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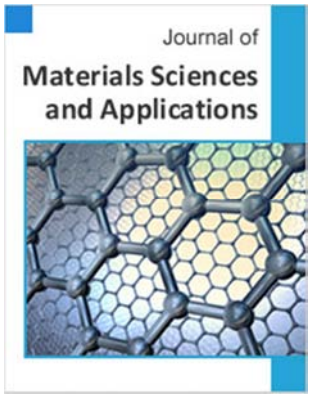

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Impact of Heat Treatment on Physico-Mechanical Properties of Thermally Modified *Anthocleistha djalonensis* Wood

Iyiola Ebenezer Adeyemi*, Olufemi Babatola, Owoyemi Jacob Mayowa, Fuwape Joseph Adeola

Department of Forestry and Wood Technology, School of Agriculture and Agricultural Technology, Federal University of Technology, Akure, Nigeria

Email address

eaiyiola@futa.edu.ng (I. E. Adeyemi), olufemibabatola@yahoo.com (O. Babatola), jacobmayowa@yahoo.com (O. J. Mayowa), joefuwape@yahoo.com (F. J. Adeola)

*Corresponding author

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Abstract

Thermal modification at relatively high temperatures is an effective method of improving the dimensional stability and mechanical properties of wood. This study was carried out to investigate the impact of heat treatment on the physico-mechanical properties of thermally modified *Anthocleistha djalonensis* wood. Thirty nine defect-free specimens of dimensions 20 mm × 20 mm × 60 mm were prepared for dimensional stability and compression tests. For the evaluation of static bending strength, thirty nine specimens of dimensions 20 mm × 20 mm x 300 mm were also prepared. After drying to a constant moisture content of 12%, samples were thermally treated at temperatures of 120, 140, 160 and 180°C for 60, 90 and 120 minutes duration respectively in the furnace. The mean values of the density showed a significant reduction as a function of treatment condition while an increase of the modulus of elasticity during the bending test was noticed. The minimum MOE value of samples was 3266 N/mm^2 at 120°C for 1h while the maximum MOE of samples was 3908 N/mm^2 at 160°C for 2h, whereas the compressive strength parallel to the fibre increased after heat treatment. It was observed that thermal modification improved dimensional stability and mechanical properties of the wood. Physical properties generally decreased with increasing temperature intensity.

1. Introduction

Wood has been preferred for residential construction since ancient times because of its natural beauty and excellent properties like high specific strength, heat insulation, and ease of handling and processing [1]. But, wood is easily degraded by environmental agents, including fire, biological organisms, water, and light, than many man-made materials [2]. Preservative treatment is effective and can extend the service life of wood and wood products [3]. However, faced with increasing environmental pressure, worldwide wood manufactures started to gradually decrease the amount chemicals used in wood treatment and are looking for alternative ways of enhancing wood properties. Heat treatment of wood at high temperature seems to be an eco-friendly and viable alternative to using chemicals [4]. The maximum temperature during heat treatment varies from 180 to 280°C and from 15 minutes to 24 hours depending on the process,

wood species, sample size, moisture content of the wood and desirable mechanical properties which affect the dimensional stability of the final product [5]. During heat treatment of lumber at temperatures around 200°C large changes in the wood properties take place. Heat treated wood exhibits better dimensional stability, better resistance to biodegradation, lower equilibrium moisture content and a change in color. Heat- treated wood has been commercialized for outdoor applications such as paneling, garden furniture and decking.

However, it is well known that distinct losses in mechanical properties can occur [6]. Cellulose, hemicelluloses and lignin are the main structural elements of wood and heat treatment involves degradation of these components to different levels. While the hemicelluloses are the component that degrades to the highest extent (Finnish Thermowood Association 2003), lignin that holds the wood cell is the least heat sensitive component of the wood [7]. Therefore, heat treated wood has a higher percentage of lignin than normal wood [4]. When wood is heat treated the color of wood changes acquiring a darker tonality which is often caused by the formation of color degradation produced from hemicelluloses [6, 8]. The color of wood is important from the aesthetic viewpoint for the consumers. Depending on cultural factors and income level, wood products may sell more solely due to their color. Heat treatment is an inexpensive alternative to darken wood from temperate regions to imitate more expensive species [9]. Wood modification is responsible environmental way of using of wood, preserving natural resources and promote the use of one of the most important renewable resources.

