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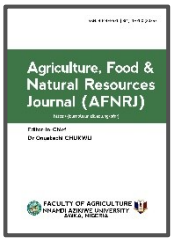

Original Article

Physical and mechanical properties of hydrothermally modified *Pinus caribaea* Morelet wood

OPEN ACCESS

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ABSTRACT

Dimensional changes in wood during service are a major threat to its longevity in service. This study therefore examined the effect of hydrothermal treatment on selected physical and strength properties of Pinus caribaea wood. Bolts were obtained from five Pinus caribaea trees at the tree's top, middle, and basal positions. Each of the bolts was processed into test dimensions and hydrothermally treated at 100°C, 120°C, and 140°C for 1 and 2 hours, while some sets were untreated. The treated and untreated wood were evaluated for their density, water absorption (WA), longitudinal, tangential, radial, and volumetric swellings (LS, TS, RS and VS, respectively), compression strength parallel (MCS//) and perpendicular to grain (MCS⊥). The data were subjected to analysis of variance at α= 0.05. The results reveal that the reduction in density was not significant after treatment, while the WA generally reduced after treatment, with the least observed at 100-1hr (96.25%). The LS was highest (0.58%) at 120-2hr and least (0.29%) at 140-1hr. The RS was significantly reduced to 4.78% at 120-2hr while TS had the significantly least value at 100-1hr (6.94%). The VS was significantly reduced to 12.17% at 140-2hr, while the highest value was found in the untreated samples (17.43%). The highest (53.68N/mm^2) and least (48.25N/mm^2) MCS// was observed in untreated wood and wood treated at 100-2hr, while the MCS⊥ was significantly reduced at 120-1hr (100.65N/mm^2). Hydrothermal treatment of P. caribaea at 100°C and 140°C for 1-2hours is capable of significantly enhancing its dimensional stability while preserving its mechanical integrity.
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KEYWORDS: *Anisotropic swelling, Carribean Pine, Dimensional stability, Hydrothermal treatment*

INTRODUCTION

Thermal treatment is a type of wood modification process which has widely gained recognition commercially in the wood industry (Hill *et al.* 2021). It involves the application of heat which alters the chemical structure of wood cell wall constituents (Candelier *et al.*, 2016; Källbom *et al.*, 2016) in order to suit some desired end uses. The concept of thermal treatment of wood emanated from the work of Tiemann in 1915 during his study on high temperature procedure of drying wood, which caused a decrease in the wood's equilibrium moisture content. Generally, wood is highly susceptible to degradation by biological and abiotic factors (Čermák *et al.*, 2015; Stirling *et al.*, 2017; Ali *et al.*, 2021), which limits their use in various technical applications. Efforts to address these limitations in wood have brought about the development of several wood property improvement techniques, one of which is the thermal modification of wood (Yan & Morrell, 2014).

Thermal treatment is known to be an environmentally friendly wood modification technique as it requires no chemical for the wood modification (Gao *et al.*, 2018; Ali *et al.*, 2021). During thermal treatment, changes such as extractive removal, hemicellulose degradation, decline in the wood's hydroxyl

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groups, alteration in the cellulose and lignin components of the wood (Boonstra & Tjeerdsma, 2006), changes in its pore characteristics (Ali *et al.*, 2021), accompanied by colour changes in the wood (Lengowski *et al*. 2021) usually occur, giving rise to an entirely "new" wood material. Thermal treatment can be done using various media such as air, water, nitrogen, and oils (Lee *et al*., 2018; Ali *et al*., 2021); with each medium impacting on the wood characteristics differently (Ali *et al.*, 2021). The efficacy of the process, however, is dependent on the process conditions such as the treatment temperature employed, duration of the treatment and the treatment media used (Ates *et al*., 2009; Freitas *et al.*, 2019; Ali *et al.*, 2021).

Hydrothermal treatment, which is a common type of thermal modification of wood, makes use of water or steam in altering wood characteristics at varying temperature ranges (Ali *et al.*, 2021). Among all other wood modification procedures, it is described as the most commercially innovative procedure for wood modification simply because water is the only substance employed during the process (Hill, 2006). Based on the temperature used for the modification treatment, Song *et al.* (2021) categorized hydrothermal treatment into three different classes: the ambient liquid-water treatment (25 - 100ºC), subcritical water treatment (100 - 374ºC), and supercritical water treatment (> 374ºC). Similar to the other thermal modification procedures, a reduction in wood strength is incurred, which is a general drawback of wood thermal modification (Ebadi *et al*., 2016; Rasdianah *et al.*, 2018). However, studies have shown that the extent of strength loss in the thermally treated wood is dependent on the temperature used and time of exposure (Ates *et al*., 2009). Thus, this research aims to investigate the effect of hydrothermal treatment at various subcritical temperatures and exposure time on the physico-mechanical properties of *Pinus caribaea* wood, which is known to be a dimensionally unstable wood (Adenaiya *et al.,* 2022).

