

WATER ABSORPTION, ANTI-SWELL EFFICIENCY, AND DIMENSIONAL STABILITY PROPERTIES OF NEEM SEED-OIL TREATED WILD GROWN *BAMBUSA VULGARIS* SCHRAD. EX J.C. WENDL. IN SOUTHWEST NIGERIA

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Bamboo culm is a hygroscopic lignocellulosic material. Hygroscopic properties may be disadvantageous in bamboo material if applied in certain applications and modes where and when extreme moisture variations are likely to occur. This study was aimed at evaluating the moisture absorption and resistance including dimensional stability properties of neem seed oil-treated split bamboo samples using two methods of treatment. Split bamboo samples from the same source were oven-dried at $103\pm 2^{\circ}\text{C}$, conditioned to 11.76% mean moisture content, and treated by completely soaking them in oil at room temperature for 24 hours and by soaking in hot oil at 60°C for 4 hours, with untreated samples as control. Results showed that samples soaked in hot oil at 60°C for 4 hours had both the least percentage water absorption and higher anti-swell efficiency, followed by samples soaked in oil at room temperature for 24 hours. Shrinkage in the longitudinal, radial, and tangential directions also followed the same trend. Conclusions and recommendations were made in line with the outcome of the study.

Key words: Bamboo; Hygroscopic lignocellulose; Water absorption; Shrinkage; Swelling; Neem seed oil

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INTRODUCTION

Bamboo is one of the lesser known/lesser used non-wood renewable natural resources that have long been identified for multi-purpose uses (Sattar 1996; Sekar et al. 1998) having long history of utilization in many parts of the world, with relics from bamboo mats and baskets dated at the Younger Stone Age between 3,300 and 2,800 BC already obtained (Ding 1996). The importance of these plant species for multi-purpose uses might have stimulated and encouraged their use for many applications in many countries (Gielis 2002; Chand et al. 2006; Nordin et al. 2007) ranging from food, handicraft, furniture, household utilities, fish traps, to various industrial products.

Bamboo is a lignocellulosic material whose dimensional stability is dependent on its state and the prevalent surrounding environmental conditions. Extreme variations in these surrounding conditions e.g. temperature, relative humidity (RH), moisture, among others, will almost always affect unprotected/untreated bamboo material in service, particularly where these variations are detrimental to certain applications. As a lignocellulosic material, it is expected that bamboo will absorb and or release moisture to

the surrounding environment at the required temperature and RH, as also influenced by the species' equilibrium moisture content (EMC) and or fibre saturation point (FSP).

Fluctuations in moisture absorption/desorption and volumetric/directional shrinkages in lignocellulosic material, particularly those in service, where these are disadvantageous, should be prevented or controlled to avoid situations where failure may occur as a result of these. Oil-based preservatives are known to be effective in this regard; although there has been growing local and global environmental and legislative pressure on the use of biocides and traditional biocide based preservatives in recent times (e.g. Homan and Jorissen, 2004). This has created new demand for alternative environmentally friendly preservation methods, especially from natural sources (Singh et al. 2006) one of which vegetable oil usage is (Leithoff and Peek 2001; Militz 2002; Razak et al. 2005; Manalo and Acda 2009).

This study was therefore aimed as contributing to these efforts by using neem (*Azadirachta indica* A. Juss) seed oil to treat bamboo (*Bambusa vulgaris* Schrad. ex J.C. Wendl.) using two different predetermined treatment methods and evaluating the water absorption and resistance including dimensional stability of oil-treated samples in comparison with control samples not treated with neem seed oil.

EXPERIMENTAL

Sourcing of Bamboo Culms and Neem Seeds

The bamboo culms that were converted and experimented upon in this study were obtained in the month of October, 2008 from a forest in Eruwa town in Oyo State (Latitude 7°31'60N and Longitude 3°25'0E) about 64.8km west of Ibadan, Nigeria. This area is located in between the humid and sub-humid tropical climate zones. The mean annual rainfall ranges from 1,117.1 to 1,693.3mm. The harvested culms had no known age or history of management. It is on record that no quantitative parameters have been established presently to identify the different growth stages of a bamboo culm for adequate harvesting purposes (Londoño et al. 2002).

