ISSN (Online): 2349-1183; ISSN (Print): 2349-9265
TROPICAL PLANT RESEARCH
The Journal of the Society for Tropical Plant Research
5(3): 343–348, 2018
DOI: 10.22271/tpr.2018.v5.i3.043

Research article

Sorption behaviour of thermally and chemically modified selected wood species

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[Accepted: 15 December 2018]

Abstract: This study investigates the sorption behaviour of thermally and chemically modified selected wood species. Wood samples of dimensions 20 mm × 20 mm × 20 mm were used in this study. The samples were oven dried and thermally treated at temperatures of 160°C, 180°C and 200°C for 30 minutes. Another set of wood samples were prepared for chemical treatment (Acetylation) inside an oven at 80°C for 180 min. The colour varied from light cream to slightly brown at 160°C and very brown at 200°C for thermally modified wood. The color of the chemical modified wood changes from being yellowish of the untreated wood to pale yellow in colour. The percentage weight loss increases with temperature from 22.62% at 160°C to 26.46% at 180°C and 20.8% for Percentage Weight Gain (PWG). The average value of water absorption ranged from 8.60 to 16 %; 26 to 40.78 %; 35 to 50.35 % and 42.88 to 57.53 % for 1 hour to 78 hrs respectively. The value for the chemically modified wood ranged between 5.22 and 5.59 %, with RH of 97% and 7% having the lowest and highest value respectively. The study revealed that there was a reduction in the weight and density of thermally treated wood as a result of thermolysis and weight was gained after chemical modification.
Keywords: Sorption - Water absorption - Temperature - Colour - Density.

[Cite as: Faruwa FA & Iyiola EA (2018) Sorption behaviour of thermally and chemically modified selected wood species. *Tropical Plant Research* 5(3): 343–348]

INTRODUCTION

Wood is a hard organic fibrous material found in the stems, branches and roots of trees and other woody plants. Wood is composed of three intimately-associated structural polymers; cellulose, hemicelluloses and lignin material which are vulnerable to degradation due to microbial and termite activities causing significant losses of wood in service (Shupe *et al*. 2007).

Wood due to its exceptional properties like good aesthetic values, easy workability, high strength to volume ratio, and good finishing (painting and polishing) has been used for various purposes like; construction, engineered wood products, furniture and utensils, arts and in sports and recreational equipment. In Nigeria, more than 80% of timber products are used for constructional purposes such as building, furniture, fuel, railways, sleepers, transmission poles, pulp and paper, plywood, veneer, composite boards, matches and fuelwood (Akanbi & Ashiru 2002).

Although wood continues to be used for many excellent material properties, it also suffers from a number of disadvantages. These disadvantages among others are; dimensional changes in response to altering atmospheric conditions, susceptibility to biological attack and changes in appearance when exposed to weathering. All these, place restrictions on the potential end-usage of wood (Hill 2006). As reported by Stamm (1964), biological degradation experienced in wood is on the account of moisture gained by wood through the hydrogen bonding on exposure to moisture, which makes the wood dimensionally unstable and also spawning biological agents. Wood exposed in exterior conditions is subject to degradation by the ultraviolet component of the solar spectrum. This degradation is essentially confined to the lignin component of wood, resulting in the gradual release of polysaccharide-rich wood cells, which are subsequently removed from the wood surface by wind and rain erosion (Hill 2006). Therefore, because of these negativities confronting wood, there is a need to enhance

Received: 13 March 2018 Published online: 31 December 2018
https://doi.org/10.22271/tpr.2018.v5.i3.043

its usability through modification via chemical modification and/or plasma modification.

Chemical modification is a reaction between some reactive part of the wood and a simple single chemical reagent, with or without the catalyst, to form a covalent bond between the two (Rowell 2005).This process exclude impregnation, coating among others. The most abundant single site for reactivity in these polymers is the hydroxyl group which makes the wood dimensionally unstable, as regards to its sorption behaviour and durability.

Thermal modification of wood is controlled pyrolysis process of wood to include some chemical changes to the chemical structures of the cell wall component and to increase its durability (Tiemann 1915). Kollmann & Fengel (1965) reported that, thermal modification of wood affects predominantly hemicelluloses; this process of thermal degradation of compounds begins at 120°C, and its intensity is proportional to the temperature gradient. Also, Boonstra *et al*. (2007) and Windeisen *et al*. (2009) observed that acetic acid is released during thermal modification via the hydrolysis of hemicelluloses. This Acetic acid plays a major role in the depolymerisation of cellulose and thus, increases its crystallinity. As observed and reported by Mazela *et al*. (2004), Peters *et al*. (2009) and Mohareb *et al*. (2012), the major impact of thermal treatments of wood is to decrease the number of hydroxyl groups responsible for bonding water molecules. When the hemicelluloses content are significantly decreased, the capacity for moisture adsorption of the material decreases accordingly and this will improves the dimensional stability and resistance to microbiological degradation.