Therefore, the objective of this study was to investigate the effect of heat treatment on the physical and mechanical properties of thermally modified *Anthocleistha djalonensis* wood to make it suitable for use in a humid environment.

2. Methods

The samples for this study were obtained from Federal University of Technology in Akure, Ondo State, Nigeria. They were machined into the required dimensions in the direction parallel to grain with a circular saw. Thirty nine defect-free specimens of dimensions 20 mm × 20 mm × 60 mm were prepared for the determination of dimensional stability and compressive strength. For the evaluation of static bending strength (modulus of rupture and modulus of elasticity), thirty nine pieces of 20 mm × 20 mm x 300 mm were prepared. Another thirty- nine specimens of dimensions 30 mm x 30 mm x 25 mm were prepared for hardness test. In total, 117 samples were prepared for the tests. They were oven dried at 103 ± 2°C until constant weight is achieved; then cooled in desiccators over silica gel. Weights of the samples were taken to determine the moisture content of freshly felled *Anthocleistha djalonensis* wood using:

$$MC(\%) = \frac{\text{wet weight} - \text{oven dry weight}}{\text{oven dry weight}} \quad (1)$$

2.1. Thermal Modification Process

The heat treatments were conducted in a closed heating chamber. The samples (at constant moisture content (m.c.) of 12%) were thermally treated at temperatures of 120, 140,160 and 180^{o}C for 60, 90 and 120 mins respectively. 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 to prevent weight change. The weight loss (WL), was determined using eq (2):

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

Where: W_o (g) is the oven-dry weight of specimens before the treatment; W_t is the weight of samples after thermal treatment.

2.2. Dimensional Stability Tests

Modified and untreated wood samples were submerged in distilled water in a stainless steel container. A metal screen was placed over the samples to hold them approximately 2.5 cm below the surface but did not impart load on them. Mass and volume measurements were assessed after 24, 48 and 72 hrs of soaking to determine the followings:

Water absorption:

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

Where:

W_{wet} = Weight of the samples after soaking in water

V_{dry} = Weight of the oven dried samples.

Void volume:

$$\text{Void volume} = 1 - \frac{\text{Dry Specific gravity}}{1.5} \text{ X } 100\% \quad (4)$$

Volumetric swelling:

$$S\ (\%) = \left(\frac{V_{Wet} - V_{dry}}{V_{dry}}\right) \times 100 \quad (5)$$

Where:

S% = Volumetric swelling

V_{wet} = Volume of the samples after soaking in water

V_{dry} = Volume of the same sample after oven drying.

Density Determination

The mass per unit volume of the samples were calculated using the formulae below

$$\rho = \frac{M}{V} (Kg/m^3) \quad (6)$$

Where ρ = Density

M = Mass (kg)

V = Volume (m^3)

Figure 1. *Taking dimension of the wood after modification.*

Figure 2. *Modified wood samples inside dessicator.*

2.3. Mechanical Properties Tests

2.3.1. Static Bending

For evaluation of static bending strength, three-point 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 carried out 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 Bluehill v2 software (Instron) in which MOR, MOE and toughness were calculated:

Modulus of elasticity:

$$\text{MOE} = \frac{\text{PL}^3}{4\text{ywh}^3}\ (\text{N/mm}^2) \qquad (7)$$

Modulus of rupture:

$$\text{MOR} = \frac{3\text{PL}}{2\text{wh}^2}\ (\text{N/mm}^2) \qquad (8)$$

Where:

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

2.3.2. Determination of Maximum Compressive Strength (MCS) Parallel to Grain

The ability of a material to resist a crushing force or stress applied on the body. Test sample size of 20mm x 20mm x 60mm samples were used.