In order to ensure minimal influence of age, lack of management, and other variables on the result of the research, only culms with mean circumferential length of 30cm at the second culm from the base were harvested and cross cut in such a way that only the basal culm portion of 300cm length were removed and placed in jute bags with nylon lined inner surface to avoid contamination from the soil. All the harvested culms in the bags were transported to and stored for 14 days in the wood workshop of the Department of Forest Resources Management, University of Ibadan, Ibadan, Nigeria for conversion to the test specimens.

The ripe neem seeds from which oil was mechanically extracted in this study were obtained from *Azadirachta indica* trees located on Benue Road on the University of Ibadan Campus located on the northern edge of the city of Ibadan (Latitude 7°20'0N and Longitude 3°50'0E) of about 10.4 square kilometres. Ibadan lies at 200m above sea level with a humid tropical climate (27°C average), a March – October rainy season (1250mm) followed by a mild dry season. Collection of the seeds was done by placing nylon sheets

around the stems in such a way that it covered a substantial cross sectional area of the crown in order to collect the seeds as they fall.

The seeds were sourced in the months of June to early August of 2008. The neem seeds obtained from the field were thoroughly washed using distilled water to remove dirt and other impurities and then air dried in an open space with regular movement for aeration for proper drying as suggested by Mitra (1963), a method also applied by Soetaredjo et al. (2008) to reduce the moisture content for proper crushing and to facilitate high oil volume recovery during mechanical extraction.

The seeds were stored in a nylon lined jute bags at room temperature and kept away from the reach of organisms such as rodents and other animals that can consume the seeds and also to prevent contamination, and daily air dried with proper monitoring to prevent damage as a result of possible moisture fluctuations.

Conversion of Bamboo Culms to Test Samples

The selected culms were carefully sawn with circular and vertical breakdown sawing machine longitudinally into strips. Each strip was planed on both the inner and outer surface, using a planing machine, in order to obtain the bamboo timber with mean culm thickness of 5 ± 0.5 mm for the tests. Bamboo timber, according to Chand et al. (2006) is the part between the bamboo skin and the pith. Bamboo skin is the outermost part of cross-section of stem wall, where no vascular bundles are seen, while pith is the part of stem wall next to bamboo cavity, and this also does not contain vascular bundles (Chand et al. 2006).

After conditioning in the laboratory for 14days, the strips were converted to test specimens. The specimens with dimensions 20mm (tangentially) x 60mm (longitudinally) x 5mm (radially) were oven-dried and stabilized in the laboratory to 11.76% mean moisture content prior to testing for water absorption, anti-swell efficiency, and tangential, radial, and longitudinal directional shrinkage tests. This dimension was used for these tests because it was difficult, and to the best of authors' knowledge, impossible to obtain *Bambusa vulgaris* with culm thickness of up to or more than 25mm in this part of the world irrespective of age and position along the culm. It is also noteworthy that presently there is still no universally accepted standard for determining many of the physical and mechanical properties of bamboo.

Extraction of Neem Seed Oil and Treatment of Bamboo Samples with it

There are several methods of obtaining oil from the seeds of neem, such as mechanical pressing, supercritical fluid extraction, and solvent extraction (Puri 1999). Mechanical extraction is the most widely used method to extract neem oil from the seeds (Fasina and Ajibola 1989; Puri 1999), since this method is effective for seeds containing 30-70% oil (Ketaren 1986). However, the oil produced with this method may have a low price, since it is turbid and contains a significant amount of water as compared to those obtained by supercritical fluid extraction and solvent extraction (Liau et al. 2008).

The neem seeds were manually cracked and separated from the shell, cleaned from dirt, then dried in the open air. Dried kernels were carefully ground into paste using a seed grinder to smaller particles ensuring no significant loss of seeds' oil. Mechanical

extraction of oil was performed by cold pressing. This method of oil extraction under cold pressing was adopted in this research at maximum pressure of 4500psi.

Mechanical extraction was performed at this pressure until the oil stopped flowing out. This is to allow for easy adoption by most of the target end-users, particularly those in the rural areas. The mechanical extraction has several advantages compared to the other methods, such as simple equipment and low investment, low operating cost, and the oil does not undergo a solvent separation process, among others (Fasina and Ajibola 1989).