MATERIALS AND METHOD

Sample Collection

Five defect-free *Pinus caribaea* trees of 31 years in age were harvested from Shasha Forest Reserve, Osun State, Nigeria. Bolts measuring 50 cm in length were gotten from the top (90%), middle (50%) and basal part (10%) of each of the tree's merchantable length. The bolts were processed into test dimensions of 60 mm x 20 mm x 20 mm and conditioned as described by Adenaiya *et al.* (2016).

Experimental Design

The experimental design used was a 3 x 4 x 2 Factorial experiment in a Completely Randomized Design, where the "3" represents the sampling height of the trees (Top, middle and Base), "4" represents the temperature levels (control, 100ºC, 120ºC and 140ºC), and "2" represents the time (1 hour and 2 hours).

Hydrothermal Treatment of Wood Samples

The conditioned wood samples were steam-treated in sets in an autoclave using three subcritical temperature regimes: 100ºC, 120ºC and 140ºC. Each set was treated for 1 hour and 2 hours. This was carried out by stacking the wood samples on a perforated metal plate in the autoclave to allow for the escape of steam from the boiling water below the steel plate in an air tight condition. Another set of wood samples which were untreated were used as the control. Subsequently after the treatment, the samples were oven-dried to constant weight in the oven at 103ºC.

Wood Density Estimation

The density of the control samples and hydrothermally treated wood samples were determined in accordance with ASTM D 2395-83 (1983) using the formula below:

$$D = \frac{M}{V}\ (Kg/m3) \qquad (1)$$

Where: D=Density, M = Weight of the wood, V = Volume of wood

Determination of Water Absorption

The treated wood samples and control samples were soaked in water in a container for 2 weeks until they were all saturated. After saturation, the weights of the samples were measured. The water absorption was estimated in accordance to the procedure adopted by Erakhrumen and Ogunsanwo (2009) as described below:

$$Water\ Absorption\ (\%) = \frac{W1-W0}{W0}\ x100 \qquad (2)$$

Where W_0 = Weight of treated wood before submerging in water, W_1 = Weight of treated wood after submerging in water

Determination of Dimensional Swelling

The radial, tangential and axial faces of the treated and control wood samples were identified and then coded as 'T', 'R' and 'L' depicting radial, tangential, and longitudinal planes respectively. The wood samples' dimensions in the oven-dried state and after saturation along these three planes were taken with the aid of a digital Vernier caliper and recorded. The percentage swelling along these planes was estimated using the method employed by Islam *et al.* (2012) which is given as:

$$Sw\ (\%) = \frac{Ds-Do}{Do} \times 100 \qquad (3)$$

Where: S_w (%) = Swelling (Either S_wR or S_wT or S_wL); D_s = Dimension at saturated condition; D_0 = Dimension before soaking.

Volumetric swelling (V_{sw}) was estimated in accordance with the method adopted by Modes *et al.* (2013) which is described below:

$$Vsw = SwR + SwT + SwL \qquad (4)$$

Where: SwR = Radial swelling; SwT = Tangential swelling; and SwL = Longitudinal swelling

Determination of Maximum Compressive Strength (MCS// and MCS ⊥)

The Maximum compressive strength tests were conducted at the Timber mechanics laboratory of the Forestry Research Institute of Nigeria, Jericho, Ibadan, Nigeria. The set of wood dimension used for tests were of modified dimensions of 2 cm x 2 cm x 6 cm. For the strength tests, three (3) trees were sampled and two samples were obtained from each axial positions of the trees. Both the Maximum Compressive Strength parallel (MCS//) and perpendicular (MCS⊥) to grain were determined from an Instron 3369 model Universal Testing Machine (UTM). The load after failure occurred was documented and the corresponding computer values were read from the machine.

Statistical Analysis

The experimental data was analysed using both descriptive and inferential analyses. The data obtained were analysed using Analysis of Variance (ANOVA). Duncan Multiple Range Test was used for post-hoc test where significant differences among treatment means were observed

RESULTS

Physical Properties

Density

Table 1 shows the mean density values for the untreated and thermally modified *P. caribaea* wood samples. The density for the control samples ranged from 538.40 kg/m^3 (middle) to 614.51 kg/m^3 (Top). Based on sampling height, the highest mean density of the treated samples (694.15 kg/m^3) was obtained from the basal wood, while the lowest density (552.15 kg/m^3) was obtained from the middle wood. There was a decrease in the density of the treated wood samples with increasing time, while the decline in the density of the wood with increasing temperature was inconsistent as reflected by the pooled means. Furthermore, none of the factors under consideration nor their interactions had a pronounced effect ($p>0.05$) on the wood density (Table 3).

