



Article Dimensional Stability and Mechanical Properties of *Gmelina arborea* Roxb. Wood Thermally Modified through Open Reactor and Low-Pressure Closed Reactor Systems

Samuel Oluyinka Olaniran ^{1,2} and Holger Militz ^{1,*}

- ¹ Wood Biology and Wood Products, Georg-August-Universität, Büsgenweg 4, 37077 Göttingen, Germany; samuel.olaniran@uni-goettingen.de
- ² Department of Forestry and Wood Technology, Federal University of Technology, Akure P.M.B. 704, Nigeria
- * Correspondence: hmilitz@gwdg.de

Abstract: This study focused on the thermal modification of *Gmelina arborea* Roxb. wood following processes using the open reactor and low-pressure closed reactor systems. The aim is to determine the optimum treatment conditions suitable for gmelina wood due to its poor drying characteristics using the low-pressure closed reactor thermal modification. Subsequent to thermal modification under both processes, the dimensional stability and mechanical properties of gmelina wood were investigated. Effects of the thermal modifications under the open and low-pressure closed reactor systems on mechanical properties were additionally reported. The outcome of this investigation revealed that mass loss increased with increasing treatment temperatures, but minimal mass losses were observed for samples modified in the low-pressure closed reactor system. Due to the low-pressure regime used in the closed reactor system, a lesser improvement was found in volumetric shrinkage, fibre saturation point and tangential-to-radial swelling compared to the improvement in these properties in the open reactor system. Results further revealed that the mechanical properties of gmelina wood deteriorated more rapidly after modification in the open reactor system. Since the properties of modified gmelina wood are comparable at 180 °C under both systems, the closed reactor system will be investigated further to arrive at a suitable treatment condition under higher pressure variations. The thermal modification of gmelina wood with the closed reactor system is more promising in delivering a better quality of modified gmelina wood.

Keywords: thermal modification; gmelina wood; open reactor system; closed reactor system; volumetric swelling; T/R ratio; impact bending; Brinell hardness; bending properties

1. Introduction

Gmelina arborea Roxb. is a fast-growing tree species native to Asia, but currently planted extensively in various parts of the world including, especially, in tropical and subtropical climes [1,2]. Although the primary uses of gmelina have been in the pulp and paper industries, it has broad areas of application in other industries including veneers and plywood, furniture and wood-based panel products, with wide range of products such as papers, furniture interiors, veneers, plywood interiors, door panels, pencils, match sticks and pallets for transport and logistics [3–5]. Gmelina wood has low to moderate density values averaging from 430 to 452 kg/m³, with attractive wood figure and colour, which is pale brown after harvesting, and later changes to yellowish-brown. Outcomes from previous studies showed that gmelina wood possesses excellent mechanical properties that are comparable to commonly used European species such as spruce and pine [6,7]. Hence, gmelina wood has great potential for use as a construction material, not only in Nigeria or Africa as a whole, but in other countries of the world. However, in terms of its physical properties, gmelina wood is dimensionally unstable and has low durability, which is characteristic of many plantation-grown timbers requiring treatments for improved



Citation: Olaniran, S.O.; Militz, H. Dimensional Stability and Mechanical Properties of *Gmelina arborea* Roxb. Wood Thermally Modified through Open Reactor and Low-Pressure Closed Reactor Systems. *Forests* **2024**, *15*, 403. https://doi.org/10.3390/ f15030403

Academic Editors: Zeki Candan and Mehmet Hakki Alma

Received: 23 January 2024 Revised: 15 February 2024 Accepted: 17 February 2024 Published: 20 February 2024



Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). durability [8–10]. The improvement of these properties forms the major motivation for the treatment of wood, achieved by choosing from a variety of possibilities ranging from treatment with chemical preservatives for improved durability to chemical and thermal modification processes.

Thermal modification (TM) presents an eco-friendly alternative in wood treatment, leading to products that are environmentally friendly and can be safely disposed of at the end of their service life. According to Candelier and Dibdiakova [11], the rising environmental concerns due to the effects of many chemicals used in conventional wood treatment, especially biocides, are fast resulting in their becoming restricted, leading to increasing interest in thermally modified wood. However, it should be noted that the associated environmental impacts of thermally modified wood vary in both hardwoods and softwoods [12,13]. This is due to the variation in chemical composition between hardwoods and softwoods. An example of such is the presence of monoterpenes and sesquiterpenes in softwoods and higher acetyl content in hardwoods, leading to the production of acetic acids in the hemicelluloses of hardwoods [13–15].

Wood treatment through thermal modification has evolved through several stages of development over several decades. Under this process, the temperature under which wood is treated ranges from 160 °C to 240 °C; above this, severe degradation of wood polymers is the end result. Depending on the process used and its associated parameters, wood is basically modified in the absence of oxygen, while other parameters such as vacuum, nitrogen atmosphere, pressure variations and immersion in oil and steam are varied [16]. Several thermal processes have evolved in Europe over the years including ThermoWood, Firmolin, Plato, WTT and others, many of which have been commercialized, and the details of their process requirements have been provided in more detail by Sandberg et al. [17]. Process conditions following the specifications of ThermoWood[®] and WTT, which have been chosen for the treatment of gmelina wood, basically work under atmospheric pressure and pressurized conditions, respectively. An advantage common to these two processes includes the inclusion of a pre-drying of wood species prior to treatment, thereby saving energy in the production process. According to Altgen [18], thermal modification through the ThermoWood process involves the treatment of wood in an open reactor system, which is characterized by a pre-drying step at temperatures between 100 and 130 °C to reduce wood moisture content prior to the attainment of the target peak temperature. In the open reactor system, thermal modification is done under atmospheric pressure, which is accompanied by the removal of volatile organic compounds emitted by the wood from the reactor by a continuous flow of steam [12], and the modification of the wood in a dry state. In contrast to this, wood modification in the closed reactor system (WTT) occurs under a pressurized atmosphere that improves heat transfer and prevents the rapid drying of wood. During the treatment process, the volatile organic compounds increasingly accumulate in the reactor and may lead to rapid thermal degradation of the wood [19]. While thermal modification in the open reactor system has been investigated previously for gmelina wood, the modification of gmelina wood in the closed reactor system is yet to be investigated.

