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Reducing Hygroscopicity of *Triplochiton scleroxylon* K. Schum. by Thermal Modification

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Abstract

The thermal modification of wood is a potential alternative method for improving wood dimensional stability. However, during thermal modification, morphological changes occur within the cell wall structure, and these confer different properties to the wood. This study investigated the effects of the thermal modification process on reducing hygroscopicity of *Triplochiton scleroxylon* wood in terms of water absorption (WA), volumetric swelling (VS), water repellent efficiency (WRE) and anti-swelling efficiency (ASE). Three different temperatures (140 °C, 170 °C and 200 °C) at a varying duration of 3 h, 6 h and 12 h were considered for the thermal modification process. *T. scleroxylon* wood modified at 200 °C had the lowest WA of 41.99% and 78.25% after 24 h and 168h respectively among all the modified samples while untreated samples had the highest WA of 59.38% and 114.16% after 24 h and 168h respectively. Heat-treated samples had lower VS between 5.47- 8.90% after 168 h when compared with the untreated samples with 14.89%. WRE and ASE values also increase considerably with an increase in treatment temperature. This has shown improvement in the physical properties of *Triplochiton scleroxylon* making it suitable for outdoor purposes as a result of a reduction in the hygroscopicity of the wood after thermal treatment.

Keywords: Thermal modification, water absorption, swelling, *Triplochiton scleroxylon,* Water Repellent Efficiency

Introduction

Wood is an ancient renewable material of biological origin with many different areas of application. Its physical and mechanical properties characterize wood as the most versatile material in biomaterials science. The obvious drawback of wood as an engineering material is its hygroscopicity. Under variations in relative humidity, wood readily absorbs or releases moisture, resulting in dimension swelling and shrinkage. In this scenario, the wood structure will experience fungal deterioration and cracking, posing hidden risks to wood constructions (Keplinger et al. 2015). The key factor responsible for the unfavourable features of wood is the hydrophilic hydroxyl groups (-OH) in the three wood polymeric components (cellulose, hemicellulose, and lignin). Through hydrogen bonding from the surrounding environment, the hydroxyl group of wood attracts water molecules causing swelling and making it dimensionally unstable (Islam et al. 2012). The decrease in the number of accessible hydroxyl groups of the cell wall to water as a result of the degradation of carbohydrates (mainly hemicelluloses), and also by crosslinking reactions of the cell wall matrix leads to a reduction in the equilibrium moisture content (EMC) of the wood (Esteves *et al*. 2007). The reactions such as this cross-linking usually lead to a reduced volume of the adsorbed water between adjacent wood polymers in the cell wall and this with the loss of hemicelluloses creates a rigid matrix that prevents the expansion of the pores in the cell wall during water adsorption (Jalaludin et al. 2010), and consequently giving rise to enhanced dimensional stability of wood (Hill 2006).

Several methods of wood modifications have been used to minimize the dimensional changes of wood due to atmospheric moisture and improvement on the inherent properties of wood polymers and their structures. The various wood modification techniques include thermal, hydrothermal, chemical, mechanical (densification) and plasma modifications (Hill, 2006; Adebawo et. al, 2016). Thermally modified woods are environmentally friendly with high dimensional stability, low moisture absorption, and resistance to decay and insects (Hill, 2006).

Over the years, thermal modification of wood has been the subject of increasing interest by scholars and is currently considered one of the most promising non-biocide alternatives to improving the performance of wood species with low natural durability (Militz 2002; Esteves and Pereira, 2009; Gérardin, 2016). Thermal modification is known as heat treatment and is generally carried out at a temperature of 140 - 260 °C for a few hours (Hill, 2006) resulting in hemicellulose degradation. This causes the number of hydroxyl groups of cellulose and hemicelluloses to decrease and the formation O-acetyl groups thereby resulting in a decrease in the adsorption of water (Mitsui *et al.*, 2008).