Water Absorption

Table 1 shows the mean water absorption values for the untreated and thermally modified *P. caribaea* wood samples. The water absorption for the control samples increased from the top (90.96%) to the base (113.69%). Along the tree bole, the water absorption of the treated samples decreased from the top (107.88%) to the base (92.12%). Contrary to the density results, an increase in water absorption with increasing treatment time was observed as reflected in the pooled mean of the treated samples. However, the trend in water absorption with increasing temperature was inconsistent, but overall tended to reflect a reduction in water absorption with rising temperature. The statistical analysis also showed that the water absorption values before and after treatment were not significantly different ($p>0.05$) (Table 3).

Longitudinal Swelling

Table 1 shows the mean longitudinal swelling values for the untreated and thermally modified *P. caribaea* wood samples. The longitudinal swelling for the control samples was inconsistent along the bole, ranging from 0.39% (middle) to 0.48% (base). Axially, the longitudinal swelling of the treated samples decreased from the base (0.39%) to the top (0.36%). There was an increase in the longitudinal swelling of the treated wood samples with increasing time, while the longitudinal swelling varied inconsistently with increasing temperature, though the least longitudinal swelling was observed in the samples treated at 140°C (0.32%) as reflected by the pooled means. The ANOVA result showed that the factors under consideration and their interactions had no significant influence ($p>0.05$) on the longitudinal swelling of the wood (Table 3).

Radial Swelling

Table 2 shows the mean radial swelling values for the untreated and thermally modified *P. caribaea* wood samples. The radial swelling for the control samples increased from the top (5.81%) to the base (9.08%). Along the tree bole, the radial swelling of the treated samples was inconsistent, ranging between 4.95% (middle) to the base (6.36%). The radial swelling generally decreased with increasing treatment temperature and time as depicted by the pooled means of the treated samples. The ANOVA result indicated that the temperature had a profound effect ($p<0.05$) on the radial swelling of the wood, while the other factors and their interactions insignificantly influenced ($p>0.05$) the radial swelling of the wood (Table 2). The thermal treatment also significantly reduced the radial swelling ($p<0.05$) of the wood (Table 3).

Tangential Swelling

Table 2 shows the mean tangential swelling values for the untreated and thermally modified *P. caribaea* wood samples. The tangential swelling for the control samples decreased axially from the base (11.94%) to the top (8.93%). Based on sampling height, the highest tangential swelling of the treated samples was recorded for the basal wood (8.27%), while the least tangential swelling was obtained from the wood at the top (7.68%). The pattern of radial swelling with increasing treatment time was inconsistent, while it generally decreased with increasing temperature as reflected by the pooled means. Similar to the radial swelling, the temperature had a statistically significant effect ($p<0.05$) on the tangential swelling of the wood but the other treatment factors and their interactions had no marked effect ($p>0.05$) on the wood tangential swelling (Table 2). The thermal treatment considerably reduced the tangential swelling ($p<0.05$) of the wood (Table 3).

Volumetric swelling

Table 2 shows the mean volumetric swelling values for the untreated and thermally modified P. caribaea wood samples. The volumetric swelling for the control samples increased from the top (15.20%) to the base (21.50%). Axially, the volumetric swelling of the treated samples was inconsistent, ranging between 13.33% (middle) to the base (15.01%). Similar to the radial swelling, volumetric swelling generally decreased with increasing treatment time and temperature as depicted by the pooled means of the treated samples. The ANOVA result showed that the treatment temperature had a marked effect ($p<0.05$) on the volumetric swelling of the wood, while other treatment parameters under consideration in the study and their interactions had no significant influence ($p>0.05$) on the wood volumetric swelling (Table 3). There was a marked reduction in the volumetric swelling of the thermal treated wood ($p<0.05$).

Table 1: Mean Density, Water absorption and Longitudinal swelling values of untreated and thermally modified *Pinus caribaea* wood