Bamboo samples for these tests were sterilized by oven-drying at $103\pm 2^{\circ}\text{C}$ for 2 hours, cooled in a dessicator, and subjected to two neem seed oil treatment regimes by completely submerging the samples in the oil, i.e. soaking of samples in oil for 24 hours and soaking in hot neem seed oil at 60°C for 4 hours, and allowed to cool at room temperature with untreated samples serving as control. Some of the physico-chemical properties of the extracted neem seed oil are: specific gravity was 0.91; iodine, acid, saponification, and peroxide value were $93.12\text{g}100\text{g}^{-1}$, $18.24\text{m}g\text{g}^{-1}$, $172.88\text{m}g\text{g}^{-1}$ and $1.42\text{m}g\text{g}^{-1}$ respectively. These values were obtained at room temperature of $25\pm 2^{\circ}\text{C}$.

Moisture Content Determination for Bamboo Samples

Test specimens of dimensions 20mm x 60mm x 5mm were used to determine moisture content using oven-drying method in accordance with ASTM D 4442-84, using the equation,

$$MC = [(W_m - W_o) / W_o] \times 100 \quad (1)$$

where: MC = Moisture content; W_m = Weight of specimens before oven-drying (g); and W_o = Weight of specimens after oven-drying (g).

Water Absorption and Anti-Swell Efficiency Tests

Sets of oil-treated and untreated bamboo specimens (20mm × 60mm × 5mm) were placed in different beakers for the different test samples, filled with distilled water. There was replacement of water with fresh distilled water daily during 14days. The samples were weighed and water absorption values (WA) and anti-swell efficiency (ASE) were calculated according to equations (2) and (3) after each water replacement, in line with procedure also adopted by Temiz et al. (2006),

$$WA = [(W_2 - W_1) / W_1] \times 100 \quad (2)$$

$$ASE = [(S_u - S) / S_u] \times 100 \quad (3)$$

where: W_2 = wet weight of the specimen after soaking in water; W_1 = oven-dry weight; S_u = volumetric swelling of untreated samples; and S = volumetric swelling of oil-treated samples.

Determination of Percentage Shrinkage

Test specimens of dimension 20mm x 60mm x 5mm were properly aligned and coded as 'R', 'L', and 'T' for the radial, longitudinal, and tangential planes respectively. They were soaked in distilled water for 14days, replacing the water every 24hours, in order to get them conditioned to moisture above FSP. Specimens were then removed one after the other and their dimensions above FSP were taken to the nearest millimetre using digital Vernier callipers and recorded. Percentage shrinkages along the three planes were measured after specimens had been oven-dried at $103\pm 2^{\circ}\text{C}$ to constant weight as,

$$S_h = [(D_s - D_o) / D_s] \times 100 \quad (4)$$

where: S_h = shrinkage (Either S_{hR} or S_{hL} or S_{hT}) (%); D_s = dimension at saturated condition; D_o = dimension of oven dried condition; S_{hR} = radial shrinkage; S_{hL} = longitudinal shrinkage; and S_{hT} = tangential shrinkage. This is in accordance with approximations by several workers e.g. Dinwoodie (1989).

The experimental design is a split-plot completely randomized design with ten samples replication per test using a one-way analysis of variance (ANOVA) to analyse whether significant variations exist in data obtained and comparison/separation of mean done using Fisher's Least Significant Difference (LSD), both at $p < 0.05$.

RESULTS

Table 1 shows the mean percentage gain in moisture for both the oil-treated and control experimental samples and ASE for the two oil-treatment methods. Statistical analysis using ANOVA revealed that there was significant variation in the data obtained for percentage moisture gain for the treated and untreated bamboo samples at $p < 0.05$, while Tables 2 have LSD of pair of means for percentage moisture gain for the oil-treated and untreated bamboo samples respectively.

Figure 1 shows the graphical representation of the trend in moisture absorption for the control and oil-treated bamboo samples when soaked in deionised water for the 14-day period, while Fig. 2 shows the graphical representation of the trend in overall moisture loss during oven-drying of the control and oil-treated bamboo samples during directional shrinkage tests. Figures 3, 4, and 5 show the graphical representations of the trend in longitudinal, radial, and tangential shrinkages for the control and oil-treated bamboo samples, respectively.