The values obtained were used to calculate the compressive strength using the equation below:

$$\partial_c = \text{P N/mm}^2 \qquad (9)$$

bd

∂_c = Maximum Compressive Strength in N/mm^2

b = width in mm

d = depth in mm

P = Load in Newton

3. Results and Discussion

The mean values for the physical properties of thermally modified *Anthocleistha djalonensis* wood presented in Table1 showed that moisture content of the treated wood samples decreased with temperature increase for very short and long treatment period. Table 1. The values ranged from 4.51% to 3.52% for 120°C at 1h and 180°C respectively at 2h compared to control 8.91%. The moisture content reduced as the temperature increased with time. These results are generally in agreement with the researchers that for beech wood treated at temperatures between 200-260°C and conditioned at similar relative humidities (66% and 86%) for eucalypt wood that increase in temperature cause reduction in moisture content because of the heat causing the water to move from higher concentration to lower concentration after which evaporation takes place [5, 10]

The reduction in moisture content is due to several factors. The degradation of hemicelluloses, which are the most hygroscopic structural compounds, plays an important role but the degradation of the amorphous regions of cellulose and the cross-linking reactions also contribute to the decrease in moisture content as reported by several authors [11, 12, 13]. The hemicelluloses content decreased 17.2% and 10.4% in relation to initial moisture content at about 3% weight loss for a treatment in air and in steam environment respectively [14]. Heat treatment results in a decrease of density. The value ranges from 446 kg/m^3 to 436 kg/m^3 for 120°C at 1h and 180°C at 2h compared to control 553 Kg/m^3. It shows from the result that as the temperature increases with time, the density decrease. The decrease of the density is caused by lower moisture content, evaporation of extractives during heat treatment, degradation of wood components, especially the hemicelluloses and evaporation and degradation of products [15]. This trend was observed for the wood species throughout the period. Heat treatment plays an important role on the change of dry density and specific volumetric dilatation of treated wood. It can be seen that after treatment the samples suffered weight loss and volume shrinkage, and therefore densities of heat-treated samples were less than untreated samples. Weight loss increased with the treatment time and with the temperature as shown in table 1 and the value ranges 3.39% to 6.12% for 120 at 1h and 180 at 2h respectively. Different mass loss was obtained with different temperatures, depending on the treatment time (table 1). The rate of weight loss was lower in the beginning of the

treatment and increasing for longer treatments in this study. The higher rate of weight loss was due to the thermal degradation of the more susceptible compounds, mainly hemicelluloses but also to the volatilization of some extractives [16].

Similar results for the degradation rate were also reported for the heat treatment of other species like cedar [17].

Table 1. *Mean values of Physical properties of thermally modified Anthocleistha djalonensis.*

S/N	Treatment(°C)	Time (min)	Density (Kg/m^3)	M.C(%)	WLoss (%)	Void volume (%)	Porosity
1	Control	0	553±43.08	8.91±3.91	-	0.96±0.001	0.03±0.005
2a	120	60	446±26.36	4.51±0.55	3.39±0.52	0.97±0.001	0.024±0.002
b	120	90	453±14.88	4.33±0.21	4.15±0.19	0.978±0.005	0.023±0.003
c	120	120	438±21.95	4.07±5.21	3.75±4.96	0.974±0.002	0.026±0.001
3a	140	60	453±16.03	4.43±0.48	4.24±0.44	0.978±0.03	0.023±0.003
b	140	90	449±39.49	4.42±0.12	4.23±0.11	0.979±0.004	0.02±0.004
c	140	120	463±9.58	4.32±0.26	4.14±0.24	0.978±0.0005	0.02±0.005
4a	160	60	441±13.20	4.96±0.18	4.73±0.16	0.979±0.002	0.02±0.002
b	160	90	438±5.85	4.82±0.28	8.31±6.31	0.979±0.001	0.021±0.001
c	160	120	443±19.48	4.79±0.58	4.58±0.53	0.98±0.001	0.02±0.001
5a	180	60	469±17.82	4.68±3.72	3.47±3.55	0.977±0.004	0.023±0.004
b	180	90	448±8.21	3.91±0.23	5.58±0.21	0.979±0.0006	0.03±0.001
c	180	120	436±63.88	3.52±0.13	6.12±0.12	0.979±0.002	0.021±0.002