Physical Properties	Temperature	Time	Sampling height			
			Top	Middle	Base	Mean
Density (kg/m3)	Control	-	614.51±139.69	538.40±141.15	582.54±151.73	578.49±142.80
		1hr	611.94±116.11	559.36±108.89	579.03±146.29	583.44±123.76
	100°C	2hrs	485.67±119.62	512.19±91.96	562.31±204.32	520.06±138.63
		Mean	548.81±117.87	535.78±100.42	570.62±175.31	551.75±131.20
		1hr	682.22±291.14	546.20±95.35	612.65±120.54	613.69±169.01
	120°C	2hrs	615.87±132.26	557.74±186.64	564.44±163.71	579.35±160.87
		Mean	649.05±211.70	551.97±141.00	579.55±142.13	596.52±164.94
		1hr	574.08±117.63	558.77±147.67	605.52±86.71	579.46±117.34
	140°C	2hrs	543.06±109.78	549.23±173.52	605.04±176.95	565.78±153.42
		Mean	558.57±113.71	554.00±160.60	605.28±131.82	572.63±135.38
		Pooled Mean	579.58±151.15	552.15±138.15	694.15±148.10	574.84±142.60
Water absorption (%)	Control	-	90.96±37.35	103.52±35.68	113.69±72.16	102.72±50.33
		1hr	92.34±21.76	99.19±19.69	97.23±33.50	96.35±24.93
	100°C	2hrs	131.25±32.53	116.25±37.84	89.04±25.40	112.18±31.92
		Mean	111.80±27.14	107.72±28.77	93.14±29.45	104.22±28.45
		1hr	100.76±71.16	98.54±19.11	85.58±14.49	94.96±34.92
	120° C	2hrs	105.57±47.14	93.25±20.33	99.94±28.16	99.59±31.88
		Mean	103.17±59.15	95.90±19.72	92.76±21.33	97.28±33.40
		1hr	100.03±31.08	99.21±20.59	96.32±21.31	98.61±24.32
	140°C	2hrs	112.27±29.24	97.16±22.42	88.02±26.00	99.15±25.89
		Mean	106.15±30.16	98.19±21.51	92.17±21.32	98.4±24.33
		Pooled Mean	107.88±39.34	98.88±23.66	92.12±23.28	100.76±37.01
Longitudinal swelling (%)	Control	-	0.46±0.49	0.39±0.40	0.48±0.58	0.44±0.48
		1hr	0.40±0.31	0.36±0.18	0.26±0.25	0.34±0.25
	100°C	2hrs	0.39±0.19	0.43±0.38	0.33±0.20	0.38±0.26
		Mean	0.40±0.25	0.40±0.28	0.30±0.23	0.37±0.25
		1hr	0.53±0.27	0.39±0.25	0.43±0.30	0.45±0.27
	120° C	2hrs	0.53±0.30	0.43±0.43	0.79±0.41	0.58±0.38
		Mean	0.43±0.29	0.41±0.34	0.61±0.36	0.48±0.55
		1hr	0.30±0.14	0.36±0.14	0.23±0.90	0.30±0.40
	140°C	2hrs	0.16±0.11	0.49±0.35	0.36±0.48	0.34±0.31
		Mean	0.23±0.13	0.43±0.25	0.30±0.69	0.32±0.36
		Pooled Mean	0.36±0.26	0.38±0.23	0.39±0.38	0.41±0.35

Values indicate the Mean±SD

Table 2: Mean Radial, Tangential, Volumetric swelling values of untreated and thermally modified *Pinus caribaea* wood

Physical Properties	Temperature	Time	Sampling height			
			Top	Middle	Base	Mean
Radial swelling (%)	Control	-	5.81±5.51	5.82±2.15	9.08±1.56	6.90±3.75
		1hr	5.63±1.57	5.51±2.19	5.70±2.90	5.61±2.22
	100°C	2hrs	5.57±1.73	5.07±0.88	4.11±2.60	4.91±1.74
		Mean	5.60±1.65	5.29±1.54	4.91±2.75	5.27±1.98
		1hr	5.44±2.18	4.54±3.46	6.69±1.71	5.56±2.45
	120°C	2hrs	5.01±3.19	4.28±2.94	5.04±2.05	4.78±2.72
		Mean	5.23±2.69	4.41±3.20	5.89±1.88	5.18±2.59
		1hr	5.44±1.11	4.35±2.54	5.50±0.94	5.10±1.53
	140°C	2hrs	5.02±1.04	4.89±2.34	5.50±2.04	5.14±1.81
		Mean	5.23±1.08	4.62±2.44	5.50±1.49	5.12±1.67
		Pooled Mean	5.45±2.09	4.95±2.30	6.36±1.82	5.62±2.69
Tangential Swelling (%)	Control	-	8.93±4.55	9.38±2.66	11.94±3.07	10.08±3.65
		1hr	6.87±1.71	7.45±2.71	6.50±2.71	6.94±2.38
	100°C	2hrs	7.45±2.83	6.27±1.45	7.18±4.39	7.00±2.89
		Mean	7.16±2.27	6.86±2.08	6.84±3.55	7.00±2.63
		1hr	6.48±5.68	9.62±5.33	8.11±2.13	8.07±4.38
	120° C	2hrs	9.42±3.66	8.46±4.27	6.32±2.01	8.07±3.31
		Mean	7.95±4.67	9.04±4.80	7.22±2.07	8.07±3.85
		1hr	7.19±2.26	6.73±2.17	7.24±0.90	7.05±1.78
	140°C	2hrs	6.19±1.67	7.25±2.12	6.65±1.43	6.70±1.74
		Mean	6.69±1.97	6.99±2.15	6.95±1.17	6.88±1.76
		Pooled Mean	7.68±2.56	8.00±3.15	8.27±2.40	8.00±3.33
Volumetric swelling (%)	Control	-	15.20±9.72	15.60±3.24	21.50±4.08	17.43±6.81
		1hr	12.90±3.00	13.32±4.52	12.47±5.46	12.90±4.33
	100°C	2hrs	13.42±4.41	11.76±1.99	11.61±5.77	12.26±4.06
		Mean	13.16±3.71	12.54±3.26	12.04±5.62	12.58±4.20
		1hr	12.45±7.20	14.55±8.27	15.50±3.62	14.17±6.36
	120° C	2hrs	14.96±5.50	13.16±5.89	12.15±4.05	13.42±5.15
		Mean	13.71±6.35	13.86±7.08	13.83±3.84	13.80±5.76
		1hr	12.92±3.05	11.44±4.44	12.51±2.88	12.29±3.46
	140°C	2hrs	11.37±2.42	12.63±4.51	12.73±2.25	12.24±3.06
		Mean	12.14±2.74	12.04±4.48	12.62±2.37	12.27±3.20
		Pooled Mean	13.50±4.19	13.33±4.56	15.01±3.72	14.02±5.43