Tabulated in Tables 3 are the mean directional shrinkage values for control and oil-treated bamboo samples. Subjecting the data obtained from these tests to ANOVA, it was observed that there was significant variation in the data obtained for the three shrinkage directions between the treated and untreated bamboo samples at $p < 0.05$, while in Table 4 is the LSD of pair of means for shrinkage values for the oil-treated and untreated bamboo samples, respectively.

Table 1. Percentage Moisture Gain after Soaking of Test Samples in Deionised Water for 14 days and Anti-Swell Efficiency Test Results

<i>Treatment</i>	<i>Mean percentage moisture gain (%)</i>	<i>Standard Deviation of mean percentage moisture gain</i>	<i>Anti-swell efficiency (%)</i>
Control	42.26	5.69	
Samples soaked in oil at room temperature for 24 hours	40.48	5.76	29.03
Samples soaked in hot oil at 60°C for 4 hours	27.47	3.07	56.09

Values are means for 10 test samples per each treatment

Table 2. Fisher's Least Significant Difference of Pair of Means for Percentage Moisture Gain for the Oil-treated and Untreated Bamboo Samples

<i>Treatment</i>	<i>Percentage moisture gain</i>
Control	42.26284 ^a
Samples soaked in oil at room temperature for 24hours	40.47528 ^a
Samples soaked in hot oil at 60°C for 4hours	27.47158 ^b

($p < 0.05$); Means with the same superscript in the same column are not significantly different

Table 3. Mean Directional Shrinkage for Control & Oil-Treated Bamboo Samples

<i>Treatment</i>	<i>Mean shrinkage values (%)</i>		
	Longitudinal	Radial	Tangential
Control	0.2380	5.3457	3.0171
Samples soaked in oil at room temperature for 24hours	0.2290	3.8014	2.834
Samples soaked in hot oil at 60°C for 4hours	0.0324	0.5770	1.0001

Values are means for 10 test samples per each treatment

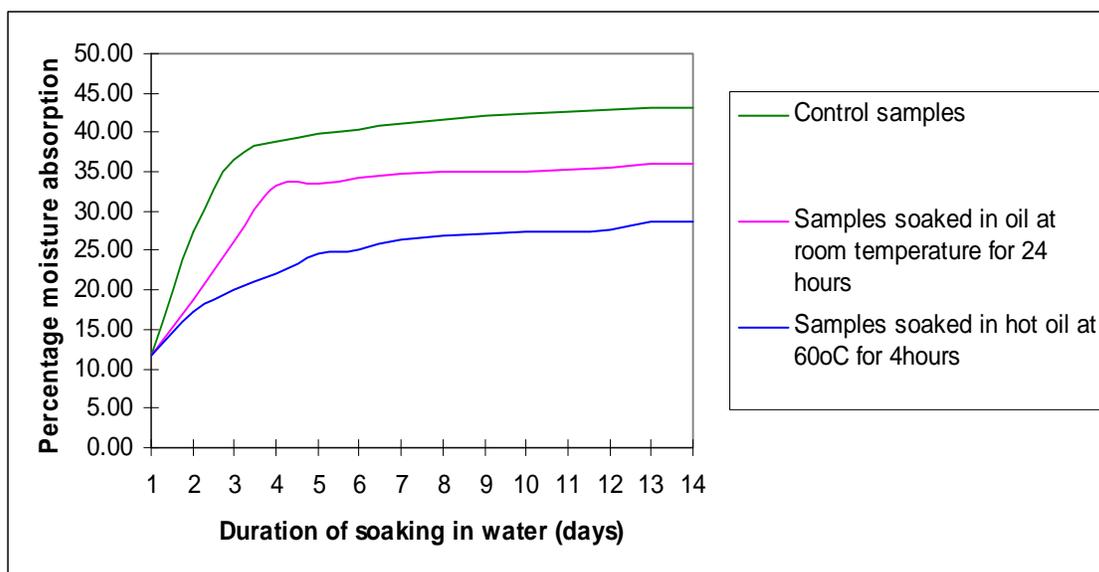
**Fig. 1.** Trends in moisture absorption for the control and oil-treated bamboo samples

Table 4. Fisher's Least Significant Difference of Pair of Means for Directional Shrinkage Values for the Oil-Treated and Untreated Bamboo Samples

