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Influence of fiber distribution and characteristics on the fiber saturation point of *Calamus ornatus* var. *philippinensis* Becc. and *Calamus merrillii* Becc.

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Abstract: The water adsorption potential of two commercially important Philippine rattan species viz., (*Calamus ornatus* var *philippinensis* Becc and *Calamus merrillii* Becc) was evaluated. Fiber saturation point of the canes ranged from 21 – 25% which is lower than wood and more or less similar to bamboo. Fiber saturation point was highly influenced by the fiber distribution including the relative compactness or packing density of the cell wall in the fibrous sheath. The low degree of crystallinity indirectly showed the abundance of amorphous region within the cellulose chain. This indicates the presence of more moisture adsorption sites that could generate higher FSP values. However, this was not observed leading to the conclusion that the fiber wall compactness or relative packing density plays a more active role in regulating the amount of moisture adsorbed by the rattan cell wall.

Keywords: Water adsorption, *Calamus ornatus* var *philippinensis*, *Calamus merrillii*, equilibrium moisture content, fiber saturation point, fiber distribution, degree of crystallinity.

INTRODUCTION

Like all biological materials, the peculiar rattan cellular structure is the backbone of the physical performance and mechanical behaviour of the stem. Structure influences the cane's mechanical (Abd Latif and Siti Norralakmam, 1992), physical (Wan Tarmeze, 1994) and chemical (Abasolo *et al.*, 2005) properties. The unique distribution of the fibers in the fibrovascular bundles along its length (Bhat and Verghese, 1991) and across the stem diameter (Bhat *et al.*, 1990) provides extreme differential depth for a material that is only several centimeters in diameter (0.5-4 cm). Such a complexity favoured the canes' internal growth stress generation (Abasolo *et al.*, 1999), its thermomechanical properties (Abasolo *et al.*, 2002) and stem flexibility.

Rattans are hygroscopic and have high affinity to moisture in both in liquid and gaseous

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state. This is because of the abundance of moisture loving free hydroxyl groups at the cellulose surface and between the amorphous regions of the microfibrils (McLaren and Rowen, 1951). Hygroscopic materials will absorb and give off moisture indefinitely until equilibrium moisture content (EMC) is reached with the surrounding atmosphere. This is dependent on a number of factors including mechanical stress, the drying behaviour of the material, species and specific gravity of the sample, extractive content and temperature (Skaar, 1998).

Fiber saturation point (FSP) has been defined as the moisture state wherein the cell wall is fully saturated with bound water (Tiemann, 1906). It is the critical state wherein abrupt changes in the physical, mechanical and electrical conductivity of wood take place. For wood, it is approximately 28 - 30% moisture content.

There are a lot of studies in rattans over the years, but information regarding the rattan-water relationships is rather limited. In general, it is assumed that the FSP of the cane is similar to wood but report on bamboos (Hamdan *et al.,* 2007) showed that monocots could have lower FSP. This study was conducted in order to understand the adsorption capability of two important Philippines rattan species and to define the fiber saturation point of the cane. The study would provide deeper insights into the physical behaviour of cane with the change in vapour pressure in the surrounding atmosphere. Besides, the influence of fiber distribution and the degree of crystallinity on fiber saturation point was also evaluated.

METHODOLOGIES

Sample Materials

Commercially important *Calamus ornatus* var *philippinensis* Becc. (Limuran) and *Calamus merrillii* Becc. (Palasan) was used in the experiment. Mature samples were randomly selected and extracted from Pagbilao, Quezon and the Makiling Forest Reserve, Laguna, Philippines. Samples were divided into basal, middle and top portions which were further subdivided radially from periphery to inner core regions. Samples were fully submerged in water prior to measurement to maintain green state and to prevent fungal and insect attack.

Moisture adsorption and fiber saturation point

Sample cubes of 1 cm^3 was prepared from the peripheral and core regions of the basal, middle and top portions of the cane. Samples were oven-dried at 102 ± 3 ºC and were then placed in a desiccator in which the relative humidity was controlled at room temperature with aqueous solutions of salts according to the Handbook of Chemistry and Physics by Weast (1981 -1982). Samples were kept in the desiccator for at least 10 days or until the equilibrium moisture content between sample and desiccator was achieved.