The cross-linking occurring between carbohydrate polymers lignin, gave rise to an increase in the crystallinity of amorphous cellulose, thus improving dimensional stability and decreasing the hygroscopicity of wood. Therefore, heat-treated wood is expected to become more hydrophobic with an increase in treatment temperature (Tjeerdsma *et al.* 2005). Previous research studies in thermal modification have centred on the use of softwoods (Ates et al. 2009; Korkut et. al, 2008; Ponksac et al., 2006), which are not native to countries with tropical weather. Also, studies on the effects of heat treatment at different temperatures and varying heat treatment times on the water absorption of tropical hardwood species are rather limited. This work, therefore, examined the hygroscopicity of thermally modified *Triplochiton scleroxylon* K. Shum wood.

Material and Methods

Wood Preparation

The wood blocks for this study were obtained from the 22 years old tree of *Triplochiton scleroxylon* at breast height and dimensioned to 20 × 20 × 60 mm (radial × tangential × longitudinal) of which forty-five (45) samples with no defects were selected. The samples were weighed, and their weight recorded, oven dried at 105±2 °C, and weighed again to determine their moisture content before heat treatment.

Thermal Modification Process

Thermal modification of the *Triplochiton scleroxylon* wood was carried out in a Muffle furnace with a temperature-controlled heating unit. The moisture content of the samples was conditioned to 12% and then subjected to heat treatment at varying temperatures of 140, 170 and 200 °C for 3, 6 and 12 h. Hence, the temperature in the furnace was adjusted to the actual temperature at which the treatment would occur before the introduction of the wood samples and this was done for each treatment. After each heat modification period, thermally modified samples were removed from the furnace, cooled and weighed.

Dimensional stability Test

Dimensional change (swelling) due to treatments was assessed with oven-dried (103°C) samples**.** Water absorption, Volumetric swelling**,** Anti-swell efficiency (ASE) and water-repellent efficiency (WRE) for both thermally modified and unmodified wood were measured according to ASTM -1037 (1999). Five oven-dried wood specimens for each treatment were soaked in a water bath at a temperature of 20±1 °C for 24, 48, 72, 96, 120, 144, and 168 h; the weight and dimension of wood specimens were determined before and after soaking.

Water Absorption (WA) and Volumetric swelling (VS) were determined using Eqn. (1) and (2) respectively.

$$WA\ (\%) = \frac{W_2 - W_1}{W_1}\ X\ 100 \qquad (1)$$

where,
WA= water absorption;
W_2 = weight of specimen after water soaking,
W_1= weight of specimen before water soaking.

$$VS\ (\%) = \frac{V_2 - V_1}{V_1}\ X\ 100 \qquad (2)$$

where, VS= volumetric swelling;
V_1= volume of wood before soaking,
V_2= volume of wood after soaking

Anti-swell efficiency (ASE) and Water repellent efficiency (WRE) was calculated according to Eqn. (3) and (4) respectively

$$ASE\ (\%) = \left(\frac{S_u - S_m}{S_u}\right) X\ 100 \qquad (3)$$

where, ASE= Anti-swell efficiency,
S_u = volumetric swelling coefficient of unmodified wood samples,
S_m = volumetric swelling coefficient of modified wood samples

$$WRE\ (\%) = \left(\frac{W_u - W_m}{W_u}\right) X\ 100 \qquad (4)$$

where, Wu= water absorption of unmodified samples,
Wm= water absorption of modified samples.

Data Analysis

Data obtained were analyzed statistically. Analysis of variance (ANOVA) was used to test significant difference among temperature (140, 170, 200 °C) and duration (3, 6, 12 h). When the ANOVA indicated a significant difference among the temperature and duration, a comparison of means was conducted, employing Duncan Multiple Range Test (DMRT) to identify which groups were significantly different at α0.05.

Results and Discussion

Water Absorption and Water Repellent Efficiency

The mean values for percentage Water Absorption (WA) and Water Repellent Efficiency (WRE) for heat-treated of *Triplochiton scleroxylon* wood soaked in water between 24 and 168 h are presented in Table 1 and 2. The WA of the untreated samples ranged between 59.38-114.16% while the WA for the treated samples across temperatures of 140, 170, and 200 °C ranged between 37.79-108.46 %, 46.46-110.81%, and 40.95-100.18 % respectively. For wood samples modified at 140 °C, as the treatment hours increase WA also increases from the period of 24 h of soaking to 168 h. Likewise, the same trend of increment in water absorption was also observed in wood samples modified at 170 °C where WA increased as the treatment duration (3-12 h) increased. However, for samples treated at 200 °C, WA decreased as treatment duration increased with 200-12 h samples having the lowest WA (78.25%) after 168 h of soaking.

