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Influence of Thermal Modification on the Physico-Mechanical Properties of *Alstonia Boonei* (De Wild) Wood

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Abstract
Thermal modification at relatively high temperatures (ranging from150 to 260◦C) is an effective method to improve the properties of wood against prevailing conditions. This study was therefore performed to investigate the impact of heat treatment on the physical and mechanical properties of *Alstonia boonei wood*. The conditioned specimens (at constant moisture content of 12%) were thermally treated at temperatures of 125, 150 and 175^{o}C for 30, 60 and 120 minutes. Twenty seven defect-free specimens of dimensions 20 mm × 20 mm ×10 mm (length × breadth × thickness) were used. The colour of thermally treated *Alstonea boonei wood* varied from pale creamy white to light brown at 150ºC for 30, 60 minutes and 120min and to very dark brown at 175°C for120 minutes. At 2hrs, the mean values of the thermally treated samples for volumetric swelling ranged from the lowest (3.73 at 150°C for 120 minutes) to the highest (10.13% at 125°C for 60 min). The thermal modification of *Alstonia boonei wood* affected its physical properties. The extent of thermal modification varied with temperature and duration of treatment. The heating temperature is applicable for the changes in the MOR of the thermally modified wood. The bending strength decreased drastically with high treatment temperature. The MOE of heat treated wood samples of this species varied from 5.94 x 10^5 MPa (175° C for 30 min) to 5.10 x 10^6 MPa (150°C for 120 min).The extent of thermal modification varied with temperature and the duration of treatment.

Keyword: Dimensional stability, Hygroscopicity, color change, Density, bending strength

Introduction
Wood is a versatile material with a wide range of applications. It is also a renewable resource with an exceptional strength-to-weight ratio. It is the fifth most important product of the world trade (Christophe and Gregoire, 2001). In Nigeria, more than 80% of the timber products are used for constructional purposes such as building, furniture, railway sleepers, transmission poles, veneer, plywood and composites boards (Akanbi and Ashiru, 2002). Wood attracts moisture through hydrogen bonding, making it dimensionally unstable, which in turn promotes biological degradation (Stamm, 1964). Over-exploitation and deforestation have left Nigerian forests with young and small diameter trees which are mostly composed of a high proportion of juvenile wood as well as lesser known or underutilized timber species. These trees contain less chemical extractives that could make wood naturally durable. Therefore, construction industries are faced with finding solution to the problem of wood degradation and instability imposed by reduction in quantity and quality timber species in terms of strength and durability. In other to stem the problem mentioned above as well as to expand the usage of underutilized wood and wood of low density, it is imperative to improve the properties of wood by an environmentally feasible procedure. The improvement on the inherent properties of wood polymers and their structures could be achieved through various wood modification techniques like thermal, hydrothermal, chemical, mechanical (densification) and plasma modifications (Hills 2006).

As an environmentally well established technology, thermal modification is one of the chemical free processes used to improve the properties of wood through alteration of the chemical properties of wood permanently. Thermal modification is invariably performed between the temperatures of 180 and 260°C, with temperatures lower than 140 °C resulting in only slight changes in material properties and higher temperatures resulting in unacceptable degradation to the substrate (Hills, 2006). When wood is heated, its chemical and physical properties undergo permanent changes and its structure is transformed (Boostra *et al*., 2007). These observed changes in the

properties are mainly due to degradation of hemicelluloses (Pfriem, 2006). Heating of wood in the temperature range of 140-260^oC for long period causes the irreversible reduction of its capability for moisture uptake (Obataya and Tomita, 2002; Gonzalez *et al*, 2004).
This study is aimed at investigating the effects of thermal modification on physical properties of *Alstonia boonei* wood. Also, since thermal treatment often brings about negative effect on mechanical properties, MOE and MOR of the treated wood were also examined for effective utilization.

Materials and Methods

Preparation of Wood Samples

Commercial lumber of *Alstonea boonei* (900 mm × 1200 mm × 3600 mm) used in this study was sourced from a local sawmill in Akure, Ondo State. The lumbers were air-dried to approximately 20% moisture content and then machined into the required dimensions for testing dimensional stability and static bending strength in the direction parallel to grain with a circular saw. Twenty seven defect-free specimens of 20 mm × 20 mm × 10 mm (length × breadth × thickness) were prepared for the evaluation of dimensional stability.
The specimens were oven dried at 105°C until constant weight was achieved; then cooled in desiccator over silica gel. Thereafter, the weights and dimensions of the specimens were measured.