RH started from 15% ($LiCl \cdot H_20$) until up to 100% (H_20). For every change in RH%, sample weight was taken in order to determine the moisture content at that particular RH%. Moisture content (MC%) was computed using the gravimetric method (Siau, 1984). Adsorption curve was prepared by plotting the RH% against the MC%. From these isotherms, MC of the sample at 100% RH was taken. This is the fiber saturation point of the sample or wherein the rattan cell wall was fully saturated with water. Triplicate measurements were taken with 5 samples each per replicate.

Fiber distribution

Using a sliding microtome, 25 – 35 μm thick transverse sections were prepared from the 0.5 cm x 0.5 cm x 3 cm blocks representing periphery and inner core regions of the basal, middle and top portions of cane. Sections were double stained with Safranin-Fast green and the sections were dehydrated in ethanol series up to 100%. The sections were then mounted in Entellan binders in a clean glass slide and slides were made permanent.

Microphotographs of the cross sections were taken using a light microscope with an attached Nikon digital camera. Using the Image J software, fiber percentage was measured following the procedure discussed in a previous article (Abasolo *et al.,* 2005). Triplicate observations were made with 5 pictures each per replicate. The average fiber distribution per section was determined.

Degree of crystallinity

Sixty – 80 mesh powdered samples were prepared from the different regions using a Wiley mill. *C. merrillii* only was selected because of the more pronounced differences in FSP values observed in this species.

The X-ray diffraction technique was used to determine the percent crystallinity of the individual samples. The powdered specimen was placed in the sample holder and was exposed to X-ray beam which subsequently diffracted the crystalline region of the microfibrils to the detector. Beginning at 40^o, the detector absorbed the beam up to an angle of 5^o at a scanning speed of 2^o min^{-1}. Beam intensity was recorded and plotted against the angle travelled by the detector. The fully automated computer system recorded both peak and background intensities that represent the crystalline and amorphous regions, respectively. Triplicate measurements were made for each sample.

Statistical analysis

Species, position along the stem (base, middle, top) and the radial position (periphery – core) as well as replications were considered in the statistical design. A 2 x 3 x 2 x 3 factorial Random Complete Block Design (RCBD), where replication as the block;

was utilized. Variation between species, different height levels and the interaction between species x position along the stem; species x different radii were subjected to statistical analysis. Test of significance was conducted at 95% confident level where N = 90. Regression analysis was performed in order to determine the relationship between fiber saturation point and position across the stem radius. Likewise, the interaction between fiber saturation point and degree of crystallinity was evaluated.

RESULTS AND DISCUSSION

Adsorption curve and fiber saturation point

Kept at a fixed temperature, a constant relative humidity is obtained when a solute is added to water, the vapour pressure (RH%) is reduced to an amount proportionate to the mole fraction of the diluted solutions. Dried rattan samples when introduced into the desiccator adsorb moisture until equilibrium moisture content (EMC) is attained. Using this behaviour, the adsorption curve of the two rattan samples were plotted (Figs 1 and 2).