The WRE range from 8.94-36.35%, 0.17-31.13%, 6.00-34.70%, 1.09-30.53%, 1.97-29.47%, 2.93-28.46% for 24, 48, 72, 96, 120, 144, 168 h respectively indicating inconsistency in the movement of water in heat-treated wood. However, after 168 h of water soak, wood samples modified at 200-12h had the highest WRE followed by 200-6h and 140-3h. Analysis of variance conducted shows that significant difference ($p<0.05$) existed between temperature (140, 170, and 200 ℃ and duration (3, 6, and 12 h) of the heat-treated wood.

Table 1: Water Absorption of thermally modified *T. scleroxylon* wood

Temperature (℃)	Duration (h)	Period of Soaking (h)						
		24	48	72	96	120	144	168
Untreated		59.38±1.39	73.33±1.92	83.29±2.44	96.40±2.28	99.86±2.65	107.71±3.18	114.16±2.99
140	3	37.79±0.85	50.18±0.93	57.37±1.08	62.95±1.15	69.37±1.35	75.97±1.57	81.68±1.71
	6	38.12±1.25	51.78±2.25	60.35±2.25	65.69±2.48	73.27±2.81	78.84±2.85	85.72±3.47
	12	52.32±1.82	71.71±4.02	83.15±4.02	90.61±4.76	98.77±4.55	104.99±4.81	108.46±4.68
170	3	46.46±0.65	59.68±0.93	65.74±0.93	70.60±0.93	77.14±0.99	83.67±1.15	87.41±1.43
	6	54.07±0.55	71.19±2.88	81.63±2.88	90.15±3.99	97.04±4.39	105.59±5.31	110.81±5.81
	12	53.33±0.71	70.76±1.77	79.57±1.77	81.95±1.82	88.47±1.90	95.82±1.77	101.98±2.16
200	3	53.94±1.92	70.23±0.75	81.07±0.75	86.87±1.08	93.30±1.18	100.18±1.45	103.82±1.63
	6	40.95±0.84	54.87±2.25	64.61±2.25	66.22±3.40	74.50±3.08	80.06±3.35	83.74±3.48
	12	41.93±1.50	56.76±0.82	62.48±0.82	66.43±0.79	71.45±0.75	79.42±2.21	78.25±0.94

**Mean±Standard Error*

Table 2: Water Repellent Efficiency of thermally modified *T. scleroxylon* wood

Temperature (℃)	Duration (h)	Period of Soaking (h)						
		24	48	72	96	120	144	168
140	3	36.35±0.28	31.57±0.49	31.13±0.49	34.70±0.46	30.53±0.53	29.47±0.64	28.46±0.60
	6	35.79±0.17	29.39±0.22	27.55±0.22	31.85±0.23	26.62±0.27	26.81±0.31	24.91±0.34
	12	11.89±0.25	2.22±0.45	0.17±0.45	6.00±0.50	1.09±0.56	2.52±0.57	4.99±0.69
170	3	21.74±0.38	18.63±0.80	21.07±0.80	26.76±0.95	22.75±0.91	22.32±0.96	23.43±0.54
	6	8.94±0.13	2.92±0.19	1.99±0.19	6.48±0.19	2.83±0.20	1.97±0.23	2.93±0.29
	12	10.17±0.11	3.52±0.58	4.46±0.58	14.99±0.80	11.41±0.88	11.04±1/06	10.67±1.16
200	3	9.15±0.14	4.25±0.35	2.67±0.15	9.89±0.36	6.570±.38	6.99±0.35	9.06±0.43
	6	31.03±0.38	25.17±0.45	22.43±0.45	31.31±0.22	25.39±0.24	25.67±0.29	26.64±0.33
	12	29.37±0.17	22.61±0.16	24.99±0.16	31.09±0.68	28.44±31.46	26.26±0.67	31.46±0.70

