Original Research Article

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

ABSTRACT

 The study examined the impact of thermal treatment on anatomical and mechanical properties *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 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 12 from 279 kg/m³ (120°C at 45 minutes) to 256 kg/m³ (160°C at 90 minutes) while that of control was 281 kg/m³ which was lower than the treated samples. The increase in temperature with time had effect of the color of wood as it changed from creamy white to dark brown. Theimage of untreated and treated samples showed no significant changes within and among treatment groups as there was no effect of treatment time and exposure on the samples.

16 The MOE of heat treated *Rhicinodendron heudelotii* increased from 2064.84 N/mm² (140°C for 45 minutes) to 17 2271.93 N/mm² (160°C for 90 minutes) while MORdecreased from 40.56 N/mm² (140°C for 90 minutes) to 33.53 18 N/mm² (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*

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 36 moisture content (M_{enc}). At this point, the wood no longer loses or gain moisture. After treatment at 180 $^{\circ}$ 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 42 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 wood rays, although, fibres are the main element that is responsible for the strength of the wood (Panshin&Zeeuw, 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 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 strength properties and expected pulp yields of timber (Philips, 1941). In softwood, the cells that make up the 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 quality 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.

2.0 Materials and Methods

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 68 specimens of $20\times20\times10$ mm were prepared for the evaluation of Density and weight loss according to ASTM standards.

2.2 Material Preparation

2.2.1 Sample Preparation

 For the evaluation of static bending strength tests (Modulus of rupture and Modulus of elasticity), twenty-eight 73 specimens of 300 mm \times 20 mm \times 20 mm were prepared. A total of 56 specimens of both treated and untreated were 74 prepared and properly labelled for the study. The specimens were oven dried at 103 ± 2 °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 blunt knife along the radial and tangential planes to make blocks in anatomical orientation, this orientation facilitates microtome sectioning. The radial sample was softened by immersion in water for hours (marked with a soft pencil) until it sinks under its own weight. Thin section was then taken from the three oriented planes: tangential, transverse and radial section using a microtone sliding machine. It was placed in a petri-dish containing methylated spirit for moistening and also to prevent rolling and flaking character of the wood section. Staining was done by adding drops of safranin solution to the sections in the petri-dish and left for few minutes.

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.

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 93 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 95 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*.

2.3 Thermal treatment process

 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 $^{\circ}$ C for the duration of 45 and 90 minutes each. The temperature of the furnace was ramped to the temperature at which the actual heat treatment occurred before introducing the wood samples. At the end of each treatment period, the samples were 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**

106 Therefore, the weight and dimension of all specimens were measured to determine the moisture

107 content (MC) using:

108 MC
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(\%) = \frac{\text{wet weight-over dry weight}}{\text{oven dry weight}}
$$
 x 100 (1)

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110 Where W_0 (g) = the oven-dry weight of specimens before the treatment and

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

114 WL (
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\%
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) = $\left(\frac{Wo - Wt}{W_0}\right) x$ 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 $103\pm2\degree$ C for 24 hours until the weight remained constant. The density ρ of 121 the wood species was determined using;

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122 \qquad \rho = \frac{M}{V} \left(kg/m^3 \right) \tag{3}
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123 Where: M= Mass (kg) and V= Volume $(m³)$

2.3.3 Mechanical Properties

 The Modulus of elasticity (MOE) and Modulus of rupture (MOR) was evaluated by subjecting samples of 20 mm x 126 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;

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)

133 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

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.

3.0 Results and discussion

3.1 Color

141 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 145 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.

3.2 Density Determination

152 The mean value of *Rhicinodendron heudelotii* wood decreases from 279 kg/m³ (120 $^{\circ}$ C at 45 minutes) to 256 kg/m³ 153 (160°C at 90 minutes) while that of control was 281 kg/m³ 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*

162 **3.3 Mass loss**

The mean value of the mass loss for *Rhicinodendron heudelotii* wood increased from 3.35 % (120^oC at 45 minutes) 164 to 5.57% (160 $^{\circ}$ 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 (Poncsac*et al*., 2006; Esteves *et al*., 2008).

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

170 **3.4 Mechanical Properties**

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172 **3.4.1 Moduli of elasticity and rupture**

173 Modulus of elasticity (MOE) implies that deformations produced by low stress are completely recoverable after 174 loads are removed. The MOE of heat treated *Rhicinodendron heudelotii* increased from 2064.84 N/mm² (140°C for

175 45 minutes) to 2271.93 N/mm² (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/mm² (140°C for 90 minutes) to 33.53 N/mm² (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).

3.5 Anatomical Properties

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

(a) 1200 C for 45 mins and 90 mins in Tangential plane

(b) 140⁰ C for 45 mins and 90 mins in Radial plane

(c) 160⁰C 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.

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

199 different temperature and pressure.

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

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

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

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 218 treatment conditions (temperature and time).

219 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 225 wood at the highest processing temperature $(160^{\circ}C)$ and no quantitative changes were observed in the dimensions of fibers.

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