Treatment	Mean shrinkage values (%)		
	Longitudinal	Radial	Tangential
Control	0.2380 ^a	5.3457 ^a	3.0171 ^a
Samples soaked in oil at room temperature for 24hours	0.2290 ^a	3.8014 ^b	2.834 ^a
Samples soaked in hot oil at 60°C for 4hours	0.0324 ^b	0.5770 ^c	1.0001 ^b

(p<0.05); Means with the same superscript in the same column are not significantly different

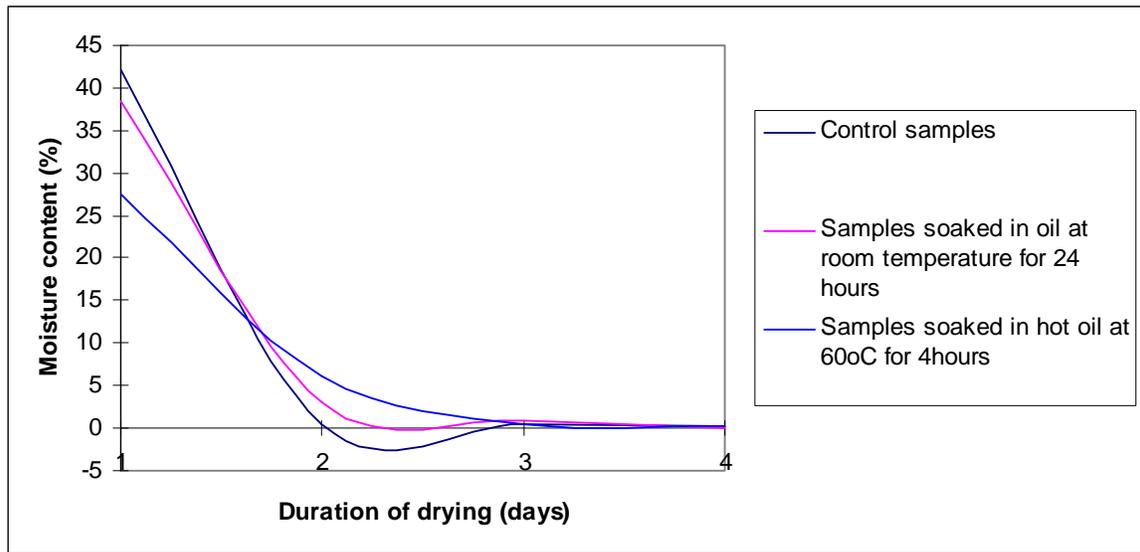


Fig. 2. Trends in overall moisture loss during oven-drying of the control and oil-treated bamboo samples during shrinkage test

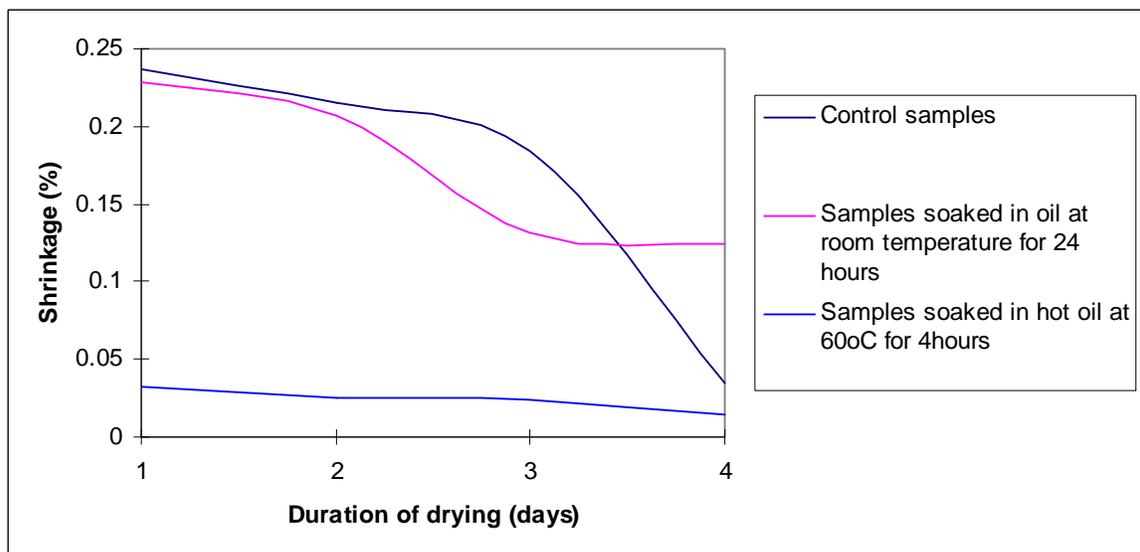


Fig. 3. Trends in longitudinal shrinkage for the control and oil-treated bamboo samples