The limitations encountered in the range of wood species that can be treated with chemical modification processes due to difficulties in impregnation, as evident in the low uptake and weight percent gain of acetylated gmelina wood [20], presents a major advantage of the thermal modification process. Hence, thermal modification is applicable to treating a wide range of wood species, and may particularly be a potential solution for many difficult-to-treat wood species. Several treatments have been used in enhancing the treatability of wood species in the literature, with the end goal of improving only their solutions' uptake and durability. However, most of these methods require approaches that lead to damage of the macro- and microstructural features, leading to low mechanical strength and aesthetic properties, with little or no influence on improving the dimensional stability and resistance to moisture adsorption.

The choice of open and low-pressure closed thermal modification processes for gmelina wood in this study was made to determine the optimum treatment process that is suitable, without severe effects on wood quality. However, an initial report by Olaniran and Militz [21] showed that as the treatment temperature approaches 200 to 220 °C (in the open reactor system), increased cracks are generated in gmelina wood, which signifies that further investigations are required to determine the treatment conditions that minimize crack formation and improve its overall quality. The instance of crack development in gmelina wood was not mentioned by other authors [22], who have carried out thermal modification of gmelina wood using the open reactor system and similar processes, but this was reported earlier by Olaniran and Militz [21]. Hence, it becomes additionally important to investigate the influence of the thermal modification in extremely dry conditions. Use of the closed reactor system can also minimize instances of crack development in thermally modified gmelina wood, hence improving its quality after modification.

Previous investigations on the thermal modification of wood under low- and highpressure closed processes have shown that the degradation of wood polymers often leads to higher mass losses [23]. Consequently, these high losses in mass due to polymer degradation often have both positive and negative effects on wood properties, including improved dimensional stability on one hand, and the loss of mechanical properties on the other hand. Therefore, a compromise must be reached to know what properties to trade off depending on targeted applications. Therefore, another objective of this study was to thermally modify gmelina wood in a low-pressure closed reactor system, and then compare the effect of this treatment on dimensional stability and mechanical properties to wood modified in the open reactor system. Thermal modification in the low-pressure closed reactor system was chosen for the treatment of gmelina wood to assure minimal degradation and to find a suitable treatment schedule.

2. Materials and Methods

2.1. Procurement and Processing of Gmelina Wood

Wood samples for this experiment were prepared from a 25-year-old *Gmelina arborea* tree, harvested at the plantation of the Federal University of Technology Akure, Nigeria. The location of the plantation within the university is N 7.18303, E 05.07468 and N 7.30875, E 05.13142. As an indication of age, over 90% of the harvested stem diameter consists of mainly the heartwood. The harvested logs from this tree were sawn with a chain saw into timber with the dimensions 1500 mm (length) \times 340 mm (width) \times 71 mm (thickness). The processed boards were stacked under a drying shed to be air-dried for three months before shipping to the Department of Wood Biology and Wood Products, University of Göttingen, Germany for this study.

Sample Preparation for Thermal Modification

Boards of gmelina wood were processed into slats with the dimensions 650 mm \times 50 mm \times 20 mm (longitudinal-tangential-radial). A total of ninety-four slats were thermally modified. Out of these, forty-six (46) slats were modified in the open reactor system, while forty-eight (48) slats were modified in the closed reactor system. No initial kiln-drying was done before the thermal treatments, and the initial weights of all slats were measured before treatment. In addition, a sample with a thickness of 15 mm was cut from each slat along the length before thermal modification to determine the initial moisture contents of the slats. Aside from the thermally modified slats, another thirty (30) slats were reserved as unmodified (control).

2.2. Thermal Modification Processes

Slats of gmelina wood were modified in a 65-litre capacity reactor (WTT Jyllandsvej, Denmark). This is a laboratory-scale reactor where thermal modification under atmospheric pressure (open reactor system) and thermal modification under pressure (closed reactor system) were performed under the conditions shown in Table 1. Thermal modification in the open reactor system entails the treatment of slats under atmospheric pressure, and the details of the schedule included stepwise increases in temperature in the reactor, starting

at 12 °C, until it reached 100 °C. This was followed by the pre-drying of the slats as the temperature was increased in steps by 2 °C per hour until it reached 130 °C. After this step, the temperature in the reactor was increased at 12 °C per hour until the target temperature for modification was attained, and a holding time of 180 min was applied at the target temperature. After the modification was completed, the temperature in the reactor was gradually decreased at the rate of -20 °C per hour until it cooled down to about 65 °C.

Peak Temp	Max Pressure (MPa)	RH (%)	Total Treatment Time (h)	Treatment Time at Peak Temperature (h)					
Low-pressure closed reactor system									
160	0.18	30	17.75	3					
170	0.23	30	19.08	3					
180	0.29	30	20.42	3					
Open reactor system									
180	Atmospheric	0	32.92	3					
200	Atmospheric	0	35.58	3					
220	Atmospheric	0	38.25	3					

Table 1. Process conditions for thermal modification of gmelina wood.

The low-pressure closed reactor system involved four major steps, which included a 50 min holding step pre-vacuum, initially applied at <14 kPa, a temperature increase at the rate of 12 °C per hour until the target temperature for the modification was attained, followed by a 180 min holding step at target temperature, and finally a stepwise decrease in temperature at the rate of -20 °C per hour until 65 °C was reached. Total duration for modification was longer in the open reactor system (up to 38 h) compared to the closed system (up to 21 h). Total duration in each process was dependent on the target treatment temperature and pressure. For each treatment temperature under this process, including 160, 170 and 180, minimum pressures of 0.18, 0.23 and 0.29 MPa, respectively, were used. After the modification was completed in each process, the reactor was allowed to cool, and the modified wood was removed and weighed. Samples of 15 mm thickness were immediately cut from each slat, and oven-dried to estimate the moisture content of thermally modified slats.