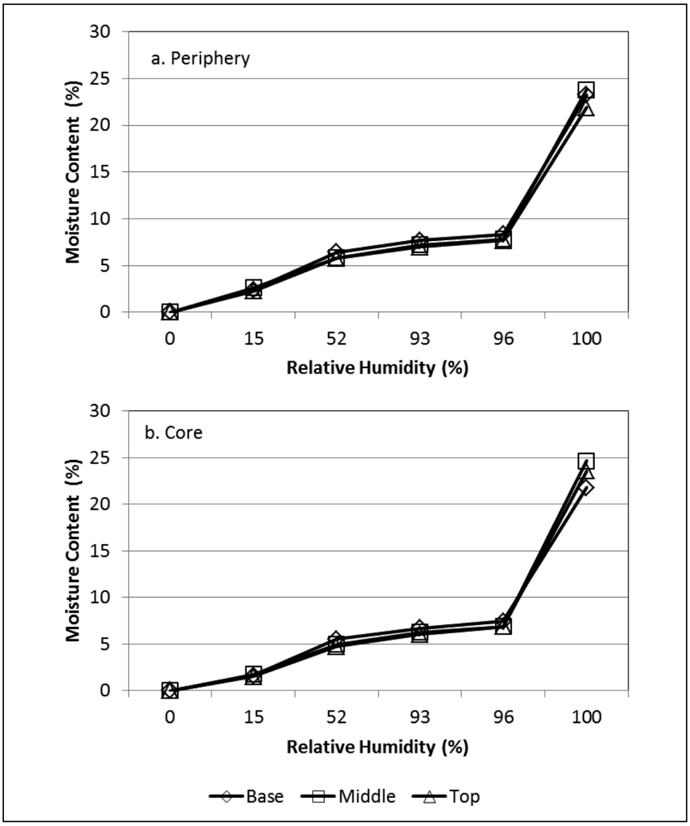

Figure 1. Sorption isotherm of *Calamus ornatus* var. *philippinensis*

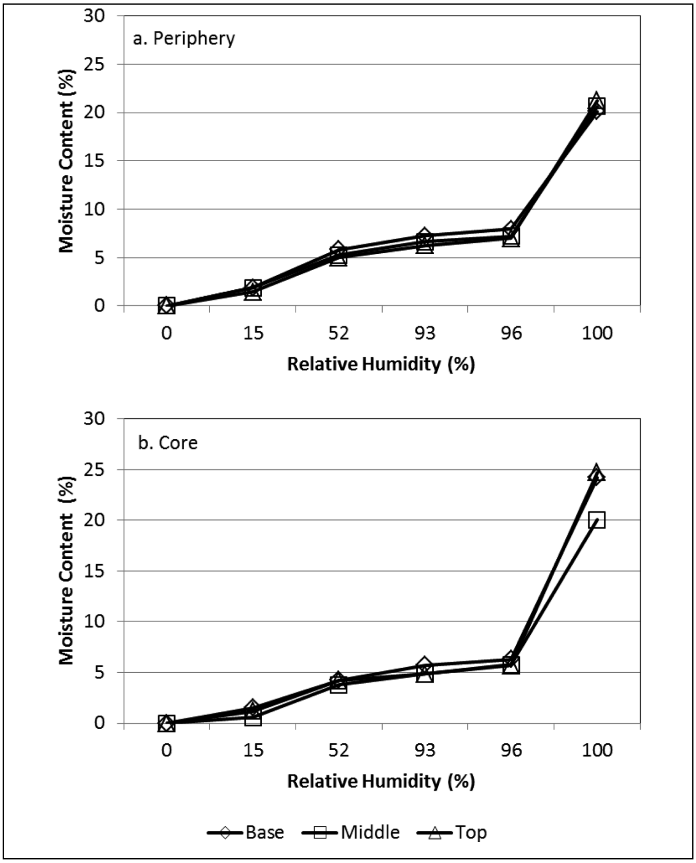

Figure 2. Sorption isotherm of *Calamus merrillii*

The relationship between percent RH and the MC of rattan samples was not linear. Regardless of species and position across the stem radius (periphery – core), moisture adsorption of the samples was slightly slow particularly from 0% upto 96% MC. This was different from the adsorption curve observed in bamboo (Hamdan *et al.,* 2007). This unexplained phenomenon was perhaps due to the formation of a weak cellulose-to-cellulose bond during the initial drying process (Haygreen and Bowyer, 1982). This made the free hydroxyl groups inaccessible to moisture during adsorption.

At 100% RH, a drastic increase in percent MC was noticed. MC% ranged from 20 – 25%, more or less similar to bamboo (Hamdan *et al.,* 2007) and slightly lower to wood (28-30%) (Skaar, 1988). This is the fiber saturation point of the material wherein the cell wall is fully saturated while the cell lumen is devoid of water. Unlike the other points below 100% RH where the values stayed very close to each other, MC at 100% RH was more variable especially at the core portion of *C. merrillii*. This was further observed when these values were extrapolated and plotted in a different graph (Fig. 3). Although, the variation in terms of FSP between samples were statistically not significant, it is worthwhile to investigate the origin of such differences.