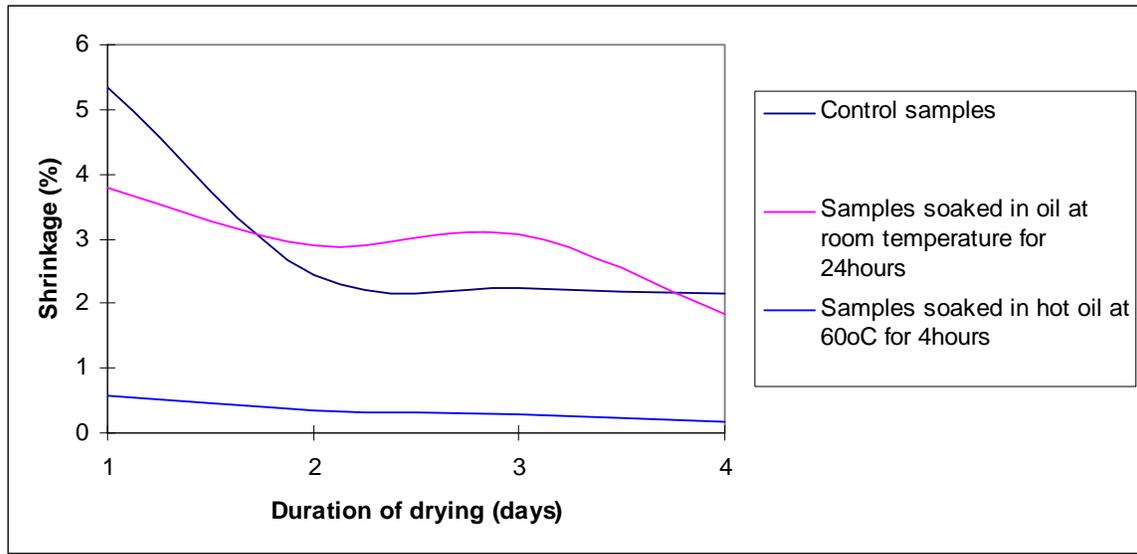


Fig. 4. Trends in radial shrinkage for the control and oil-treated bamboo samples

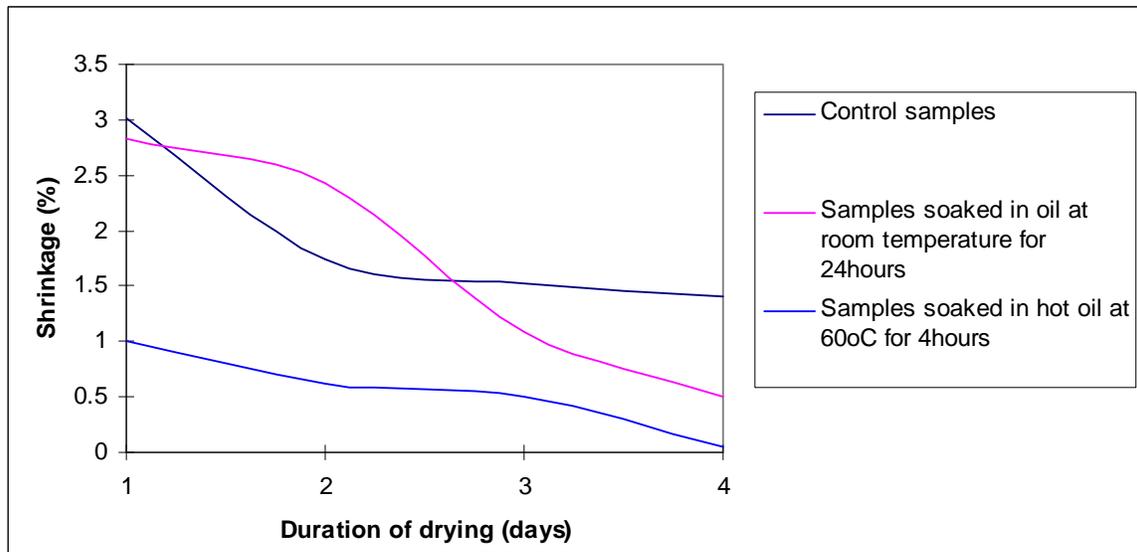


Fig. 5. Trends in tangential shrinkage for the control and oil-treated bamboo samples