2.3. Determination of Extractive Content and Correction of Mass Loss

In accordance with the procedure described by Metsä-Kortelainen et al. [24], dry masses of modified slats were determined, and since the degradation products were expected to accumulate in addition to extractives present, the weight of extractives had to be deducted to ensure that mass losses are comparable for both TM processes. We determined the proportion of extractives using a method similar to that described in the TAPPI standard [25]. To determine the extractive content, samples from the thermally modified slats (selected from all treatment variations and the unmodified gmelina wood) were milled into particle sizes of about 0.3 mm. Thereafter, amounts of 5 g of dried wood particles were weighed into extraction tubes, and extracted with hot distilled water using a Soxhlet apparatus for 8 h. After extraction, the initial dry weight of particles and final dry weight were considered in estimating the extractive content using the following formula:

$$Extractive \ content = \frac{W_b - W_a}{W_b} \times 100 \tag{1}$$

where W_b is the dry weight of wood particles before extraction, and W_a is the dry weight of wood particles after hot-water extraction.

The corrected weights of slats and corrected mass loss were calculated as described by Wentzel et al. [16]:

$$C_w = Dry_w - \frac{(Dry_w \times Extractive \ content)}{100}$$
(2)

where C_w is the corrected weight of thermally modified slats, Dry_w is the dry weight of slats calculated before and after thermal modification.

$$CML = \frac{Dry_{cwb} - Dry_{cwa}}{Dry_{cwb}} \times 100$$
(3)

where CML is the corrected mass loss; Dry_{cwa} is the corrected dry weight (in grams) of slats after modification, and Dry_{cwb} is the corrected dry weight before modification.

2.4. Volumetric Swelling and Anti-Swelling Efficiency

Samples with dimensions 25 mm \times 25 mm \times 10 mm were prepared from unmodified (serving as control) gmelina wood and thermally modified slats with varying treatment processes and schedules. A total of two hundred and ten (210) samples were prepared, with thirty samples per treatment (Table 2). Prior to oven-drying at 103 \pm 2 °C, initial weights and dimensions of the samples were measured, followed by pre-conditioning in a climate chamber at 65% relative humidity (RH) and 20 °C until the samples attained a constant weight. After oven-drying, the samples were measured again for their oven-dry weights and dimensions. Subsequently, the samples were immersed in water for ten days to achieve complete saturation, followed by measurement of saturated mass and volume of wood samples. Volumetric swelling (VS) and anti-swelling efficiency (ASE) were calculated after conditioning at 65% RH and after saturation in water as follows:

$$VS(\%) = \frac{V_2 - V_1}{V_1} \times 100$$
(4)

$$ASE(\%) = \frac{VS_{unmod} - VS_{mod}}{VS_{unmod}} \times 100$$
(5)

where V_1 and V_2 are the volume of samples with conditioning at 65% RH and saturation in water and the volume of oven-dried samples, respectively. VS_{unmod} and VS_{mod} represent the volumetric swelling of unmodified wood and volumetric swelling of modified wood, respectively.

Table 2. Indicators of dimensional stability of gmelina wood subsequent to thermal modification through open and closed reactor systems.

Peak Temp (°C)	No. of Samples	FSP (%)	Vol Swelling (%)	ASE (%)	T/R Ratio			
Low-pressure closed reactor system								
160	30	17.65 (1.73) ^b	7.42 (0.41) ^d	24.13 (7.66)	1.72 (0.57) ^{ab}			
170	30	17.35 (2.19) ^b	6.83 (0.45) ^c	30.02 (8.70)	1.67 (0.69) ^b			
180	30	12.83 (2.87) ^c	5.26 (0.88) ^b	46.44 (8.77)	1.67 (0.44) ^b			
Open reactor system								
180	30	13.01 (1.58) ^c	5.41 (0.71) ^c	44.85 (7.40)	1.65 (0.42) ^b			
200	30	7.95 (0.99) ^b	3.31 (0.25) ^b	66.09 (4.32)	1.44 (0.14) ^b			
220	30	7.93 (0.57) ^b	3.08 (0.23) ^b	68.46 (3.84)	1.39 (0.34) ^c			
* Unmodified (control)	30	22.18 (3.52) ^a	9.86 (0.89) ^a	-	2.03 (0.20) ^a			

Means with the same letters vertically are not significantly different (p > 0.05). * Each process is to be compared with the unmodified (control) samples for FSP, volumetric swelling and T/R ratio.

2.5. Determination of Fibre Saturation Point and Radial/Tangential Swelling

The fibre saturation points (FSPs) of unmodified and thermally modified gmelina wood were determined from the volumetric swelling of wood samples calculated using Equation (3), and the ratio of the density of water to the density of oven-dried wood samples was calculated according to the equation given by Jankowska and Kozakiewicz [26]:

$$FSP = VS(\%) \times \frac{\rho_w}{\rho_0} \tag{6}$$

where VS is the volumetric swelling of the unmodified and the modified wood, ρ_w is the density of water and ρ_o is the density of the oven-dried wood samples. The ratio of tangential to radial swelling was calculated from the swelling values of the samples after complete saturation in water after soaking for ten days.

2.6. Determination of Mechanical Properties

The mechanical properties, including bending properties (MOE and MOR), work to maximum load in bending (WMLB), Brinell hardness, and impact bending strength were tested for the unmodified and thermally modified samples. Bending properties and Brinell hardness were tested using the 10 kN Zwick-Roell Z010 (Zwick, Ulm, Germany), and the impact bending strength was tested using the Resil Impactor (CEAST, Martinsried, Germany). Samples were conditioned at the relative humidity of 65% and a temperature at 20 °C for two weeks prior to the tests.