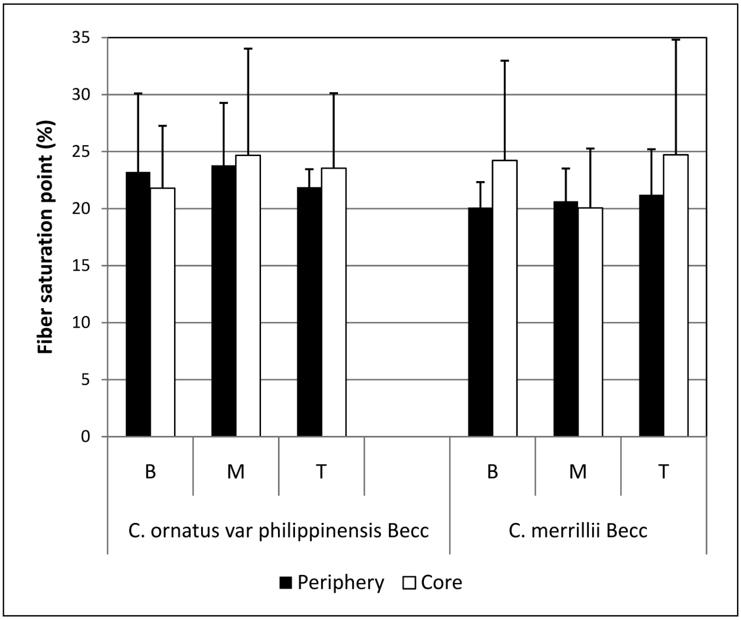

Figure 3. Fiber saturation point of *Calamus ornatus* var. *philippinensis* and *Calamus merrillii*

Fiber distribution

Rattan fibers occurs as fibrous sheath within the vascular bundle (Fig. 4) providing mechanical support to both water conduits (metaxylem) and food (metaphloem), typical of all *Calamus* spp. (Weiner and Liese, 1990).

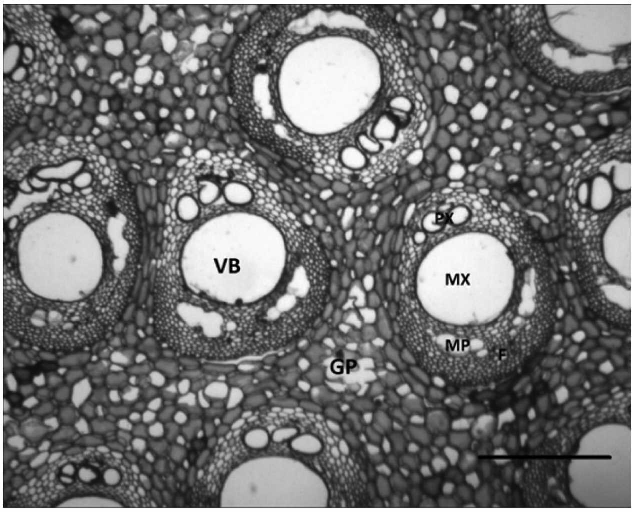

Figure 4. Structure of *Calamus ornatus* var. *philippinensis*
VB = Vascular bundle; MX = Metaxylem; GP = Ground parenchyma; Scale bar = 500 µm.

Table 1 provides the average fiber distribution along the length and across the stem radius. Fiber percentage ranged from 9.55% (basal-core) to 59.93 (basal-periphery). Regardless of species and position along the length, fibers were more congested towards the periphery than the inner core. The peculiar fiber grouping at the peripheral region clearly indicates that the vascular bundles at this region were primarily meant for mechanical support. In fact at the cortical regions just below the hypodermis, the vascular bundles occurs as fiber strands and were no longer connected with each other and radially flattened towards the epidermal layer (Tomlinson, 1990). Vascular bundles are more or less oval shape in the core regions and thin walled, scattered in the parenchymatous ground tissues and functioning for food and water conduction.

Analysis of variance (ANOVA) as shown in Table 2 clearly indicates the variability in fiber distribution. Although variation along the length of the cane (base-middle-top) was not significant, between species as well as across the stem radius (periphery-core) was statistically significant. Interactions between factors were also found

Table 1. Average values of fiber area percentage at different position across the radius and along the length of the cane of *Calamus ornatus* var. *philippinensis* and *Calamus merrillii*

	Periphery			Core		
	Base	Middle	Top	Base	Middle	Top
Rep 1	14.99 (1.58)	16.80 (1.83)	19.25 (5.12)	9.55(1.15)	12.50 (1.20)	11.10 (1.11)
	53.50 (13.62)	*34.51 (10.83)*	*37.79 (2.35)*	*16.09 (1.25)*	*15.78 (3.44)*	*15.57 (1.91)*
Rep 2	20.50 (2.75)	19.75 (3.23)	16.75 (4.02)	11.75 (2.13)	11.33 (1.80)	10.20 (0.78)
	32.68 (7.99)	*27.76 (5.01)*	*25.50 (2.41)*	*11.03 (2.52)*	*15.24 (1.67)*	*15.48 (1.61)*
Rep 3	59.93 (5.06)	39.66 (10.73)	35.44 (4.85)	15.65 (3.28)	21.78 (2.32)	23.77 (2.22)
	59.12 (4.94)	*32.92 (9.92)*	*31.93 (5.42)*	*19.40 (2.17)*	*21.55 (1.14)*	*22.10 (1.50)*

Calamus ornatus var. *philippinensis* = non-italized values; *Calamus merrillii = Italized values;* Standard Deviation = values in parentheses; Number of samples per replicate = 5

Table 2. Result summary of the 2 x 3 x 2 x 3 factorial experiment showing the variation in fiber distribution within the rattan stem between and within species.