**Mean±Standard Error*

During the period of immersion, the amount of water absorbed for the heat-treated wood increased as the time of immersion increased thus making 24 and 168 h have the lowest and highest percentage WA. Based on this, it is evident that thermal treatment has a significant effect on the water absorption of wood. According to Pereira *et al.,* 2020, who explained that thermal modification showed an important influence on water absorption. Hence, wood exposed to high temperatures fails in its ability to reabsorb water (Kocaefe *et al.*, 2007). Esteves and Pereira (2008) explained that after the wood modification treatment, the wood shows a lower weight, less hygroscopicity, a general reduction in mechanical properties, and a darker colour. In particular, the variation in the hygroscopicity of the wood is a consequence of the reduction of the bonding sites available for water within the cell wall. Similarly, the consequence of the reduction of the bonding sites available for water within the cell wall is a result of variation in the hygroscopicity of the wood with an average decrease of 40 % in hygroscopicity (Boonstra *et al.,* 1998; Tjeerdsma *et al*., 1998).

Volumetric Swelling and Anti-swelling Efficiency

Table 3 presents the mean percentage of Volumetric Swelling (VS). The untreated wood recorded the highest VS of 9.65 % and 14.89% after 24h and 168h of soaking respectively while the VS for heat-treated samples decreases at each temperature as the hour of exposure to heat treatment increased. The VS ranged from 5.47- 8.55% to 7.03 - 11.26 % after 24 h and 168 h of water soak respectively. The wood samples treated at 200℃ for 12 h had the lowest VS of 5.47% and 7.03% after 24 h and 168 h respectively. The wood samples started swelling at a higher rate and after five days (120 h), the rate of swelling slowed down till 168 h.

Enhancement of the dimensional stability of wood occurs because volumetric swelling caused by water absorption is under control as a result of the reduction in the number of water accessible at hydroxyl sites. Kato and Cameron (1999) reported that heat treatment of wood caused the irreversible creation of new hydrogen bonds in the amorphous regions of cellulose and hemicelluloses which gave rise to a decrease in water retention of fibres due to structural modification of heat-treated wood. Generally, heat-treated wood at high temperature has lower hygroscopicity than untreated wood. Due to a decrease of hydroxyl groups on carbohydrate chains, the cell wall of heat-treated wood absorbs less water (Ates *et al.,* 2009). As a consequence of the reduced number of hydroxyl groups, swelling is reduced.

Table 4 presents the Anti-swelling Efficiency (ASE) of heat-treated wood of *Triplochiton scleroxylon* after 168 h of water soak. The ASE of wood samples treated at 140 ℃, 170 ℃, 200℃ were found to increase as the temperature increased. Treatment at higher temperatures gave rise to more dimensionally stable wood as shown by increasing ASE values (up to 53%). This is similar to the findings of Shukla (2019) who reported that the amount of swelling of *Acacia auriculiformis* wood was found to decrease with increasing treatment temperatures where the maximum dimensional stability of thermally modified wood at 240ºC was in the range of 60-65%. Moreover, in the study carried out by Repellin and Guyonnet (2005) on the effect of treatment temperature and duration on swelling behavior of heat-treated wood, they reported that increased temperature and longer duration of heat treatment gave rise to reduced wood swelling.

The result of this study is in line with other studies where lower water absorption was observed after thermal modification (Pereira et al. 2020). Many other reports have shown that the reduction in wood swelling brought about by thermal modification is not only due to the degradation of the hemicelluloses but also to a structural modification and a chemical change of lignin (Repellin and Guyonnet, 2005). The reduction in the swelling of the wood is due to various changes in the chemical structure of the wood by heat treatment. The heating process degrades hemicelluloses first resulting in the reduction of -OH bonds and the formation of O-acetyl groups. Consequently, there is cross-linking between wood fibres which makes wood to become more hydrophobic. Hence, cell-wall modifications and changes in sorption properties as a result removal of hydrophilic hydroxyl-groups bring about lower volumetric swelling and increased anti-swelling efficiency. Therefore, changed cell wall structure is attributed to reduced hygroscopicity of heat-treated wood

Conclusion

As an outcome of this study, it was discovered that heat treatment of *Triplochiton scleroxylon* wood improved significantly its dimensional stability. When compared to samples of untreated wood, the treated wood's water absorption and volumetric swelling were significantly reduced. Additionally, heat-treated wood outperformed untreated wood in terms of its ability to repel water and resist swelling. Reduced water absorption and swelling, as well as higher water repellent and anti-swelling performance of heat-treated wood, were the results of reduced adsorption sites and structural changes during the heat modification process.