A three-point bending test was conducted according to the DIN 52186 standard [27], using sample dimensions of 10 mm \times 10 mm \times 180 mm (R \times T \times L). Tested samples consisted of unmodified and thermally treated gmelina wood from open and closed reactor systems, using 30 samples each for the unmodified and individual treatment temperatures. Tests were made at crosshead speed of 3 mm/min to achieve failure of samples within 90 s. An external extensometer was used to determine the strain in the samples within the elastic limit. At the end of the test, MOE, MOR and WMLB were automatically generated from the software, TextXpert[®] III, V1.51 (2020), and exported into Microsoft Excel files.

A hardness test was conducted according to DIN EN 1534 standard [28], with sample dimensions of 15 mm \times 50 mm \times 50 mm (R \times T \times L) having been prepared from unmodified and thermally modified samples. For each treatment, including the unmodified and treated samples, 30 samples were tested. A steel ball of 10 mm with a maximum pressure of 1000 N was exerted on the radial surface of each sample for a maximum of 90 s. Brinell hardness (BH) was calculated:

$$BH = \frac{2.F}{\pi \cdot D \left[D - \sqrt{(D^2 - d^2)} \right]}$$
[MPa] (7)

where F is the maximum force (N); D is the diameter of the steel ball, d is the diameter of the imprint on the tested samples, and π is 3.142.

For impact bending strength (IBS) tests, samples with dimensions 10 mm \times 10 mm \times 180 mm (R \times T \times L) were used, and consisted of unmodified and thermally modified wood. The Resil Impactor was connected to an interface (DAS 8000), with a hammer energy of 15 joules. Fifteen (15) samples were tested for each treatment batch. Impact bending strength (w) was calculated from total energy and sample dimensions:

$$w = \frac{W}{bh} \times 1000 \ [kJ/m^2] \tag{8}$$

where w is the total force required to break the sample, while b and h are the samples' cross-sectional dimensions.

2.7. Statistical Analysis

Data collected from experiments in this study were analysed with data-analytical software including Microsoft Excel (Microsoft Office Professional Plus 2021), Origin Pro (version 2020), and analysis of significant differences among the variables in the study were analysed using SPSS software v20 (IBM Statistics Inc., Chicago, IL, USA).

3. Results and Discussion

3.1. Mass Loss of Thermally Modified Gmelina Wood

One of the major changes that occur during thermal modification is mass loss due to the degradation of wood polymers. Figure 1 shows the degree of mass loss in samples thermally modified in open and closed reactor systems. Generally, mass loss increases with increases in treatment temperature, showing the degree to which wood polymers, most importantly hemicelluloses, are thermally degraded during treatment, and is accompanied by conversion of some extractives to volatile organic compounds [29]. Variations also exist between mass loss and corrected mass loss under both processes, with a larger difference in the open process as the treatment temperature increases compared to the low-pressure closed process. This difference could be attributed to the level of degradation that occurred during both processes, with the open reactor system yielding more degradation products as the temperature of the system increased compared to the low-pressure closed process. High corrected mass loss values were recorded in the thermal modification of *Eucalyptus nitens*, as previously reported by Wentzel et al. [23], under the open process. It was, however, observed from that study that the corrected mass loss increased in the closed reactor system when thermal treatment was carried out under high pressure. For example, Eucalyptus nitens wood modified at 160 °C under the low pressure of 0.18 MPa and 30% RH had a corrected mass loss of 5.0%; samples modified at 160 °C under the high pressure of 0.61 MPa and 100% RH recorded a corrected mass loss of 18.6%; while samples modified under an open process at 160 °C under atmospheric pressure recorded a corrected mass loss of 5.4%. When compared to this study, gmelina wood modified at 160 °C under the low pressure of 0.18 MPa and 30% RH recorded a mean corrected mass loss of 4.3%. This implies a possibility of higher amounts of degradation products from hemicelluloses and more conversion of extractives to VOCs if gmelina wood is further modified under high pressure, despite a low treatment temperature.



Figure 1. Mass loss and corrected mass loss of thermally modified slats of gmelina wood in the (**a**) open reactor system, and in the (**b**) low-pressure closed reactor system.

3.2. Dimensional Stability of Thermally Modified Gmelina Wood

The following results, presented in Table 2, show the influence of thermal treatment through open and low-pressure closed processes on the dimensional stability of gmelina

wood. The fibre saturation point (FSP) of unmodified gmelina wood is 22.2%. For samples modified in the low-pressure closed reactor system, FSP values of 17.7%, 17.4% and 12.8% were obtained at treatment temperatures of 160 °C, 170 °C and 180 °C, respectively. The FSP was significantly different at treatment temperatures of 160 °C, 170 °C and 180 °C in the low-pressure closed system when compared to the unmodified samples, while samples treated at 180 °C recorded the lowest FSP in this treatment category. Samples modified in the open reactor system recorded FSP values of 13%, 7.9% and 7.9% at modification temperatures of 180 °C, 200 °C and 220 °C, respectively. For this process, the FSPs of samples treated at 200 and 220 °C showed similar values, and were significantly different from those treated at 180 °C in the open and closed reactor systems recorded comparable FSP values of 13% and 12.8%.