Thermal Modification Process

The heat treatment was conducted in a closed process vessel, a Muffle furnace, and a temperature controlled heating unit. The conditioned specimens (at constant moisture content of 12%) were thermally treated at temperatures of 125, 150 and 175°C for the duration of 30, 60 and 120 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, and their weights and dimensions were determined after cooling in a dessicator over silica gel to account for the weight change. The percentage weight loss, PWL, was determined using Equation 1.

$$\mathrm{PWL}(\%) = \left(\frac{W_o - W_t}{W_o}\right) \times 100 \qquad (1)$$

Where W_o (g) = the oven-dry weight of specimens before the treatment and W_t (g) = the oven-dry weight of specimens after the treatment.

Physical Properties Test

Dimensional Stability Tests

Modified and untreated wood samples were submerged in distilled water in a stainless steel container. A metal screen were placed over the samples to hold them approximately 2.5 cm below the surface but were not imparted by the load on them. Water absorption and thickness swelling were assessed after 2, 48 and 168 hrs of soaking in water. Mass and thickness measurements were recorded.
From the measurement of the dimensions and weights of the specimens, the swelling coefficient or volumetric swelling coefficient (S) was calculated using equations 2.

$$\mathrm{S}\ (\%) = \left(\frac{V_{wet} - V_{dry}}{V_{dry}}\right) \times 100 \qquad (2)$$

Where: V_{wet} = Volume of the samples after soaking in water and V_{dry} = Volume of the same sample after oven drying.

Mechanical Properties Tests

To assess the effect of thermal treatment on mechanical properties, static bending strength, three points flexural tests were performed on both control and modified samples in accordance with ASTM 143 standard (2009). The dimensions of wood samples for the test were 20 mm × 20 mm × 300 mm. Three replicates were tested for each treated wood sample on Tensometer load cell. Data are collected and processed using statistical package for social sciences (SPSS) software in which MOR and MOE were calculated by the software Equations 3 and 4 were used to estimate MOR and MOE respectively

$$MOE = \frac{PL^3}{4ywh^3} \qquad (3)$$

$$MOR = \frac{3PL}{2wh^2} \quad (4)$$

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.

Data Analyses method

Data obtained in the study were analyzed using 2x2x3 factorial experiment in Completely Randomized Design (CRD). Test of significance of the different treatment variables were estimated. Treatment means were separated using the Duncan's Multiple Range Test at $\alpha = 0.05$.

Results

Physical properties of thermally treated and untreated *Alstonia boonei*

Table1: Water Absorption and Volumetric swelling of thermally modified *Alstonia boonei* wood

Treatment temperature (°C)	Treatment time (min)	Volumetric swelling coefficients (%)		
		2hour	48hours	168 hours
Control	-	11.76±1.1	14.70±1.09	14.64±0.94
125	30	6.67±0.62	11.47±0.6	20.95±1.52
	60	10.13±1.66	11.01±0.6	15.23±3.54
	120	7.36±1.13	10.49±0.60	17.27±2.71
150°C	30	6.29±0.61	10.79±0.99	12.92±0.64
	60	7.59±0.6	12.99±0.5	22.50±1.6
	120	3.72±1.23	9.40±0.5	14.80±0.78
175°C	30	5.23±0.6	12.83±1.15	23.13±2.71
	60	6.52±3.09	13.67±2.18	19.48±3.40
	120	4.99±1.5	9.51±1.05	12.37±1.17

Values are mean± standard deviation

In order to statistically evaluate the reduction in selling under different treatment and experimental variations, Duncan multiple comparison range test (DMRT) was used in the analysis. Table 2 represents the volumetric swelling for different hours of soaking, and DMRT result. It shows that the volumetric swelling for different soaking hours were significantly reduced after the thermal treatment at any temperature/time ($p<0.05$).

Table 2: Comparison of Swelling under the different Temperature and Times regimes

Temperature	Time	2hours		48hours		168hours	
Control	0	11.76^{a}	11.76a	14.70^{a}	14.69a	14.64^{a}	14.64a
125	30	8.12^{b}	6.13b	12.00^{b}	11.69bc	14.74^{a}	19.0a
150	60	5.6^{b}	8.09b	11.06^{b}	12.55ab	18.81^{a}	19.06a
175	120	5.58^{b}	5.36b	10.99^{b}	9.60c	18.33^{a}	14.81a

The degrees of significance are represented by letters in superscript.