SV	df	SS	MS	F Comp	F Tab (95%)
Block	2	1349.68	674.84	12.93	3.44 **
Treatment	11	3494.20	317.65	6.08	2.26 **
Species (A)	1	270.64	270.64	5.18	4.3 **
Base-Middle-Top (B)	2	266.26	133.13	2.55	3.44 ns
Periphery-Core(C)	1	270.64	270.64	5.18	4.3 **
A x B	2	94.69	47.35	0.91	3.44 ns
A x C	1	1980.42	1980.42	37.93	4.3 **
B x C	2	2443.93	1221.97	23.41	3.44 **
Error	22	1148.60	52.21		
Total	35	5992.47			

** = significant at 95 %; ns = not significant

significant. Thus, the properties of the cane material differ across the stem radius, it is very important to consider the radial position while evaluating the properties.

Degree of crystallinity

The crystalline region is the portion along the microfibrils where the cellulose chains are arranged in an orderly and compact manner because of direct hydrogen bonding between chains (Panshin and de Zeeuw, 1978). Conversely this value indirectly shows the amount of amorphous region or the portion within the microfibrils that is disorderly and loosely arranged. Such arrangement is due to the inability of adjacent chains to form hydrogen bonding with each other making the hydroxyl groups accessible to water and other chemicals during adsorption.

The degree of crystallinity of the cane ranged from 9 % to 21% (Fig. 5), comparatively lower to the crystallinity of either wood (Andersson *et al.,* 2003) or bamboo and was essentially similar to the crystallinity of bacterial cellulose (Roger and Perkins, 1968). Peripheral crystallinity was higher in the core.

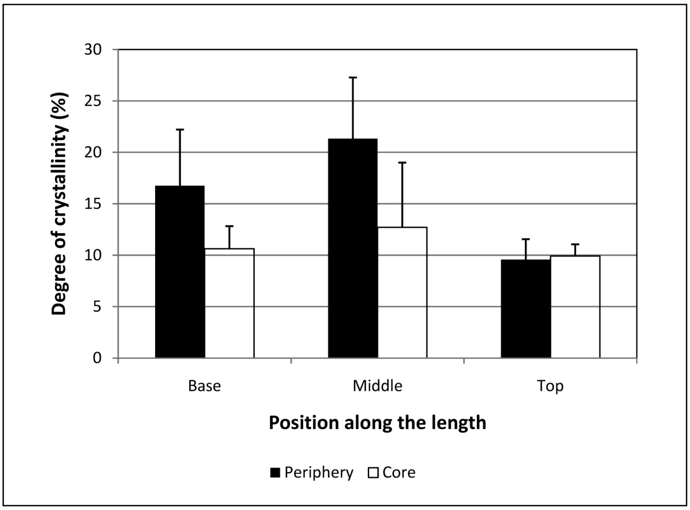

Figure 5. Degree of crystallinityof *Calamus merrillii*

Influence of fiber distribution on the fiber saturation point

FSP differences in bamboo within culm position were attributed to varying FSP of the fibers and parenchyma cells (Liese, 1985). Fiber distribution of the peripheral region did not significantly influence the FSP of the material (Fig. 6) while significantly influencing the FSP of the core.

The measurement of area percentage using ImageJ software is a method that considers the frequency or number of individual cell types as well as the relative thickness of its

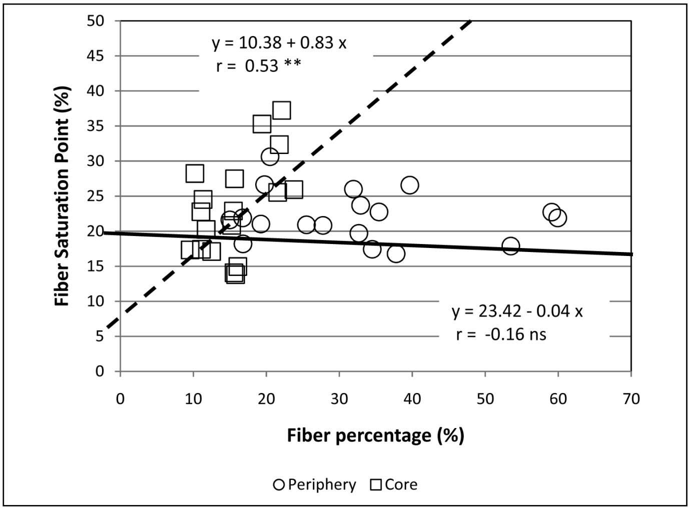

Figure 6. Influence of fiber distribution on the fiber saturation point.
Solid line = periphery; broken line = core.