These results indicated that the FSPs of thermally modified gmelina wood were significantly reduced in samples modified in the open reactor system, which could be a result of higher degradation of hemicelluloses, which are mostly responsible for moisture uptake to available sorption sites in the wood cell walls. The inability of the samples treated at 160 °C and 170 °C in the low-pressure closed reactor system to achieve significantly low FSPs, as in other treatment temperatures, may also point to low degradation of hemicelluloses and the availability of many more sorption sites in their polymeric structure. As shown in Figure 2, mass loss significantly influenced FSP, such that it decreased as mass loss increased, but this relationship is stronger for samples modified in the open reactor system (a) compared to those in the low-pressure closed reactor system (b). This observation also points to a severe reduction of OH groups, and correspondingly low FSPs have been previously attributed to possible polycondensation reactions that occur during thermal modification, leading to cross-linking of lignin, and replacement of hydroxyl groups by O-acetyl groups in crystalline cellulose due to increased crystallinity [30,31].



Figure 2. Influence of mass loss on fibre saturation point and volumetric swelling of gmelina wood thermally modified through the open reactor system (**a**,**b**) and low-pressure closed reactor system (**c**,**d**).

Under a fully saturated condition, unmodified gmelina wood recorded a mean volumetric swelling of 9.9%. Samples modified in the open reactor system recorded mean volumetric swelling of 5.4%, 3.3% and 3.1% at treatment temperatures of 180 °C, 200 °C and 220 °C, respectively, and were all significantly different from the unmodified samples. There is no significant difference between samples modified at 200 °C and 220 °C. In contrast, samples modified under the low-pressure closed reactor system recorded higher volumetric swelling of 7.4%, 6.8% and 5.3% at treatment temperatures of 160 °C, 170 °C and 180 °C, respectively, and were also significantly different from the unmodified samples. A closer look into the volumetric swelling under both processes and treatment temperatures showed that samples modified at 180 °C using both systems recorded similar swelling values.

As shown in the regression between volumetric swelling and mass loss (Figure 2c,d), the correlation coefficient was higher for samples modified through the open reactor system. For both processes, volumetric swelling decreases with increasing mass loss due to increases in modification temperature. In general, a significantly low volumetric swelling was recorded due to thermal modification; to a higher degree in the open reactor system and a lower degree in the low-pressure closed reactor system. This provides further confirmation of results from previous studies [32–34] that indicate that thermal treatment at high temperatures makes treated wood less hygroscopic through hemicelluloses' degradation, which represents a major contributor to dimensional stability as the number of free hydroxyl groups available for bonding to moisture is severely reduced.

Further than the effect of the various thermal modification processes on swelling of gmelina wood is the anti-swelling efficiency (ASE) of the modified wood as revealed in the applied processes. In Table 2, it can be observed that the ASE of the treated samples increased with increasing treatment temperatures for both systems. Samples modified in the open reactor system recorded ASE values of 44.9%, 66.1% and 68.5% at modification temperatures of 180 °C, 200 °C and 220 °C, respectively.

ASE values of 24.1%, 30% and 46.4% were recorded at treatment temperatures of 160 °C, 170 °C and 180 °C for samples treated with the low-pressure closed reactor process. Similar to previous observations for FSP and volumetric swelling, samples modified at 180 °C under both processes recorded 44.9% and 46.4% in the open and low-pressure closed reactor systems, respectively. Regression analysis performed between ASE and mass loss revealed that samples modified in the open process had a higher correlation coefficient compared to those modified in the low-pressure closed process (Figure 3). This emphasizes that wood samples modified in the open reactor system may have undergone significant changes in their cell walls, especially the degradation of moisture-reactive hemicelluloses leading to more pronounced mass losses with a corresponding influence on ASE.



Figure 3. Influence of mass loss on anti-swelling efficiency of gmelina wood modified through open reactor system (ASE-OP) and low-pressure closed reactor system (ASE-CL).

The ratio of tangential swelling to radial swelling (T/R ratio) was determined to measure the dimensional stability of gmelina wood before and after thermal modification

under the selected systems. Wood species are dimensionally stable if their T/R ratio is approximately 1 [35]. For gmelina wood, the T/R ratio of unmodified wood is 2, indicating that it is not dimensionally stable. Subsequently, after modification in the open reactor system, it attained T/R ratios of 1.7, 1.4 and 1.4 at treatment temperatures of 180 °C, 200 °C and 220 °C, respectively. The T/R ratios after treatment in the open reactor system were significantly different from those of the unmodified samples. In the low-pressure closed reactor system, T/R ratios of 1.7, 1.7 and 1.7 were obtained, respectively, at treatment temperatures of 160 °C, 170 °C and 180 °C. Under this system, however, the T/R ratio was not significantly different from the unmodified samples. This indicates that gmelina wood is more dimensionally stable after thermal modification compared to its unmodified state, and we recorded a significantly lower T/R ratio only after treatments at 200 and 220 °C in the open process.

3.3. Mechanical Properties of Thermally Modified Gmelina Wood

The result of our analysis of the bending properties of gmelina wood following thermal modification through the open reactor system and low-pressure closed reactor system is shown in Figure 4. In the former (as shown in Figure 4a,c), a slight decrease in the modulus of elasticity (MOE) was observed as the treatment temperature increased, with the exception of samples treated at 180 °C, where the MOE increased to 15,630.2 MPa from 13,378.9 MPa in the unmodified samples. This represents an increase of about 16.8% at this temperature, while there was a proportional decrease in the MOE of 5.9% for samples treated at 220 °C. In the low-pressure closed reactor system (Figure 4b,d), there was a slight increase in MOE as the temperature increased from 160 °C to 180 °C. This difference in the increase between the unmodified samples and those modified under the low-pressure closed reactor system is not significant.