Table 3: Mean MOE (N/mm^2) and MOR (N/mm^2) under different temperatures and time regimes.

Treatment	MOE(N/mm^2)	MOR(N/mm^2)
CONTROL	725365.2 ±80916.18	129.5 ±26.42
125°C for 30min	857275.1 ±281689	150.5 ±16.04
125°C for 60min	666270.3 ±100524.54	122.5 ±26.42
125°C for 120min	706327.4 ±159827.06	150.5 ±21.86
150°C for 30min	844620.5 ±205881.37	157.5 ±10.5
150°C for 60min	757785.6 ±54648.46	101.5 ±21.86
150°C for 120min	5104058 ±1408160	875 .0±121.24
175°C for 30min	594000 ±54375.05	119 .0±12.12
175°C for 60min	619056.7 ±266165.2	105 .0±21.00
175°C for 120min	733438.5 ±151171.85	129.5 ±16.04

Table 4: Analysis of Variance of Modulus of Elasticity of thermally modified wood and the untreated wood

Source	Sum of Squares	Df	Mean Square	F	Sig.
Temp	1.43E+13	2	7.13E+12	31.72	0.00*
Time	1.28E+13	2	6.39E+12	28.45	0.00*
Temp * time	2.44E+13	4	6.09E+12	27.08	0.00*
Error	4.50E+12	21	2.25E+11		
Total	**5.65E+13**	**29**			

***Values are significant ($p < 0.05$)**

Table 5: Analysis of variance of Modulus of rupture of thermally modified and untreated wood

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Temp	372963.5	2	186481.8	102.099	0.00*
Time	407288	2	203644	111.50	0.00*
Temp * time	711431	4	177857.8	97.38	0.00*
Error	36529.5	21	1826.475		
Total	**1546738**	**29**			

***Values are significant ($p < 0.05$).**

Discussion

Swelling Properties

As a function of temperature and time, Table 1 represents the result of the volumetric swelling test of the thermally treated wood. The absorption for 2 hours of soaking ranged between 37.49 ± 2.4% and 40.53± 6.45%, 30.67± 0.8% and 42.16± 3.0% and 29.68 ± 2.90% and 45.99±3.55% for 125°C, 150°C and 175°C respectively. The absorption for control is 31.46± 3.42%. The percentage water absorption values of thermally modified wood samples were slightly different from the average value recorded for untreated samples (control sample) (Table1). As the treatment temperature increased from 125°C to 175°C, the volumetric swelling were found to reduce for all the water saturated treated samples for the duration of the soaking (Table 1).

The changes in chemical composition mostly affect the sorption relations between wood and water.Wood absorbs less water and swelling of wood is reduced due to less water absorption. The hydrophilicity of thermally treated wood sample was reduced by cleavage that occurred in the hemicelluloses and structural modification impacted by lignin and the decrease in the number of hydroxyl groups in the primary and secondary wood cells (Akgul *et al*., 2007). As reported by Ahmed and Moren (2012), macro pores in the cell lumen are blocked by water repellant compounds which are essentially hydrophobic substances. As a result, the resulting chemical and anatomical changes that occurred during thermal modification result in lower swelling effect. Ahmed and Moren (2012) In their study, it was confirmed that when higher temperature or longer time were used in treatment, more hemicelluoses were broken down resulting in lower swelling percentages. Swelling reduction of the heat treated wood were lower compared to untreated wood, indicating that swelling in different temperature regime decresed during heat treatment process. According to Jamsal *et al* 1999, Gunduz *et al* 2010; Gunduz and Aydemir 2009, it is well known that heat treatment significantly reduces tangential and radial swelling to very low values.

Mechanical Properties of Thermally modified *Alstonia boonei* wood

Modulus of Rupture

Table 3 presents the mean values and standard deviation of MOE and MOR under the different temperatures and times. MOR reflects the maximum load carrying capacity of a member in bending and is proportional to maximum moment or force borne by the specimen. The variation in the MOR of thermally treated *Alstonia boonei* ranged from 129.5 N/mm^2 (175° C for 120 min) to 150.5 N/mm^2 (125° C for 30 min). Some factors like atmosphere, temperature, duration, rate of heating, species, weight and dimensions of the samples are responsible for the difference in MOR of the heat treated and untreated wood as reported by Vernois (2000) and Kocaefe *et al*., (2008). The heating temperature duration and other factors mentioned above are responsible for the changes in the MOR of *Alstonia boonei* thermally modified wood.