Therefore, with these two major types of wood modification, these negativities befalling wood and especially dimension stability (sorption behaviour) would be combated. Hence, this study focused on the sorption behaviour of thermally and chemically modified selected *Entandrophragma utile* (Dawe & Sprague) Sprague wood species. *Entandrophragma utile* has light brown sapwood and is clearly differentiated from the heartwood. When newly cut, the heartwood is pinkish-brown, but it darkens to a deep red-brown on exposure. The grain is typically interlocked with a medium texture. Quarter sawn surfaces can display irregular wide stripe or ribbon figure. *E. Utile* is more durable and often used for external joinery such as windows and doors.

METHODOLOGY

Preparation of Wood Samples

Commercial lumber of *Entandrophragma utile* (Dawe & Sprague) Sprague wood used for this study was obtained from a commercial sawmill (Njoku and Sons Ltd. Naze, Owerri Imo State, Nigeria).Wood samples of dimensions 20 mm × 20 mm × 20 mm were used in this study. The substrate (wood samples) that were used were determined based on density determination of different species of wood in the market. The chemical used for different relative humidity test were: sodium hydride, potassium nitrate, sodium nitrate, ammonium sulphate, copper sulphide while the chemicals used for modification were acetic acid and acetic anhydride.

The wood samples were air-dried to approximately 20% moisture content and machined into the required dimensions in the direct parallel to grain with a circular saw. Twenty specimens of dimensions 20 mm × 20 mm × 20 mm (length × breadth × thickness) were prepared. The specimens were oven dried at 105^{o}C until the constant weight of 12 % was achieved; thereafter, the dimensions and weight of specimens were measured.

Heat Treated Process

Heat treatment was carried out in a closed process vessel a muffle furnace with a temperature controlled heating unit. Wood conditioning went through thermal modification at different temperature. The furnace was ramped to desire temperature (160°C, 180°C and 200°C) before introducing wood samples. The samples were heated for 30 mins. At the end of the furnace conditioning, the conditioned wood samples were cooled in a desiccator over silica gel before they were subjected to further analysis.

Chemical Modification of Wood Specimens

In the chemical modification**,** set of selected wood samples were submerged in a preheated 70% of acetic anhydride and 30% of acetic acidic solution contained in a closed stainless container placed inside an oven at 80^{o}C for 180 min, under the atmospheric pressure and in the air. The wood samples were air-dried for 2 mins to remove excessive moisture content in the wood sample and cooled.

The volume and the weight of specimen were determined after the samples had been cooled over silica gel in desiccators for their dry weight. The percentage weight gain was determined using equation 1.

$$PWG\ (\%) = \frac{w_t - w_0}{w_0} \times 100 \qquad \text{Equation 1}$$

Where, w_o (g) is the oven-dry weight of specimens before treated and w_t is the oven-dry weight of specimens

after treatment. The reaction for acetylation of wood is shown below:

$$\text{Wood-OH} + CH_3\text{-C (=O)-O-C(C=O)-}CH_3 \longrightarrow \text{Wood-O-C (=O)-}CH_3 + CH_3\text{-C (=O)-OH}$$

Wood Acetic Anhydride Acetylated Wood Acetic Acid

Percentage weight gain (PWG) is the mass of chemical reagent retained in a sample as a percent of the original theoretical oven-dry mass of the sample.

Physical Properties Test

i. Dimensional Stability Tests: Modified and untreated wood were submerged in distilled water in a plastic container. A metal sheet was placed over the samples to hold them at approximately 2.5 cm below the surface but this did not add or have any impact on the samples. Mass and volume measurement were measured. Weight and volume gained were measured after 24, 48 and 72 hours respectively. At the end of 72 hours, the sample were removed drained of its excess water, oven dry and then cooled over silica gel in a desiccator.

ii. Sorption Behaviour Test: For evaluation of sorption behaviour tests, fifteen substrates were used and subjected to different humidity (Sodium hypochloride 7%, Potassium nitrate 48%, Sodium nitrate 65%, ammonium sulphate 80% and copper sulphide 97%). The samples used in this study (control and modified wood) were subjected to the same humidity so as to maintain one source of variation Wood samples were kept in the desiccator that contains saturated solution and left in there until it has attained equilibrium moisture content then moved to the next solution. Data in form of weight and volume were taken as samples from one solution to the other. The dimension of wood samples for the test was 20 mm × 20 mm × 25 mm. Data collected were then processed. Randomized Complete Block Design (CRD) was used to test for the significance of the different treatment variables. Treatment means were separated using the Analysis of Variance (ANOVA). For data obtained from this study using Excel and Statistical Package for Social Sciences (SPSS) Range test at a= 0.05. The follow-up test was conducted to determine the difference between means and choose the best treatment.