Increases in temperature led to a pronounced decrease in the modulus of rupture (MOR), as this property decreased from 87.9 MPa in the control/unmodified samples to 81.4 MPa, 66.9 MPa and 62.2 MPa for samples modified at 180 °C, 200 °C and 220 °C, respectively. This translates to a proportionate loss of 23.9% and 29.2% in MOR for samples modified at 200 °C and 220 °C, respectively. In contrast to the effect of heat treatment on samples modified in the open reactor system, a slight decrease in MOR was observed only after treatment at 180 °C, wherein the MOR decreased from 87.9 in the unmodified samples to 81.5 MPa. These results revealed the influence of the two systems on the bending properties of gmelina wood. While it appeared that the two processes did significantly influence the MOE, the effect of thermal modification on the MOR, on the other hand, was significant for samples modified with the open reactor system, and to a lower extent in the low-pressure closed reactor system. It is well known that thermal modification affects the cell wall polymers, leading to losses in mechanical properties, but this depends on the process conditions. It was earlier reported that wood modified under open and highpressure closed reactor systems has the tendency to be more brittle [23] as hemicelluloses' degradation during the thermal modification process is known to have a corresponding effect on decreases in mechanical properties [36]. However, it will be observed that in this study, this assertion is mainly true for samples modified in the open reactor system. The correspondingly low effect of the low-pressure closed reactor system may present a different scenario and implications for strength properties if the samples are modified under a high-pressure system.

Results of work to maximum load in bending (WMLB) analysis, shown in Figure 5, showed that for samples modified in the open reactor system, WMLB was found to decrease more severely compared to those modified under the low-pressure closed reactor system. This decrease may point to a reduction in the strength and toughness of the samples modified in the open reactor system. For samples modified in the low-pressure reactor system, a decrease in WMLB was also significant when compared to the unmodified samples, showing that even though the mass losses are not as high as for those modified in the open reactor system, decreases in the strength and toughness of the materials

may have been a corresponding effect of the degradation of hemicelluloses despite the low-pressure thermal treatment. Decreases in toughness and strength resulting from the brittleness of thermally modified wood have been previously linked to the loss of amorphous polysaccharides in addition to the crystallization of amorphous cellulose due to thermal modification, with subsequent increases in brittleness and deterioration of mechanical properties [29,37,38].



Figure 4. Bending properties of gmelina wood thermally modified through open reactor system (a,c) and low-pressure closed reactor system (b,d). The box plot can be interpreted as follows: the centre line = the median, open square = the mean, box length = 25th to 75th percentile, whiskers 5th to 95th percentile, the black rhombuses are the outliers. Means with the same letters are not significantly different (p > 0.05).



Figure 5. Work to maximum load in bending for thermally modified gmelina wood through open (a) and low-pressure closed (b) reactor systems. Means with the same letters are not significantly different (p > 0.05).

As shown in Figure 6a,b, samples thermally modified through open and low-pressure closed reactor systems responded differently in terms of changes in hardness. At the treatment temperature of 180 °C in the open reactor system, there was an initial increase in hardness from 15.5 N/mm² in unmodified samples to 18 N/mm². Then, a decrease in hardness occurred at modification temperatures of 200 and 220 °C to 14.7 and 13.6 N/mm², respectively. Hardness was increased following treatment in the low-pressure closed process only at 170 °C, while there was no significant difference in hardness between the unmodified samples and those treated at 160 and 180 °C in the low-pressure closed reactor system. The average value of hardness recorded for unmodified gmelina wood is 15.34 MPa, and the increase in hardness recorded due to thermal modification to 23.26 MPa still would not qualify gmelina wood to be used as a flooring material when compared with some European wood species used for the same purpose. It is clear, however, that decreases in hardness have been kept at a minimum due to the lower modification temperatures used in the low-pressure closed reactor system. Brinell hardness measures the resistance of materials to indentation, and is of utmost importance when considering wood-based materials for floor tiling. However, since thermally modified wood becomes more brittle by losing its elasticity, the intensity of the treatment that is the modification temperature can be kept at a minimum to prevent excessive strength loss [38].



Figure 6. Brinell hardness and impact bending strength of thermally modified gmelina wood through open (a,c) and low-pressure closed (b,d) reactor systems. Means with the same letters are not significantly different (p > 0.05).

The impact bending strength (IBS) test is a dynamic test that measures the amount of energy required to cause a rupture in wood materials upon exposure to impact loads. Unmodified and thermally modified gmelina wood samples were tested for their resistance to impact loads on the basis of their treatment process (Figure 6c,d). Gmelina samples modified in the open reactor system recorded a continuous decrease in impact bending strength as the temperature increased from 180 °C to 220 °C, from 10.9 kJ/m² in the unmodified samples to 5.9 kJ/m² after treatment at 220 °C. This decrease was significantly different between the unmodified samples and samples with treatments from 180 °C to 220 °C. For samples modified under the low-pressure closed reactor system, there were large variations when comparing the unmodified samples and the modified samples from 160 °C to 180 °C, with samples modified at 180 °C recording an impact bending strength of 9 kJ/m², which is not significantly different from the unmodified samples. Generally, gmelina wood has a low impact bending strength when compared to other European hardwoods like beech (with an average IBS value of 59 kJ/m²), although this property is influenced by material thickness [39], and can be as high as 100 kJ/m² [40]. Upon exposure to thermal treatment, especially in the open reactor system, the IBS of gmelina wood further decreased significantly with increases in temperature, which justifies the previous assertion that the exposure of wood to high temperatures significantly influences its IBS [39].

4. Conclusions and Outlook

This study has examined the dimensional stability and mechanical properties of gmelina wood thermally modified using the open and low-pressure closed reactor systems. The effects of the two thermal modification systems on the dimensional stability of gmelina wood has shown that thermal modification in the open reactor system had a greater influence on indicators of dimensional stability and moisture resistance such as fibre saturation point, volumetric swelling, anti-swelling efficiency, and the ratio of tangential to radial swelling (T/R ratio). This outcome points to a higher degradation of moisturereactive hemicelluloses in the open reactor system, as reported in earlier studies, than in the low-pressure closed reactor system. This is expected, in the first place, due to variations in modification temperatures and pressure from both systems. However, it was earlier mentioned that higher degradation usually occurs under high-pressure closed reactor systems, even at lower temperatures, compared to open reactor systems. Bending strength, work to maximum load in bending, and impact bending strength of gmelina wood were mostly affected by thermal modification in the open reactor system, and to a lesser extent in the low-pressure closed reactor system. Due to the variation in temperature under both systems, it is not presently possible to figure out a suitable treatment condition for gmelina wood. However, due to the advantage of the better quality of modified gmelina made possible in the closed process, future studies will focus on comparing treatments at varying temperatures and pressures (higher than in the present study) in the closed system. Further investigations will also cover the durability of gmelina wood subsequent to thermal modification under both open and closed reactor systems.