Modulus of Elasticity

Modulus of elasticity (MOE) implies that deformations produced by low stress are completely recoverable after loads are removed. The MOE of heat treated wood samples varied from 5.94 x 10^5 N/mm^2 (175° C for 30 min) to 5.10 x 10^{6}N/mm^2 (150°C for 120 min). This agrees with the finding of Janusz *et al* (2013). In their study on effect of thermal modification on selected physical properties of wood of Scots pine recorded that greater changes were observed when modification conducted at high temperature and the strength of the wood decrease gradually.

Statistically, the change in MOE of the treated samples was significantly different to the untreated samples (Table4). It was also revealed in table 5 that there was significant difference in the treatment and the heating temperature which showed that the treated wood has been altered compared to that that of the control. This indicate that as the temperature increase with time, the strength properties (MOR) of the wood decreases.The hemicelluloses and and the lignin binding the cellwall of the wood together has been altered. The bending strength decreased 844620.5N/mm^2 (175^{o}C for 30minutes) to 594000N/mm2 (175^{o}C for 30minutes) drastically with high treatment temperature as it shown in table 3.

In the studies of Finnish ThermoWood on pine wood treated at temperatures of 100, 120, 140, 160, 180, 200, 220, and 240°C, it was reported that heat treatment did not significantly changed the MOE. Similar observation was made by Rapp and Sailer (2000) with the oil heat treatment and dry-air heat treatment of pine (*Pinus sylvestris* L.) treated at 180, 200, and 220°C.It was deduced from this finding that the MOE of the wood was significantly changed and this is due to the fact that the chemical components inside the wood has been altered and the different in wood species. Therefore, this research work disagrees with finding by Rapp and Sailer (2000). This research agrees with the finding of janusz *et al* (2013) which reported that compressive strength dependence on density, the changes in Elasticity of modulus are the highest when the heating at 200^{o}C is applied for 10hours.

Color changes

It was virtually observed that there was a colour change on the surface of the samples subjected to thermal modification. The color of thermally treated *Alstonia boonei wood* varied from pale creamy white to brown at 150ºC at 30, 60 and 120 minutes to very dark brown at the 175°C and for 120 min. The color of untreated control samples was creamy white to yellowish brown. This color change in wood can be attributed to some chemical reactions that took place during heat process. During the thermal modification, aldehydes and phenols may have been formed from degraded carbohydrates, and this could be responsible for the formation of coloured compounds after chemical reactions. This observation is similar to the study reported by McDonald *et al*. (2000). For a given treatment time, the degree of color change was greater for specimens treated at 175°C than for specimens treated at 125 °C.

Similarly, specimens treated at a given temperature of 175°C for 120 min were darker than those treated at the same temperature for 30 minutes. However, the treatment temperature has a more profound influence than the treatment time according to Mitsui *et al*. (2004). According to Sundqvist (2002), the changes in color of thermally modified wood are attributed to oxidative changes, which predominate over hydrolysis reactions. The effect of extractives in the colour of heat-treated wood showed that unextracted and acetone extracted samples had different/varying color (Sundqvist 2002). He concluded that both extractives and structural components (hemicelluloses and/or lignin) took part in colour change of heat-treated wood. Also, the color of the wood becomes darker due to the thermal degradation of lignin.

Conclusion

The thermal modification of *Alstonia boonei wood* affected its mechanical properties. The extent of modification varied with temperature and duration of treatment. The demerit of thermal modification according to this research is that there is significant reduction in the mechanical properties of the wood most especially the toughness under a long period of heat treatment at 175°C. The thermal modification improves the level at which the wood can rupture at heat treatment of 150°C for 120mins.

The colour of the wood changed from creamy white to dark brown with increasing treatment temperature and duration. The hygroscopicity and dimensional stability improved as measured by reduced water absorption and volumetric swelling. Therefore, the thermally treated *Alstonia boonei* can be used in wood siding, ship decks and garden furniture. Based on the findings of this research, (i.e. the effect of heat on physical properties of *Alstonia boonei)*, it is evidenced that heat treated wood has greater dimensional stability than the untreated wood.

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