RESULTS AND DISCUSSION

Results of the sorption behaviour of thermally and chemically modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood for the experiment were presented using statistical models, tables and percentages as relevant to the objectives of the study.

Physical Properties

The results of the visual observation of colour changes for the thermally modified wood at two different temperature and duration levels are presented. The results of the water absorption (WA), Percentage Weight Gain (PWG) and Percentage Weight Loss (PWL) of the modified *Entandrophragma utile* wood were presented in tables.

Appearance

The visual observation revealed that thermally treated *Entandrophragma utile* varied in relation to temperature. Also, the colour after chemical modification was altered. The colour varied from light cream to slightly brown at 160°C and very brown at 200°C for thermally modified wood. The color of the chemical modified wood changes from being yellowish of the untreated wood to pale yellow in colour. The colour of the control remained creamy. This degree of colour change can be attributed to some factors such as the chemical composition of the extractives, wood pH, drying temperature, time and heating medium which can cause hydrolysis and/or oxidation of wood components which corroborate the report of Sehlstedt-Persson (2003), Owoyemi & Iyiola (2016). This colour change can be attributed to some chemical reactions that took place during the heat process. The color change was noted throughout the cross-section, with the intensity being greater on the surfaces than in the core. For a given treatment time, the amount of color change was greater for specimens treated at 200ºC than for specimens treated at 160ºC at the same time duration. However, as observed in this study, temperature regimes were found to have profound impact that time used on all the samples. The changes in the color of thermally modified wood are attributed to oxidative changes, which predominate over hydrolysis reactions (Owoyemi & Iyiola 2016). Furthermore, as reported by Owoyemi & Iyiola (2016), the un-extracted and extracted acetone presents in the heat-treated wood could be the reason for varying colour after the treatment. They further conclude that both the extractives and the structural component (hemicelluloses and/or lignin) possibly took part in colour change experienced. Their observation also held for this study; the treated

wood was affected by thermal degradation which made the wood darker due to thermal degradation of lignin.

Percentage Weight Loss and Percentage Weight Gain

The result of Percentage Weight Loss (PWL) and Percentage Weight Gain (PWG) is presented in table 1. The result gave a clear indication that PWL increases with temperature *i.e.* from 22.62% at 160°C to 26.46% at 180°C and 20.8% for PWG. This result agreed with the finding of Iyiola *et al.* (2017) on the impact of heat treatment on physico-mechanical properties of thermally modified *Anthocleistha djalonensis* A.Chev. wood that weight loss increases with increase in temperature and time. Statistically, the result of ANOVA revealed that there were no significant differences recorded within the temperature range at P values ≤ 0.05 (Table 2). The result of this change can be attributed to the various chemical changes that occur during thermolysis of wood such as degradation of hemicelluloses, loss of water and volatile extractives and cellular breakdown of cell wall polymers as reported by Hill (2006). The weight gain was as a result of replacing of hydroxyl group with acetyl group at the sorption site and reduction in monolayer water sorption for acetylated wood. Several studies have shown that if the wood is chemically modified well enough it will be highly durable compared to durability class 1 *i.e.* it will be able to stand rottenness for up to 25 years.

Table 1. Percentage Weight Loss for thermally modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood.

Treatment	Mean	Std. Error
160°C	22.62	1.12
180°C	26.47	0.97
200°C	24.53	1.73
Chemical Treatment (PWG)	20.84	3.05

Table 2. ANOVA of percentage weight loss for thermally modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood.

	Sum of Squares	Df	Mean Square	F	Significance
PWL	110.961	2	55.480	2.137	**0.131**
Error	1090.515	42	25.965		
Total	**1201.476**	**44**			

Dimension Stability

The result of water absorption is presented in table 3. The result revealed that the value ranged from 8.60 to 16 %; 26 to 40.78 %; 35 to 50.35 % and 42.88 to 57.53 % for 1 hour to 78 hrs respectively. The result showed that all through the period of water immersion, the woods that were chemically modified had the lowest rate of water absorption. This is closely followed by thermally modified wood at 200°C**.** The result of ANOVAs as shown in table 4 showed that there was a significant difference among various treatment and duration of immersion. While the relationship between time and treatment as no significant difference in water absorption at P value ≤ 0.05. The decrease in the hygroscopicity of thermal and chemical modified wood is attributed to the decrease in the hemicellulose content as evident in the infrared spectra in the as shown by studies. Although in the hygroscopic ranges, the moisture content of specimens is dominated by the number of hydrophilic sites in wood, especially hydroxyl groups of carbohydrate, WA after immersion is mainly determined by the permeability of wood (Ilker & Arif 2000). The main factor influencing the permeability of wood is the size and volume of the gross capillary system comprising the vessels and pits. When wood is subjected to thermal modification, lignin softens and blocks the cell pores probably decreasing the radius and number of effective openings on pit membranes (Kocaefe *et al.* 2008). This could be one of the contributing factors to the reduction in water absorption of thermally modified wood.