Author Contributions: Conceptualization, S.O.O. and H.M.; methodology, S.O.O.; formal analysis, S.O.O.; investigation, S.O.O.; resources, H.M.; data curation, S.O.O.; writing—original draft preparation, S.O.O.; writing—review and editing, H.M. and S.O.O.; visualization, S.O.O.; supervision, H.M.; project administration, H.M. and S.O.O.; funding acquisition, S.O.O. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Alexander von Humboldt Foundation with a Postdoctoral Fellowship awarded to Dr. Samuel Oluyinka Olaniran, with the funding number NGA-1217525-GF-P.

Data Availability Statement: Data are contained within the article.

Acknowledgments: The efforts of Mirko Kueppers, Dieter Varel and Bernd Bringermeier are sincerely acknowledged for their technical support, transport of experimental materials and sample preparation processes, respectively.

Conflicts of Interest: The authors declare no conflicts of interest.

References

- 1. Naik, D.; Singh, D.; Vartak, V.; Paranjpe, S.; Bhargava, S. Assessment of Morphological and Genetic Diversity in *Gmelina arborea* Roxb. *New For.* **2009**, *38*, 99–115. [CrossRef]
- Singh, P.; Rana, A.; Panwar, N.S.; Kumar, A. Tree Improvement, Breeding and Biotechnology of *Gmelina arborea* Roxb. *Indian For.* 2021, 147, 1075. [CrossRef]
- 3. Singh, S.; Kumar, A.; Senthilkumar, N.; Singh, A.N. Strategies for the Management of *Craspedonta leayana* (Coleoptera: Chrysomelidae) in *Gmelina arborea*, Rain Forest Research Institute, Deovan, Jorhat, Assam. *Indian For.* **2006**, *132*, 581–588.
- 4. Kumar, A. Growth Performance and Variability in Different Clones of *Gmelina arborea* (Roxb.). *Silvae Genet.* **2007**, *56*, 32–35. [CrossRef]
- 5. Krishen, P. Jungle Trees of Central India: A Field Guide for Tree Spotters; Penguin Books India: Gurugram, India, 2013.
- 6. Atagbua, C.O.; Enwelu, C.; Aderibigbe, W.; Okiwe, E.O. A Comparative Study of Some Mechanical Properties of *Gmelina arborea*, *Parkia biglobosa* and *Prosopis africana* Timbers for Structural Use. *Int. J. Tech. Res. Appl.* **2015**, *3*, 320–324.
- Iwuoha, S.E.; Seim, W.; Onyekwelu, J.C. Mechanical Properties of *Gmelina arborea* for Engineering Design. *Constr. Build. Mater.* 2021, 288, 123123. [CrossRef]
- Haslett, A.N.; Zoung, G.D.; Britton, R.A.J. Plantation Grown Tropical Timbers. 2. Properties, Processing and Uses. J. Trop. For. Sci. 1991, 3, 229–237.
- 9. Okon, K.E. Variations in Specific Gravity and Shrinkage in Wood of a 25-Year-Old *Gmelina arborea* in Oluwa Forest Reserve, South West Nigeria. *Arch. Appl. Sci. Res.* 2014, *6*, 271–276.
- 10. Owusu, F.W.; Apetorgbor, M.M.; Nsor, C.A.; Adu, S.; Alorbu, C.; Apetorgbor, A.K. Evaluation of Natural Durability of Plantation Grown *Gmelina arborea* Roxb. Ex Sm. Wood from Two Vegetation Zones in Ghana. *Ghana J. For.* **2022**, *38*, 55–66.
- 11. Candelier, K.; Dibdiakova, J. A Review on Life Cycle Assessments of Thermally Modified Wood. *Holzforschung* **2021**, *75*, 199–224. [CrossRef]
- 12. Mayes, D.; Oksanen, O. ThermoWood®Handbook; Finnish Thermowood Association: Helsinki, Finland, 2003.
- Hyttinen, M.; Masalin-Weijo, M.; Kalliokoski, P.; Pasanen, P. Comparison of VOC Emissions between Air-Dried and Heat-Treated Norway Spruce (*Picea abies*), Scots Pine (*Pinus sylvesteris*) and European Aspen (*Populus tremula*) Wood. *Atmos. Environ.* 2010, 44, 5028–5033. [CrossRef]
- 14. Peters, J.; Fischer, K.; Fischer, S. Characterization of Emissions from Thermally Modified Wood and Their Reduction by Chemical Treatment. *BioResources* 2008, *3*, 491–502. [CrossRef]
- 15. Wolpert, B. Emission and Abundance of Biogenic Volatile Organic Compounds in Wind-Throw Areas of Upland Spruce Forests in Bavaria; Technische Universität München: Munich, Germany, 2012.
- 16. Altgen, M.; Militz, H. Effect of Temperature and Steam Pressure during the Thermal Modification Process. In Proceedings of the 8th European Conference on Wood Modification, Helsinki, Finland, 26–27 October 2015.
- 17. Sandberg, D.; Kutnar, A.; Mantanis, G. Wood Modification Technologies—A Review. *iForest—Biogeosciences For.* **2017**, *10*, 895–908. [CrossRef]
- Altgen, M. Impact of Process Conditions in Open and Closed Reactor Systems on the Properties of Thermally Modified Wood; Cuvillier Verlag: Göttingen, Germany, 2016; ISBN 978-3-7369-8421-9.
- 19. Stamm, A.J. Thermal Degradation of Wood and Cellulose. Ind. Eng. Chem. 1956, 48, 413–417. [CrossRef]
- 20. Gaitán-Alvarez, J.; Berrocal, A.; Mantanis, G.I.; Moya, R.; Araya, F. Acetylation of Tropical Hardwood Species from Forest Plantations in Costa Rica: An FTIR Spectroscopic Analysis. *J. Wood Sci.* **2020**, *66*, 49. [CrossRef]
- Olaniran, S.; Militz, H. Modification of *Gmelina arborea* Wood for Utilization in Nigeria. In Proceedings of the 54th Annual Meeting of the International Research Group on Wood Protection, Cairns, Australia, 28 May–1 June 2023; The International Research Group on Wood Protection: Cairns, Australia, 2023.
- 22. Moya, R.; Fallas-Valverde, L.; Berrocal, A.; Méndez-álvarez, D. Durability of Thermally Modified Wood of *Gmelina arborea* and *Tectona grandis* Tested under Field and Accelerated Conditions. J. Renew. Mater. **2017**, *5*, 208–219. [CrossRef]
- 23. Wentzel, M.; Fleckenstein, M.; Hofmann, T.; Militz, H. Relation of Chemical and Mechanical Properties of *Eucalyptus nitens* Wood Thermally Modified in Open and Closed Systems. *Wood Mater. Sci. Eng.* **2019**, *14*, 165–173. [CrossRef]
- 24. Metsä-Kortelainen, S.; Antikainen, T.; Viitaniemi, P. The Water Absorption of Sapwood and Heartwood of Scots Pine and Norway Spruce Heat-Treated at 170 °C, 190 °C, 210 °C and 230 °C. *Holz Als Roh-Und Werkst.* **2006**, *64*, 192–197. [CrossRef]
- 25. *Test Method T 204 cm-07;* Solvent Extractives of Wood and Pulp. Technical Association of the Pulp and Paper Industry (TAPPI): Atlanta, GA, USA, 1997.
- 26. Jankowska, A.; Kozakiewicz, P. Determination of Fibre Saturation Point of Selected Tropical Wood Species Using Different Methods. *Drew. Pr. Nauk. Doniesienia Komun.* 2016, *59*, 89–97. [CrossRef]
- 27. DIN 52186; Prüfung von holz—Biegeversuch. Deutsches Institut für Normung e.V.: Berlin, Germany, 1978.
- 28. DIN EN 1534; Parkett und andere Holzfußböden—Bestimmung des Eindruckwiderstands—Prüfmethode. Deutsches Institut für Normung e.V.: Berlin, Germany, 2010; p. 12.
- 29. Esteves, B.; Pereira, H.M. Wood Modification by Heat Treatment: A Review. BioResources 2007, 4, 370–404. [CrossRef]
- 30. Boonstra, M.J.; Tjeerdsma, B. Chemical Analysis of Heat-treated Softwoods. Holz Als Roh-Werkst. 2006, 64, 204–211. [CrossRef]
- 31. Kocaefe, D.; Poncsak, S.; Doré, G.; Younsi, R. Effect of Heat Treatment on the Wettability of White Ash and Soft Maple by Water. *Holz Als Roh-Werkst.* **2008**, *66*, 355–361. [CrossRef]