DISCUSSION

The results tabulated in Table 1 showed that water resistance was greater in bamboo samples treated with neem seed oil at room temperature for 24 hours when compared with control samples as depicted by the mean values for percentage moisture gain. These particular results are also represented by the graphical trends in moisture absorption for the control and oil-treated bamboo samples in Fig.1. The graph revealed that both the oil-treated and control samples absorbed water at different rate and quantity

up to a particular stage when the trends remained constant, although more water was absorbed by the control samples as compared to the oil-treated ones.

This observation might be as a result of the capability of natural oils to prevent water uptake by lignocellulosic material, as also observed by Sailer et al. (2000). Samples soaked in hot oil at 60°C for 4 hours absorbed the least amount of water, while those for the control experiment had the highest absorption, followed by samples soaked in oil at room temperature for 24 hours. Low values of water uptake for the bamboo samples soaked in hot oil at 60°C for 4 hours may be attributed to the thermal degradation of hemicellulose, which is the most hygroscopic polymer in the lignocellulosic cell wall, as also observed in other studies, e.g. Tjeerdsma et al. (1998); Temiz et al. (2006).

These results in the first instance implied that using this vegetable oil or any other oil to treat bamboo as also documented in literature enhances its water resistance capability, although, it appeared that the increase in water resistance capability was more for bamboo samples thermally modified through hot oil treatment. Similar observations concerning increased dimensional stability of lignocellulosic material as treatment temperature increased have been made by other workers e.g. Leithoff and Peek (2001); Rapp and Sailer (2001); Militz (2002); Razak et al. (2005); Manalo and Acda (2009), although it is noteworthy that the effectiveness of hot oil treatment seems to be affected by the type of oil used (Wang and Cooper 2005).

It is noted that oil facilitates fast and uniform heat transfer and provides limited oxygen in the heating vessel (Rapp and Sailer 2001; Militz 2002). Wang and Cooper (2005) also noted that oils used in thermal modification undergo chemical reactions with cellulose or hemicelluloses, altering its physical properties. This kind of reduction in hygroscopic properties or improvement in water resistance is believed to be due to chemical changes of cell wall components such as decomposition of hemicelluloses or demethoxylation of lignin during treatment resulting in reduction or modification of adsorption sites in the cell wall (Burmester 1973; Giebel 1983; Tjeerdsma et al. 1998).

The ANOVA results showed that there was significant variation in the data for percentage moisture gain among the control and the treated samples at 5% significance level. The result of LSD (Table 2) showed that percentage moisture gain in the control experiment and those soaked in oil at room temperature for 24 hours had mean values that were not significantly different while both significantly differed from the mean value for samples soaked in hot oil at 60°C for 4 hours.

The result of the ASE tests (Table 1) also revealed that samples soaked in hot oil at 60°C for 4 hours had a higher value of 56.09%, compared with samples soaked in oil at room temperature for 24 hours with 29.03%. The implication of this result is that reduction in the quantity of moisture absorbed by bamboo samples treated with neem seed oil in this study was optimized as the oil temperature increased, and this might be as a result of modification of cell wall substances responsible for moisture absorption in this lignocellulosic material as stated earlier, although treating the samples with neem seed oil at room temperature for 24 hours also had an ASE. Similar observations for ASE owing to increased temperature for treating lignocellulosic material had been obtained by other workers, e.g. Temiz et al. (2006).

The mean values for the three directional shrinkage percentages for the control and oil-treated bamboo samples are tabulated on Tables 3. Bamboo, like any other

lignocellulosic material, is hygroscopic, thus the moisture content varies with the variations in the temperature and RH of the surrounding environment. Dimensional stability is very crucial in structural products made from lignocellulosic material, because the safety and comfort in a structure made from such lignocellulosic material, for instance, usually depends on it.