Table 3: Volumetric swelling of thermally modified *T. scleroxylon* wood

Temperature (°C)	Duration (h)	Period of Soaking (h)						
		24	48	72	96	120	144	168
Untreated		9.65±0.77	10.37±0.82	10.50±0.87	11.61±0.93	12.72±0.98	13.86±0.43	14.89±1.09
140	3	6.77±0.46	7.42±0.52	7.65±0.51	7.99±0.51	8.23±0.54	9.84±0.50	10.89±0.48
	6	7.04±1.05	8.54±1.15	8.91±1.15	9.03±1.18	9.18±1.22	10.25±1.20	11.26±1.20
	12	6.30±0.23	6.99±0.23	7.13±0.27	8.27±0.21	9.31±0.28	9.81±0.26	10.40±0.29
170	3	8.55±0.51	8.96±0.52	9.11±0.55	9.38±0.60	9.58±0.65	9.80±0.61	9.94±0.71
	6	8.90±0.58	9.38±0.72	9.81±0.80	10.08±0.66	10.43±0.65	10.79±0.61	10.96±0.60
	12	7.17±0.98	7.64±0.21	8.28±0.54	8.71±0.07	9.14±0.15	9.75±0.15	9.90±0.13
200	3	8.78±0.81	9.09±0.84	9.51±0.93	10.24±0.97	10.51±0.96	10.80±0.96	10.92±1.00
	6	6.39±1.19	6.61±0.33	7.03±0.31	7.87±0.39	8.22±0.38	8.27±0.36	8.43±0.36
	12	5.47±0.59	5.93±0.61	6.14±0.43	6.55±0.43	6.88±0.60	7.00±0.32	7.03±0.39

**Mean±Standard Error*

Table 4: Anti-swell efficiency of thermally modified *T. scleroxylon* wood

Temperature (°C)	Duration (h)	Period of Soaking (h)						
		24	48	72	96	120	144	168
140	3	29.92±0.43	28.38±0.31	27.14±0.24	31.23±0.04	35.35±0.66	29.02±0.30	26.86±0.67
	6	27.07±1.45	17.58±0.47	15.13±0.88	22.27±0.47	27.83±0.68	26.03±0.44	24.35±0.49
	12	32.61±0.11	32.03±0.59	28.77±1.09	26.79±0.59	29.18±0.80	29.18±0.80	30.17±0.85
170	3	11.47±0.22	13.58±0.03	13.26±0.56	19.25±0.03	24.67±1.16	29.31±0.56	33.19±0.88
	6	7.82±1.13	9.53±0.30	6.55±0.37	13.18±1.34	17.97±0.85	22.12±0.10	26.38±0.22
	12	25.70±1.21	26.32±0.81	21.14±0.74	24.96±0.81	28.14±0.74	29.62±0.31	33.54±0.23
200	3	9.02±0.10	12.22±0.51	9.39±0.16	11.82±0.51	17.37±0.26	22.20±0.68	26.63±0.77
	6	33.81±0.18	36.27±0.70	33.03±0.71	32.28±0.70	35.37±0.88	40.30±0.48	43.38±0.83
	12	43.32±0.32	42.77±0.23	41.50±0.27	43.56±0.23	45.91±0.25	49.44±0.71	52.77±0.65

**Mean±Standard Error*

References

Adebawo, F., Naithani, V., Sadeghifar, H., Tilotta, D., Lucia, L., Jameel, H., & Ogunsanwo, O. (2016). Morphological and interfacial properties of chemically modified tropical hardwood. RSC Advances, 6(8): 6571-6576.

ASTM D1037 (1999). Standard test methods for evaluating properties of wood-based fiber and particle panel materials, Philadelphia, PA: American Society for Testing and Material, 1999.

Ates, S., Akyildiz, M.H., and Ozdemir, H. (2009). Effects of Heat Treatment on Calabrain Pine (*Pinus Brutia* Ten.) Wood. *BioResources* 4(3), 1032-1043.