- Gündüz, G.; Korkut, S.; Korkut, D.S. The Effects of Heat Treatment on Physical and Technological Properties and Surface Roughness of Camiyani Black Pine (*Pinus nigra* Arn. Subsp. *pallasiana var. pallasiana*) Wood. *Bioresour. Technol.* 2008, 99, 2275–2280. [CrossRef] [PubMed]
- 33. Inoue, M.; Norimoto, M.; Tanahashi, M.; Rowell, R.M. Steam or Heat Fixation of Compressed Wood. *Wood Fiber Sci.* **1993**, 25, 224–235.
- 34. Baysal, E.; Kart, S.; Toker, H.; Degirmentepe, S. Some Physical Characteristics of Thermally Modified Oriental-Beech Wood. *Maderas Cienc. Tecnol.* **2014**, *16*, 291–298. [CrossRef]
- 35. Bowyer, J.L.; Shmulsky, R.; Haygreen, J.G. Forest Products and Wood Science: An Introduction, 5th ed.; Blackwell Pub: Ames, IO, USA, 2007; ISBN 978-0-8138-2036-1.
- Zaman, A.; Alén, R.; Kotilainen, R. Thermal Behavior of Scots Pine (*Pinus sylvestris*) and Silver Birch (*Betula pendula*) at 200–230. Wood Fiber Sci. 2000, 2, 138–143.
- 37. Yildiz, S.; Gümüşkaya, E. The Effects of Thermal Modification on Crystalline Structure of Cellulose in Soft and Hardwood. *Build. Environ.* **2007**, *42*, 62–67. [CrossRef]
- Nhacila, F.; Sitoe, E.; Uetimane, E.; Manhica, A.; Egas, A.; Möttönen, V. Effects of Thermal Modification on Physical and Mechanical Properties of Mozambican *Brachystegia spiciformis* and *Julbernardia globiflora* Wood. *Eur. J. Wood Wood Prod.* 2020, 78, 871–878. [CrossRef]
- 39. Gašparík, M.; Gaff, M.; Šafaříková, L.; Vallejo, C.R.; Svoboda, T. Impact Bending Strength and Brinell Hardness of Densified Hardwoods. *BioResources* 2016, 11, 8638–8652. [CrossRef]
- 40. Wagenfür, R. Holzatlas, 5th ed.; Fachbuchverlag im Carl Hanser Verlag: Leipiz, Germany, 2000.

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.