From the results of volumetric swelling (S), (Table 2), The mean values of the thermally treated *Anthocleistha djalonensis* wood samples for volumetric swelling ranged from the highest, 4.43% (180°C at 90 min) to the lowest, 13.60% (120°C at 90 min) soaked in water for 24hrs. Relative to the untreated wood samples (S = 32.36%), The mean values of the thermally treated *Anthocleistha djalonensis,*wood samples for volumetric swelling ranged from the highest, 18.69% (180°C at 60 min) to the lowest, 6.77% (160°C at 120 min) soaked in water for 48hrs. Relative to the untreated wood samples (S = 27.53%). The mean values of the thermally treated *Anthocleistha djalonensis* wood samples for volumetric swelling ranged from the highest, 17.29% (180°C at 60 min) to the lowest, 9.72% (160°C at 60 min) soaked in water for 72hrs. Relative to the untreated wood samples (S = 31.05%). The mean values were lower for heat-treated wood compared to control specimens. In general, the S of treated samples decreased with increased treatment temperature and duration. The reason of the difference between treated samples and the untreated may be that in cellulose amorphous region the hydroxyl groups on the cellulose macromolecular chains and the hydroxyl groups of lignin are partly combined with each other that results in getting rid of molecule of water to form -O- structure. Therefore, the total amount of hydroxyl groups is decreasing significantly and reducing the capacity of water absorption to improve dimensional stability of heat-treated samples [5]. The decrease in volumetric swelling and increase in dimensional stability of heat treated wood is attributed to a decrease in moisture sorption [5].

It is also a very interesting finding that treated specimens have a significant sharp reduction in moisture uptake when the heat treatment conditions were at 160°C at 120 min, 180°C at 90 min and 180°C at 120 min. The reason for material losses in the cell lumen and hemicelluloses degradation was due to high applied temperature [18]. It is known that the weight of wood material and its swelling decrease when heat treatment is applied lowering water uptake in the wood cell wall because of the decrease of the amount of hydroxyl groups in the wood. As a consequence of the reduced number of hydroxyl groups, the swelling and shrinking were lower with the same proportion corresponding to their anti-swelling efficiencies.

Swelling reductions of heat-treated wood were lower compared to untreated wood, indicating that swelling in different sections decreased during heat treatment process. It is well known that heat treatment significantly reduces tangential and radial swelling to very low values [19, 20]. Wood subjected to abrupt high temperatures looses its capacity to reabsorb water on the contrary to the hydrophilic behavior of the conventionally dried wood [21].

Table 2. *Mean values of Physical properties of thermally modified Anthocleistha djalonensis.*

Treatment(°C)	Time (min)	WA (%) (24h)	WA (%) (48h)	WA (%) (72h)	VS (%) (24h)	VS (%) (48h)	VS (%) (72h)
Control	0	59.65±6.95	77.29±8.53	86.2±10.07	32.36±3.88	27.53±0.40	31.05±8.80
120	60	58.72±9.04	80.89±9.35	93.05±9.85	12.06±0.98	11.59±2.76	15.35±1.50
120	90	60.72±3.55	81.06±6.78	97.57±7.80	13.60±0.67	10.67±0.92	14.05±9.84
120	120	64.08±9.44	86.3±10.05	99.4±11.78	11.36±0.47	11.91±3.08	14.19±3.00
140	60	60.23±3.18	82.53±2.82	93.46±4.05	10.80±0.77	11.33±1.89	13.69±3.04
140	90	61.7±13.87	78.6±19.99	84.2±21.58	10.49±1.18	6.93±4.05	11.81±0.69
140	120	56.78±4.39	77.88±1.18	84.82±7.97	10.47±1.41	9.12±0.52	13.06±0.78
160	60	67.91±5.35	85.39±5.50	98.43±7.37	11.74±1.79	9.07±3.92	9.72±4.49
160	90	69.66±9.39	90.98±7.85	104.4±7.22	10.63±3.44	7.89±1.94	10.26±4.09
160	120	67.14±6.16	85.69±7.99	97.85±9.11	9.89±3.47	6.77±4.22	14.41±3.78
180	60	59.26±5.12	79.24±5.13	86.57±5.39	11.02±2.64	18.69±3.29	17.29±1.72
180	90	54.9±45.09	80.39±4.38	85.05±1.22	4.43±1.86	9.93±1.41	10.23±2.45
180	120	59.85±4.39	76.2±13.47	89.79±11.5	6.39±1.79	10.59±0.80	10.04±1.03