Boonstra M.J., Tjeerdsma, B.F., and Groeneveld, H.A.C. (1998). Thermal modification of non-durable wood species. 1. *The PLATO Technology: Thermal Modification of Wood,* International Research Group on Wood Preservation, Document no: IRG/WP 98-40123.

Esteves, B., and Pereira, H. (2009). "Wood modification by heat treatment: A review," BioResources 4(1), 370-404.

Esteves, B., Marques, A. V., Domingos, I., and Pereira, H. (2007). "Influence of steam heating on the properties of pine (Pinus pinaster) and eucalypt (Eucalyptus globulus) wood," Wood Sci. Technol. 41(3), 193-207.

Gérardin, P. (2016). New alternatives for wood preservation based on thermal and chemical modification of wood - a review. Annals of Forest Science, DOI 10.1007/s13595-015-0531-4.

Hill, C.A.S. (2006). Wood Modification: Chemical, Thermal and Other Processes; John Wiley & Sons, Ltd.: Chichester, UK, 357p

Islam Md. S., Hamdan S., Rusop M., Rahman Md. R., Ahmed A. S., and Mohd Idrus M. A. M., (2012). Dimensional Stability and Water Repellent Efficiency Measurement of Chemically Modified Tropical Light Hardwood. BioResources 7(1), 1221-1231

Jalaludin, Z., Hill, C.A.S, Xie, Y., Samsi, H.W., Husain, H., Awang, K., Curling, S.F. (2010) Analysis of the water vapour sorption isotherms of thermally modified acacia and sesendok. Wood Mater. Sci. Eng. 5:194-203

Kato, K. L.; Cameron, R. E. (1999). A Review of the relationship between thermally accelerated ageing of paper and hornification. *Cellulose* 6(1):23-40.

Keplinger, T., Cabane, E., Chanana, M., Hass, P., Merk, V., Gierlinger, N., Burgert, I. (2015) A versatile strategy for grafting polymers to wood cell walls. *Acta Biomater.* 11:256–263

Kocaefe, D., Younsi, R., Poncsak, S., and Kocaefe, Y. (2007). Comparison of different models for high-temperature heat treatment of wood. *Int. J. Therm. Sci.* 46, 707-716.

Korkut, S., Akgül, M., and Dündar, T. (2008). "The effects of heat treatment on technological properties of Scots pine (*Pinus sylvestris* L.) wood," *Bioresource Technol.* 99(6), 1861-1868.

Militz, H. (2002). Thermal treatment of 327 wood: European processes and their background, The International Research Group on Wood Preservation. IRG/WP 02- 40241.

Mitsui, K.; Inagaki, T.; Tsuchikawa, S. (2008). Monitoring of hydroxyl groups in wood during heat treatment using NIR spectroscopy. *Biomacromolecules* 9: 286–288.

Pereira, G. F, Iwakiri S., Trianoski R, D'angelo Rios P., Raia R. Z. (2020). Influence of Thermal Modification on the Physical and Mechanical Properties of *Eucalyptus badjensis* Mixed Particleboard / Osb Panels. *FLORESTA*, Curitiba 51(2):419-428,

Poncsak, S., Kocaefe, D., Bouazara, M., and Pichette, A. (2006). "Effect of high temperature treatment on the mechanical properties of birch (Betula papyrifera),"*Wood Sci. Technol.* 40(8), 647-663.

Repellin, V.; Guyonnet, R. (2005). Evaluation of Heat-Treated Wood Swelling by Differential Scanning Calorimetry in Relation to Chemical Composition. *Holzforschung*, 59, 28–34.

Shukla S.R. (2019). Evaluation of Dimensional Stability, Surface Roughness, Colour, Flexural Properties and Decay Resistance of Thermally Modified *Acacia Auriculiformis. Maderas. Ciencia y tecnología* 21(4): 433 – 446.

Steves, B., Graca, J., Pereira, H. (2008) Extractive composition and summative chemical analysis of thermally treated eucalypt wood. *Holzforschung* 62:344-351

Tjeerdsma, B., and Militz, H. (2005). "Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood," Holz. Roh. Werkst. 63(2), 102-111.

Tjeerdsma, B.F., Boonstra, M.J., and Militz, H. (1998). Thermal modification of non-durable wood species. 2. *Improved Wood Properties of Thermal Treated Wood.* IRG 98-40124.