Values indicate the Mean±SD

Table 3: Effect of hydrothermal treatment at different temperatures and time on physical properties of *Pinus caribaea* wood

Treatment condition	Density (Kg/m^3)	WA (%)	LS (%)	RS (%)	TS (%)	VS (%)
Control	578.49±142.80^a	102.72±50.33^a	0.44±0.48^{ab}	6.90±3.75^a	10.08±3.65^a	17.43±6.81^a
100-1h	583.44±117.72^a	96.25±23.99^a	0.34±0.24^{ab}	5.62±2.12^{abc}	6.94±2.28^b	12.90±4.13^c
100-2h	520.06±139.69^a	112.18±41.34^a	0.38±0.25^{ab}	4.92±1.84^{bc}	6.96±2.95^b	12.26±4.11^c
120-1h	613.69±185.13^a	94.96±40.74^a	0.45±0.26^{ab}	5.64±2.58^{abc}	8.07±4.52^{ab}	14.17±6.31^{abc}
120-2h	579.35±152.74^a	99.59±31.99^a	0.58±0.39^b	4.78±2.59^c	8.07±3.46^{ab}	13.42±4.97^{bc}
140-1h	579.46±112.86^a	98.52±23.01^a	0.29±0.13^a	5.10±1.66^{abc}	7.05±1.76^b	12.44±3.11^c
140-2h	565.78±147.73^a	99.15±26.23^a	0.33±0.35^{ab}	5.14±1.77^{abc}	6.69±1.69^b	12.17±3.19^c

Mean±SD values with different superscript within the same column are significantly different (p>0.05). WA - Water Absorption, LS – Longitudinal Swelling, TS – Tangential Swelling, RS – Radial Swelling and VS - Volumetric Swelling

Mechanical properties

Maximum Compressive Strength parallel to grain (MCS //)

As shown in Table 4, the MCS// for the control samples increased from base (48.12 N/mm^2) to top (57.23 N/mm^2). Based on sampling height, the highest mean MCS// of the treated samples (54.93 N/mm^2) was obtained from the middle wood, while the lowest MCS// (51.65 N/mm^2) was obtained from the basal wood. Generally, the MCS// of the treated wood samples decreased with increasing treatment temperature and time as reflected by the pooled means. The ANOVA result indicated that the factors under consideration in this study and their interactions had no significant influence (p>0.05) on the MCS// of the wood (Table 5 and Fig. 1a).

Maximum Compressive Strength Perpendicular to grain (MCS ⊥)

Table 4 shows the mean MCS⊥ values for the untreated and thermally modified *P. caribaea* wood samples. For the control samples, the MCS⊥ progressively decreased from the top (121.67 N/mm^2) to the base (117.67 N/mm^2) along the tree bole. After treatment however, the MCS⊥ of the wood appeared to increase from the top (121.65 N/mm^2) to the base (126.34 N/mm^2) axially. For the treatment temperature, the MCS⊥ tended to decrease with increasing temperature but a sudden spike occurred at 140^{0}C. For the duration of treatment on the other hand, it appeared to increase consistently with increasing treatment time. The ANOVA result showed that all the factors under consideration and their interactions (except for the interaction between sampling height and time) considerably influenced (p<0.05) the MCS⊥ of the wood (Table 5 and Fig. 1b). As also shown in Fig. 1, treatment of the wood at a temperature of 120°C significantly decreased the MCS⊥ of the wood.