The mean values for the mechanical properties of thermally modified *Anthocleistha djalonensis* wood are presented in Table 3

Table 3. *Mean values of Mechanical properties of thermally modified Anthocleistha djalonensis.*

S/N	Treatment(°C)	Time (min)	MOR (N/mm^2)	MOE (N/mm^2)	Compression test(N/mm^2)	HRF Hardness test
1	Control	0	66.70±9.33	3828±347.28	39.54±5.10	74.80
2a	120	60	79.29±2.83	3266±216.28	44.62±1.75	69.07
b	120	90	69.76±8.63	3555±590.73	43.08±4.72	73.63
c	120	120	76.43±7.42	3380±751.49	41.95±5.16	76.57
3a	140	60	74.94±4.38	3387±324.15	46.31±2.57	69.87
b	140	90	81.42±3.96	3656±257.85	48.34±0.15	74.50
c	140	120	83.65±3.10	3683±269.70	50.94±0.06	72.67
4a	160	60	68.7±14.69	3890±386.65	48.78±3.69	76.40
b	160	90	78.98±7.57	3886±294.24	48.56±2.31	73.07
c	160	120	80.66±5.13	3908±628.92	48.14±2.46	74.37
5a	180	60	67.33±4.79	3632±329.53	46.89±1.55	88.13
b	180	90	74.30±8.51	3833±875.11	48.43±2.84	85.37
c	180	120	64.53±2.80	3705±586.28	49.70±0.14	81.80

Table 4. *Analysis of Variance of Modulus of Rupture of Anthocleistha djalonensis.*

Source	Type III Sum of Squares	Df	Mean Square	F	Sig.
Temperature	591.298	3	197.099	3.813	0.022*
Time	106.686	2	53.343	1.032	0.370 ^{ns}
Temp * Time	562.586	6	93.764	1.814	0.135 ^{ns}
Error	1344.137	27	51.698		
Total	2795.497	38			

*significant at (P<0.05) probability level; ns=not significant at (P>0.05) probability level

Table 5. *Analysis of Variance of Modulus of Elasticity of thermally modified Anthocleistha djalonensis.*

Source	Type III Sum of Squares	Df	Mean Square	F	Sig.
Temperature	1195946.894	3	398648.965	1.632	0.206 ^{ns}
Time	221136.498	2	110568.249	0.453	0.641 ^{ns}
Temp * Time	128921.340	6	21486.890	0.088	0.997 ^{ns}
Error	6350330.111	27	244243.466		
Total	7985434.718	38			

*significant at (P<0.05) probability level; ns=not significant at (P>0.05) probability level