Shrinkage was more in the tangential plane, (however, the tabulated values may not show this because of the sample dimension as related to how the directional shrinkage percentages were calculated) followed by radial shrinkage and least, almost negligible, on the longitudinal plane, as also observed in other studies (Abd.Latif et al. 1993; Yu et al. 2008). These results were at variance with the result from the study by Lee et al. (1994), with all the studies agreeing on longitudinal shrinkage being the least.

It was observed that shrinkage was more in untreated (control) samples as compared to the oil-treated samples. The result showed that shrinkage was lowest in samples soaked in hot oil at 60°C for 4 hours. Thus, dimensional stability in terms of lower shrinkage value was more in bamboo samples treated with oil, with higher stability obtained in samples soaked in hot oil at 60°C for 4 hours. Similar observation exist in literature, e.g. Manalo and Acda (2009).

The trend of shrinkage in the oil-treated and control samples followed similar pattern as shown in Fig. 2, where the graph showed that both the oil-treated and untreated bamboo samples lost moisture during oven drying up to a point where all the samples had constant weight, irrespective of the fact that the oil-treated and untreated samples had different initial moisture content before oven drying. A peculiar shrinkage characteristic for bamboo is that it starts to shrink both in the wall thickness and diameter as soon as it starts to loose moisture (Tewari 1992). This behaviour is unlike wood, where most of the properties will start to change when it reaches the FSP.

Shrinkage, compared to control samples, was reduced by 3.78% for soaked bamboo samples in neem seed oil for 24 hours, while it was reduced by 86.39% for samples treated with hot oil at 60°C for 4 hours in the longitudinal direction. It was reduced by 28.89% for soaked bamboo samples in neem seed oil for 24 hours and reduced by 89.21% for samples treated with hot oil at 60°C for 4 hours in the radial direction, while the reduction was 6.07% for soaked bamboo samples in neem seed oil for 24 hours and 66.85% for samples treated with hot oil at 60°C for 4 hours in the tangential direction.

The graphical representations of directional shrinkage patterns (Figs. 3-5) showed that shrinkage was lower in bamboo test sample treated with hot oil at 60°C for 4 hours in the longitudinal, radial, and tangential directions. The same graphs also gave inconsistent patterns in the downward trends during shrinkage for control bamboo samples and those soaked in neem seed oil at room temperature for 24 hours in the longitudinal, radial, and tangential directions. The shrinkage pattern for control bamboo samples and those soaked in neem seed oil at room temperature for 24 hours appeared to be comparatively more consistent in the longitudinal direction (Fig. 3), as compared to radial and tangential directions (Figs. 4 and 5, respectively).

The one-way ANOVA result for the shrinkage values obtained for the control and treated bamboo samples revealed that there was statistically significant variation in the data for all the shrinkage directions. Thus, comparing the mean values, LSD of pair of

means (Table 4) revealed that longitudinally, shrinkage value for control samples and samples soaked in oil at room temperature for 24 hours were not significantly different, but both values were statistically significantly different from mean data for bamboo samples soaked in hot oil at 60°C for 4 hours at $p < 0.05$.

Results of LSD for tangential shrinkage also followed a similar trend as that for longitudinal shrinkage, while that for radial shrinkage had mean values that were significantly different for the control and treated samples (Table 4). It is noteworthy that physical and mechanical properties of bamboo depend on the species and its condition, site/soil and climatic condition, silvicultural treatment, harvesting technique, age, density, moisture content, position along and across the culm, nodes or internodes, and biodegradation, among others (Lee et al. 1994).

CONCLUSIONS

(1) Based on the outcome of this study, oil from neem seeds, which has little or no socio-economic value in this part of the world, can be used to improve water resistance and dimensional stability of bamboo culms and its products particularly at high oil-treatment temperature.

(2) Bamboo samples soaked in hot neem seed oil at 60°C for 4 hours had better water resistance and dimensional stability compared to samples soaked in oil at room temperature for 24 hours. This implies that thermal modification of bamboo using neem seed oil largely contributed to improved water resistance and dimensional stability in the bamboo samples as observed in other documented studies on different vegetable oils.

(3) Further studies are necessary to ascertain the performance of this oil in this regard at higher temperatures using these and other treatment methods.

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