Table 3. Water absorption of thermally modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood.

Treatment	Hours/WA (%)			
	1	**24**	**48**	**72**
Control	16.00±6.54	40.78±4.70	48.57±3.62	51.49±3.57
160 °C	11.96±3.04	37.85±9.10	50.35±11.46	57.53±11.92
180 °C	9.75±2.75	33.12±10.23	42.69±10.96	47.90±9.89
200 °C	11.52±0.99	33.09±3.76	43.38±4.41	49.19±4.43
Chemical	8.60±0.95	26.11±1.54	35.79±0.46	42.88v0.46

Note: Values are mean ± Stdv.

Adsorption

The result of the rate of adsorption for modified and unmodified wood is presented in table 5. The result showed an increase increases relative humidity. For the chemically modified wood the value ranged between

5.22% and 5.59%, with RH of 97% and 7% having the lowest and highest value respectively. The ANOVAs as presented in table 6 showed that there was a significant difference on the effect of treatment on rate of adsorption. While the different RH used as no significant effect on adsorption rate.

The weight of the wood increases as it moves from low relative humidity to a higher one, which contains different salt constant like sodium hypochloride, potassium nitrate, sodium nitrate, ammonium sulphate, copper sulphide. The effect of the chemical modification of wood on different relative humidity after the chemical modified wood has been modified with a mixture of 30% of acetic acid and 70% acetic anhydride and exposed to different relative. Ranging from 7% of sodium hypochloride to 48% of potassium nitrate, 65% of sodium nitrate, 80% of ammonium sulphate and 97% of copper-sulphate. The weight of the wood increases as it move from a low relative humidity to a higher one for thermally modified wood and decreases for chemically modified wood untreated wood samples.

Table 4. ANOVA of water absorption for chemically and thermally modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood.

Source	Sum of Squares	Df	Mean Square	F	Sig.
Treatment	1015.876	4	253.969	4.726	0.003
Time	12788.203	3	4262.734	79.322	0.000
Treatment *time	187.887	12	15.657	0.291	0.987
Error	2149.597	40	53.740		
Total	**16141.563**	**59**			

Note: Values are mean ± Stdv.

This is an indication that thermal modification *Entandrophragma utile* at different relative humidity has an effect on the lignin concentration. The results clearly indicate that thermal modification significantly reduces moisture adsorption in wood. In general, the WA of thermally modified wood increases with an increase in the relative humidity. However, at different relative humidity, there was no significant difference ($p>0.05$). This indicates that an increase in the different relative humidity has more impact on water absorption of chemically modified wood compared to the thermally modified wood.

Table 5. Adsorption Rate for chemically and thermally modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood Under Different Relative Humidity.

Treatment	Relative Humidity (%)				
	7	**48**	**65**	**80**	**97**
Control	5.58±0.09	5.38±0.07	5.19±0.16	5.24±0.37	5.21±0.35
160 °C	4.34±0.33	4.57±0.40	4.53±0.31	4.65±0.39	4.71±0.48
180 °C	4.41±0.39	4.45±0.29	4.56±0.08	4.59±0.08	5.62±0.08
200 °C	4.36±0.20	4.37±0.04	4.48±0.04	4.54±0.08	4.65±0.09
Chemical	5.59±0.45	5.38±0.36	5.22±0.30	5.22±0.30	5.22±0.30

Note: Values are mean ± Stdv.

Table 6. ANOVA of Percentage Weight Gain for chemically and thermally modified *Entandrophragma utile* (Dawe & Sprague) Sprague wood.

Source	Sum of Squares	Df	Mean Square	F	Sig.
Treatment	10.852	4	2.713	34.541	**0.000**
RH	0.028	4	0.007	0.089	**0.986**
Error	5.184	66	0.079		
Total	**16.064**	**74**			

CONCLUSION

Sorption behaviour of modified wood was investigated in this study using heat and chemical. The study revealed that there was a reduction in the weight and density of thermally treated wood as a result of thermolysis and the weight gain from chemical modification was above 20%. This is an indication that wood properties were found to be improved in terms of water absorption and bulkiness. It was found also that, water permeability is based on the moisture content which is influenced by modification and not the relative humidity. From the result obtained in this study, it was clear that chemical and thermal modification control can be used to increase morphological properties of wood which utilization.

ACKNOWLEDGEMENTS

The authors sincerely acknowledge the Technologist of the Department of Forestry and Wildlife Technology, Owerri, Nigeria for their time and advice in the course of this study.

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