultimate load (N); and D= Axial deformation

#### 135 2.4 Statistical Analysis

The experimental design for the study was a 2x3 Factorial experiment in Completely Randomized Design, the experiment was designed to include the predictor variables; Factor A (temperature) and Factor B (time). Data was analyzed using descriptive statistics, general linear model and univariate Post Hoc Test at logistic regression at 0.05.

#### 139 3.0 Results and discussion

#### 140 **3.1 Color**

For both maximum temperatures tested, *Rhicinodendron heudelotii* woods became darker at 160°C 90 minutes of treatment. The changes in color were higher with elevated temperature and this is in accordance with the report of previous researchers (Olufemi *et al.*, 2018; Aksoy *et al.*, 2011) who assessed the color changes of oven heat treated Leucaena *leucocephala* and scot pine at elevated temperatures. The authors reported that heat-treated wood at temperatures above 160°C acquired darker tonality. Heat treatment improved some wood properties such as color being a good approach to improve low market valued tropical woods (Diego *et al.*, 2014). According to Sundqvist (2004), the changes in color of thermally modified wood are attributed to oxidative changes which predominate over hydrolysis reactions. The effect of extractives in the color of heat-treated wood showed that unextracted and acetone extractives had varying color (Sundqvist, 2004). The report by Sundqvist asserted that both extractives and structural components such as hemicelluloses and lignin took part in color change of heat treated wood.

### 151 **3.2 Density Determination**

The mean value of *Rhicinodendron heudelotii* wood decreases from 279 kg/m<sup>3</sup> (120 °C at 45 minutes) to 256 kg/m<sup>3</sup> (160°C at 90 minutes) while that of control was 281 kg/m<sup>3</sup> as shown in Fig.1. The density of the treated samples was lower compared to control samples. It was observed that the density of wood samples decreases with time of exposure to heat treatment. The results are presented in Figure 1 as density decreases with elevated temperature. Heat treatment increase mass loss due to the release of by-products from the wood during thermal degradation of lignin and as a result of the evacuation of volatile compounds which reduces the density of wood. (Olufemi *et al.*, 2018)







Fig 1: Mean values of Density of Thermally treated Rhicinodendron heudelotii

#### 3.3 Mass loss 162

163 The mean value of the mass loss for Rhicinodendron heudelotii wood increased from 3.35 % (120°C at 45 minutes) 164 to 5.57% (160°C at 90 minutes) in Fig 2. The mass loss of treated wood samples was higher as the temperature 165 increased with time. It was observed that higher temperature and time has a significant effect on mass loss. Mass 166 loss in wood increases with increasing temperature or treatment time which was consistent with previous study 167 (Poncsacet al., 2006; Esteves et al., 2008).



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Fig 2: Mean values of Weight loss (%) of thermally treated Rhicinodendron heudelotii. 169

#### **3.4 Mechanical Properties** 170

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#### 3.4.1 Moduli of elasticity and rupture 172

173 Modulus of elasticity (MOE) implies that deformations produced by low stress are completely recoverable after loads are removed. The MOE of heat treated Rhicinodendron heudelotii increased from 2064.84 N/mm<sup>2</sup> (140°C for 174 45 minutes) to 2271.93 N/mm<sup>2</sup> (160°C for 90 minutes).

- 176 Modulus of rupture reflects the maximum load carrying capacity of a member in bending and is proportional to
- 177 maximum moment or force borne by the specimen. The variation in the MOR of thermally treated Rhicinodendron
- 178 heudelotii ranged from 40.56 N/mm<sup>2</sup> (140°C for 90 minutes) to 33.53 N/mm<sup>2</sup> (160°C for 90 minutes). MOR of the
- 179 samples therefore declined with increasing temperature and time in a pattern that was consistent with the previous
- 180 studies (Boonstra etal., 2007; Ding et al., 2011; Lekounougou & Kocaefe, 2014).

#### **181 3.5 Anatomical Properties**

182 Microscopic investigations was performed on each treatment group in the three planes that is, the transverse, 183 tangential and radial plane to better understand the effect of elevated temperatures on the anatomical properties of 184 wood. Images of untreated and treated wood are shown in Figure 3 for comparison, although no significant changes 185 were noticed within and among treatment groups.



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(b) 140<sup>°</sup> C for 45 mins and 90 mins in Radial plane



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(c) 160<sup>°</sup>C for 45 mins and control sample

Fig 3 SEM images of the three sections of untreated *Rhicinodendron heudelotii* wood and samples thermally
 modified at 120, 140 and 160 °C and at 45 and 90 minutes each. Arrow showing crystal in *Rhicinodendron heudelotii* wood.

196 No significant changes were noted in the structure of the fibrous and parenchymatous tissues.

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## TABLE 1 Density, Weight loss (%) and Moisture Content of Rhicinodendron heudelotii wood at

different temperature and pressure.

Temp	Time	Density(Kg/m <sup>3</sup> )	Weight loss (%)	MC(%)
0	0	281.06	0	6.82
120	45	271.16	13.39	3.47
120	90	259.02	14.16	3.75
140	45	262.59	15.73	4.09
140	90	258.94	18.02	4.77
160	45	257.64	18.21	4.94
160	90	256.71	22.28	6.23



Temp	Time	MOE(N/mm <sup>2</sup> )	MOR (N/mm <sup>2</sup> )
0	0	2173.575	37.59422
120	45	2127.808	35.52422
120	90	2144.686	41.42156
140	45	2064.84	38.84484
140	90	2149.611	40.55906
160	45	2217.154	33.66984
160	90	2271.928	33.52969

TABLE 3 Fiber length (FL), Fiber diameter (FD), Lumen width (LW), Cell wall thickness (CWT),

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Runkel ratio (RR), Flexibility coefficient (FL), and Slenderness of the fibre (SF)

		Fibre	Fibre	Lumen	Cell wall	Runkel	flexibility	slenderness	
Temp	Time	length	diameter	width	thickness	ratio	coefficient	of the fibre	
0	0	1.56	0.41	0.32	0.04	0.28	78.03	3.82	
120	45	1.70	0.55	0.33	0.11	0.65	60.55	3.13	
120	90	1.65	0.53	0.40	0.06	0.32	75.87	3.14	
140	45	1.61	0.53	0.34	0.10	0.56	63.90	3.06	
140	90	1.64	0.50	0.30	0.10	0.68	59.60	3.29	
160	45	1.71	0.54	0.42	0.06	0.28	78.32	3.19	
160	90	1.63	0.54	0.43	0.05	0.24	80.44	3.05	

#### 214 **4.** Conclusions

The thermal modification negatively affects the density and improve the modulus of elasticity of *Rhicinodendron heudelotii* wood. The mass loss increases with increase in temperature and time. Heat treatment decreased wood density and increased the weight loss of the species. The degree of thermal modification effects depends on the treatment conditions (temperature and time).

The color of the wood changed from creamy white to dark brown with increasing treatment temperature and duration. Based on the results obtained from this research, it is clear that heat treated wood has greater dimensional stability than the untreated wood. The effect of thermal modification on ray parenchyma width requires further investigation.

The qualitative analysis using SEM indicated that no significant changes occurred in the structure of ray parenchyma, vessels and fibers tissues. Even crystals were preserved in the vessel elements of thermally modified wood at the highest processing temperature (160°C) and no quantitative changes were observed in the dimensions of fibers.

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