The variations in mechanical properties of heat-treated wood were showed in Table 3 at different temperatures and durations. The MOE value for *Anthocleistha djalonensis* ranged from 3908 N/mm^2 (160°C at 2h) to 3266 N/mm^2 (120°C at 1h). Compared with control samples 3828 N/mm^2, MOE of treated samples increased. When the treatment temperature increased, the MOE increased, simultaneously. Statistically, the results showed that the temperature of the wood species were significantly different while both time and interaction between the temperature and time were not significant at 5% level of probability. The minimum MOE value of samples was 3266 N/mm^{2}at 120°C for 1h while the maximum MOE of samples was 3908 N/mm^2 at 160°C for 2h (Table 3) and later decreased as the temperature increases. There was a significant increase between control samples and the heat treated samples. But this increase has continued to decrease from temperature 160°C to 180°C. Similarly, there was an increase of 17% in fir wood [22]. The change of modulus of elasticity value was insignificant [23]. Higher temperature and longer treatment time had positive effect on the MOE because of the partially crystal micro fibrils nature of wood and the large scale hemicelluloses and lignin [24]. When wood is modified above a certain temperature, most of the amorphous polymeric components may convert their glassy structures to elastic. At the conversion temperature from glassy structure to elastic, particular polymers have enough energy that reduces the mutual gravitational forces. Thus the wood polymers can be converted to elastic or mostly plastic construction.

Recognizable increase in the modulus of elasticity might be based on the increase of the relative cellulose content after heat treatment, although the hemicelluloses may have degraded. Lower moisture content of treated wood than control also effects the modulus of elasticity [15]. It is observed the values of both wood portions (sapwood and heartwood) for modulus of rupture (MOR) increased and later decreased through thermally modified. The untreated wood still obtained the highest strength values compared to treated wood. The strength properties of wood usually decrease with increasing temperature and increase with decreasing temperature [25]. For the thermally modified wood, the highest values of MOR of every portion were starting to decreased when the treatment temperature reached 180°C. This strength values respectively decreased when treated at 180°C at 2h for the treated species. From the results obtained, the values of MOR of treated wood at high

temperature showed a decrement with increasing different parameters. Reductions in mechanical strength properties were reported [26, 21, 22]. The variations in MOR among and within species can be explained by the decrease in maturity of wood and fibre length from the base to the top of the tree [27]. Wood treated at 180 to 200°C in the presence of moisture resulted in a large reduction in the resistance to MOR, MOE and compression strength [28]. Some researchers reported that at temperature over 200°C, MOE and MOR of wood reduced by up to 50% [29, 30, 31]. The increased treatment duration from 90 to 120 minutes also prolong the decreasing effect on strength. This testifies that a value of MOR for treated wood is influenced by treatment temperature and duration. The higher the temperature and the longer the treatment duration the lower is the strength value [32, 33, 34].

The Janka hardness results in different directions for untreated and treated wood specimens revealed that surface hardness of *Anthocleistha djalonensis* wood increased with increased temperature and duration. Hardness values in the longitudinal sections of wood treated at 120°C for 1 h were approximately 69.07. It can be seen from the result that the hardness values differ depending on the species. When the duration was increased for the same exposure temperature, hardness values in the different sections increased to a greater extent for 90minutes and 2 h durations than they did for the 1 hrs duration. The Janka hardness parallel to the grain increased after heat treatment. In general, the variation of the results of the different strength tests increased after heat treatment. This is in agreement with previous findings which also noted a significant increase in surface hardness after thermal-modification of wood [35, 36].

4. Conclusion

Based on the result from this finding, it can be concluded that thermal modification is effective to improve dimensional stability and mechanical properties of wood samples. Technological properties generally decreased with increasing heat-treatment intensity.

The modulus of Elasticity of the wood were greatly influenced and enhanced by temperature significantly but by time limited. The Modulus of Rupture of torrefied wood was affected by both time and temperature compared to the control.

Heat treatment revealed a clear effect on the mechanical properties of this wood species. The tensile strength parallel to the grain showed a rather large decrease, whereas the compressive strength parallel to the fibre increased after heat treatment. The bending strength, which is a combination of the tensile stress was lower after heat treatment. The bending strength showed a large decrease after heat treatment. An increase of the modulus of elasticity during the bending test was noticed after heat treatment. Changes and/or modifications of the main wood components appear to be involved in the effects of heat treatment on the mechanical properties.

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