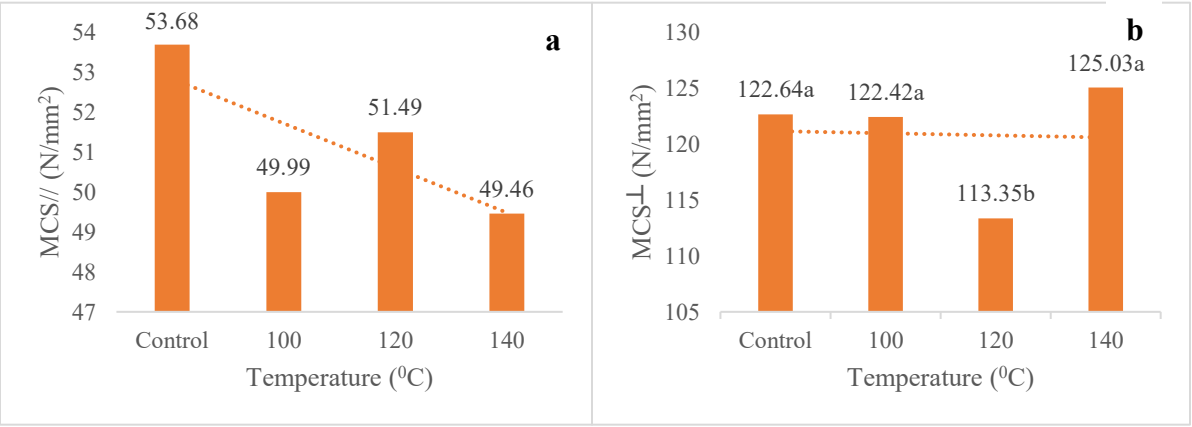

Figure 1: Effect of temperature on the (a) Maximum Compressive Strength parallel to grain of *Pinus caribaea* wood, and (b) Maximum Compressive Strength Perpendicular to grain of *Pinus caribaea* wood

Table 4: Maximum Compressive Strength// (N/mm²) mean values for thermally modified *Pinus caribaea* wood

Mechanical Properties	Temperature	Time	Sampling height			
			Top	Middle	Base	Mean
Maximum Compressive Strength parallel to grain (MCS //)	Control	-	57.23±1.17	55.70±19.18	48.12±17.49	53.68±14.68
	100°C	1hr	54.71±8.55	58.28±13.39	52.19±14.15	55.06±12.03
		2hrs	47.98±14.48	55.92±4.97	42.48±2.50	48.79±7.32
		Mean	51.35±11.52	57.10±9.18	47.34±8.33	49.99±11.72
	120°C	1hr	46.66±17.08	54.68±10.85	60.64±20.49	53.99±16.14
		2hrs	51.37±4.66	54.39±15.13	57.00±12.53	54.25±10.77
		Mean	49.02±10.87	54.54±12.99	58.82±16.51	51.49±14.78
	140°C	1hr	60.97±4.50	44.17±12.96	48.33±8.97	51.16±8.81
		2hrs	47.33±13.22	51.62±6.42	47.73±7.50	48.89±9.05
		Mean	54.13±8.86	47.90±9.69	48.03±8.24	49.46±10.23
		Pooled Mean	52.92±8.14	54.93±12.36	51.65±12.19	51.16±12.83
Maximum Compressive Strength Perpendicular to grain (MCS ⊥)	Control	-	127.67±2.05	124.46±4.94	117.67±7.88	123.27±4.96
	100°C	1hr	119.22±8.10	126.16±0.58	119.50±4.13	121.63±4.27
		2hrs	117.29±5.65	126.09±0.72	122.35±6.12	121.91±4.16
		Mean	118.26±6.88	126.13±0.65	120.93±5.13	121.77±4.22
	120° C	1hr	82.93±6.42	105.12±8.66	110.14±10.08	99.40±8.39
		2hrs	125.16±0.12	126.06±0.71	126.99±0.20	126.07±0.34
		Mean	104.04±3.27	115.59±4.69	118.57±5.14	112.73±4.37
	140°C	1hr	118.36±9.43	126.24±0.48	125.89±1.93	123.50±3.95
		2hrs	124.94±0.43	126.99±0.70	126.79±0.46	126.24±0.53
		Mean	121.65±4.93	126.62±0.59	126.34±1.20	126.54±2.24
		Pooled Mean	117.91±4.28	123.20±2.72	120.88±4.84	120.66±3.94

Values indicate the Mean±SD

Table 5: Effect of hydrothermal treatment at different temperatures and time on mechanical properties of *Pinus caribaea* wood

Treatment condition	MCS// (N/mm²)	MCS⊥ (N/mm²)
Control	53.68±15.13[a]	122.64±7.40[a]
100-1h	51.74±13.74[a]	122.26±5.72[a]
100-2h	48.25±9.81[a]	122.59±5.70[a]
120-1h	51.17±17.74[a]	100.65±16.36[b]
120-2h	51.81±12.21[a]	126.04±0.93[a]
140-1h	49.87±11.61[a]	123.82±6.02[a]
140-2h	49.05±9.33[a]	126.24±1.07[a]

Mean±SD values with different superscript within the same column are significantly different (p>0.05). MCS// - Maximum Compressive Strength Parallel to grain, and MCS⊥ – Maximum Compressive Strength Perpendicular to grain.

DISCUSSION

Effect of Hydrothermal Treatment on Density of *Pinus caribaea* wood

The inconsistent pattern of axial density variation in this study is in contrast with the reports of several studies. Ugbong *et al*. (2024) observed a consistent decrease in the density of *Nauclea diderrichii* wood from base to the top, while a similar pattern was observed by Ogunsanwo and Onilude (2000) for *Triplochiton scleroxylon* wood.

Canolego *et al*. (2012) asserted that the best index for evaluating the quality of thermally-modified wood is its specific gravity. The hydrothermal treatment at the different temperatures and duration of treatment, though led to a reduction in the density of the wood, did not induce a significant influence on the wood density. A similar observation was reported by Hadi *et al*. (2016) where Poplar wood samples hydrothermally treated at 130°C and 180°C for

2hrs and 3hrs showed no significant change in their oven-dry density. Similarly, there was no considerable reduction in the density of thermally modified *Tectona grandis* wood treated at 160ºC (Lengowski *et al*., 2021). This indicates that the treatment temperature caused little or no degradation of the wood structural polymers (Freitas *et al*. 2017; Freitas *et al*. 2019). However, the appreciable reduction observed after treatment at 100°C for 2 hours may be attributed to the evaporation of volatile extractives and hemicelluloses degradation in the wood, thereby resulting in the loss of mass of the wood (Lengowski *et al*., 2021). Similar observation was also reported by Freitas *et al*. (2019) where hydrothermal treatment of *Eucalyptus grandis* wood treated at 140ºC at three different time intervals caused no appreciable change in the density of the wood. Furthermore, Esteves & Pereira (2009) also observed that heat treatment at moderate temperatures result into a minor density change in wood, while high treatment conditions cause significant loss of mass as a result of thermal degradation. As stated by Hill (2006), changes in the density of thermally modified wood is commonly associated with the degradation of wood polysaccharides rather than a change in the volume of the wood. By implication, irrespective of the position the wood was obtained from, the hydrothermal treatment would cause an inconsequential effect on the density of *P. caribaea* wood.

Effect on Water Absorption of *Pinus caribaea* wood

The higher reduction in the water absorption of the basal wood samples than the middle and top after treatment may be attributed to the higher density of the basal wood samples. This is because higher density wood usually comprises of cells with thick walls and narrow lumen (Karlsson *et al.*, 2011), which usually restricts free capillary movement of water in wood.

The general trend after the hydrothermal treatment showed a reduction in the water absorption of the wood, which is an indication of improved moisture resistance of the treated wood. This agrees with some previous studies on hydrothermal treatment of wood where reductions in moisture uptake were recorded (Charani *et al.,* 2007; Hadi *et al*., 2016; Freitas *et al*. 2017). This improvement may be attributed to increase in the crystallinity of the cellulose and the decrease in accessible hydroxyl groups resulting from hemicellulose degradation of the wood (Charani *et al.,* 2007; Yildiz & Gümüşkaya, 2007). These changes in the chemical constituents of wood reduce the wood's hygroscopic nature and consequently results in a decline in its affinity for water (Esteves & Pereira, 2009). However, contrary observation was reported by Ali *et al*. (2022) where an increase in water absorption was observed in hydrothermally treated *Hevea brasiliensis* wood as temperatures increased from 160°C to180°C in different buffered media. This was attributed to damages like cracking within the wood structure that was heat-induced. This may likely explain the increase in water absorption of some of the *P. caribaea* wood samples in this study as the treatment duration increased. Despite, the reduction in water absorption of the wood samples, significant reduction in the moisture uptake was not observed, indicating that the hydrophilic ability of *P. caribaea* wood cannot be reduced when hydrothermally treated at the sub-critical temperature range adopted in this study.

Effect of Hydrothermal Treatment on Swelling Properties of *Pinus caribaea* wood

The trend in anisotropic swelling observed in this study is similar to that reported by Adenaiya *et al.* (2022), where an inconsistent pattern in the swelling properties of *Pinus caribaea* was observed from base to top, though the highest anisotropic swelling occurred in the wood samples from the base.

Dimensional changes in wood is more pronounced in the tangential plane, followed by the radial plane, while the least is experienced along the longitudinal plane, which is most time practically insignificant (Hill, 2006; Freitas *et al*. 2019). In this study, the hydrothermal treatment caused a marked change in the swelling of the wood along the three-dimensional planes of the wood. The longitudinal swelling of the wood was considerably reduced when treated at 140°C for 1 hour, the radial swelling experienced pronounced reduction when treated at 120°C for 2 hours, while the tangential and volumetric swelling of the wood significantly reduced when treated at 100°C and 140°C. This is similar to reports by Lengowski *et al*. (2021) where thermal treated *Tectona grandis* showed significant reduction in its radial, tangential and volumetric shrinkages. According to Kocaefe *et al*. (2007), the water reabsorption capacity of wood diminishes when subjected to high temperatures. Freitas *et al.* (2017) observed a significant tangential retraction of Eucalyptus wood when hydrothermally treated at 100°C for 15hrs. Hadi *et al.* (2016) also reported a significant reduction in the volumetric swelling of Poplar wood when hydrothermally treated at 130°C and 160°C for 2 and 3 hrs. This reduction in the swelling characteristics of the treated wood could be as a result of the heat-induced modification of the wood cell walls (Rowell & LeVan-Green, 2005; Freitas *et al*. 2019) or the increase in the crystallinity of the cellulose microfibrils (Yildiz & Gümüşkaya, 2007). Tjeerdsma & Militz (2005) also stated that hemicelluloses degradation resulting from thermal changes can improve the dimensional stability of wood.

Effect of Hydrothermal Treatment on Mechanical Properties of *Pinus caribaea* wood

The inconsistent pattern of compressive strength along the tree bole in this study is in agreement with the reports of Ogunjobi *et al.* (2013), where an inconsistent variation in the MCS// of *Vitex doniana* wood was aboserved along the bole of the tree.

Chemical alterations of wood which occur due to thermal treatment often result into property changes in the wood, which includes a reduction in the wood's strength properties (Ebadi *et al*., 2016; Rasdianah *et al.*, 2018). The degree of the loss in mechanical property has been observed to depend on the

temperature used and exposure time (Ates *et al*., 2009). In this study. It was revealed in the study that both the treatment temperature and time of exposure had no profound effect on the MCS// of *P. caribaea* wood. This implies that the structural integrity of the wood was preserved despite treating the wood at temperatures up to 140°C. This is in contrast to report by Ebadi *et al.* (2016) where the MCS// of hydrothermally treated oil palm wood was significantly reduced at a temperature of 140°C in different buffered media. This might be due to the difference in the wood species. According to Ali *et al*. (2021), the difference in the anatomical features and chemical composition of wood may cause variations in the wood properties of a thermally modified wood. Hill (2006) stated that the dimensional stability of wood when treated at mild temperatures below 160°C are generally improved but causes minimal reduction in their mechanical properties. Furthermore, Esteves & Pereira (2009) asserted that the decline in the mechanical integrity of wood becomes more significant only when it is exposed to higher temperatures or prolonged treatment durations, which is usually caused by hemicellulose degradation and damage to the cell wall.

In contrast, the treatment temperature and time of exposure significantly influenced the MCS⊥ of *P. caribaea* wood. The significant reduction at 120°C for 1 hour may be as a result of a temporary weakening of the cell wall structure, while the increment in the mechanical property at a higher temperature and prolonged treatment duration may indicate a promotion of lignin condensation, which generally improves cell wall rigidity (Esteves & Pereira, 2009). The sampling positions of the wood also significantly influenced the MCS⊥, implying that wood of *P. caribaea* will have their MCS⊥ significantly altered when hydrothermally treated at these various treatment variables.

CONCLUSION AND RECOMMENDATIONS

Hydrothermal treatment of *Pinus caribaea* wood at the various temperatures and exposure period caused an insignificant change in the density of the wood but was incapable of significantly reducing the moisture absorption of the wood. However, the anisotropic swelling of the wood was considerably reduced, particularly when treated at 100°C and 140°C at the different exposure periods employed in the study. These enhancement in the dimensional stability of the wood was brought about by the modification of the wood's chemical constituents, chiefly the hemicelluloses, as well as the increment in the cellulose crystallinity of the wood. Though reductions in the strength property of the wood was observed at certain treatment conditions, these reductions were generally insignificant except for the drastic reduction of the wood's compressive strength perpendicular to the grain when treated at 120°C for 1 hour. Generally, the mechanical integrity of the wood was preserved after the hydrothermal treatments. Hydrothermal treatment of *P. caribaea* wood at 100°C and 140°C is therefore concluded to be the most ideal treatment conditions in order to enhance the wood's dimensional stability and maintain its structural integrity. This study also reveals that the position of the wood within the tree only had a significant influence on the wood's maximum compressive strength perpendicular to the grain before and after the hydrothermal treatment.

Authors' Contributions

Authors AOA & PAA were responsible for the data collection and analysis. AOA & BO were responsible for literature searches and writing of the manuscript. OYO was responsible for supervision of research and review of manuscript. All authors read and approved the final manuscript

Ethical Statement

Not Applicable

AI Declaration Statement

The authors declare that ChatGPT was sparingly used during the course of this write-up. All outputs were critically reviewed, verified, and approved by the authors, who accept full responsibility for the content of the manuscript.

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