cell wall. One distinct difference between the fibers of the peripheral and core region is with regard to their cell wall thickness. The fibers at the periphery were highly lignified, thick-walled and exhibit polylamellate layers (Parameswaran and Liese, 1985). The fibers at the core, on the other hand, are thin walled and less lignified. This would mean that although both are fibers, the two regions differ in the rigidity and compactness of its cell wall. The more the cell wall layer, the stronger the cell wall that could inhibit moisture from being adsorbed within its structures as in the case of the peripheral region. While the less compact cell wall of the fibers at the core is more accommodating to moisture. Packing density is more relevant than bulk density to a number of properties including water-adsorption capacity (Hillis, 1984). Actually, the same cell wall firmness could have limited the longitudinal shrinkage in rattans, and that shrinkage was attributed to the microfibril angle (MFA) of its wall (Abasolo *et al.*, 2000).

Influence of degree of crystallinity on fiber saturation point

Degree of crystallinity influences the elasticity, adsorptive capacity and other important physical attributes of the fiber (Assaf *et al.,* 1944). Regardless of radial position of the cane, fiber saturation point was significantly affected by the degree of crystallinity (Fig. 7). Degree of crystallinity of the site was inversely associated to fiber saturation point. Similar behaviour was observed in pure cellulose powder (Mihranyan *et al.,* 2004) and wood cellulose (Nakamura *et al.,* 1981). This was expected because the lateral bonding between individual cellulose chains at the crystalline region is so strong that it is inaccessible to moisture and other solvents. It is even more resistant to microbial and enzymatic degradation (Eriksson *et al.*, 1990). Water can only associate

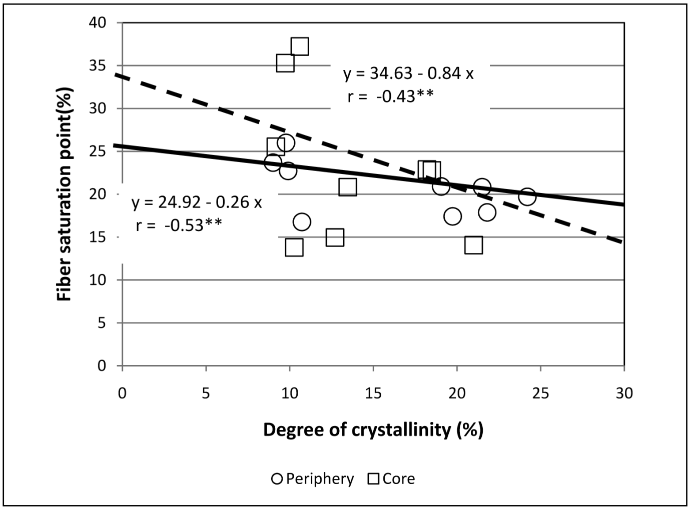

Figure 7. Influence of the degree of crystallinity on the fiber saturation point. Solid line = periphery; broken line = core.

with the cellulose at the outer surfaces, in between microfibirls (Mark, 1967) and also in the disordered portion of the chain.

Rattan cellulose crystallinity was very much lower than that observed in other wood materials. Instead of occupying approximately 2/3 of the cellulose chain, it was relegated to a maximum of only about 21%. This would indirectly mean that the amorphous region dominated the chain implying that a lot of adsorption sites would be available for moisture. However, this was not the case. The rattan cell wall accommodated a maximum moisture content of about 24%. Although, there were a lot of sites for water adsorption within the chain, the degree of compactness of the walls or the relative packing density played a more crucial role in its ability to adsorb moisture. Thus, the maximum water holding capacity of its cell wall was still lower to wood which has a higher degree of crystallinity (50-60 %).

CONCLUSION

The water adsorption behaviour of rattan canes was more or less similar to wood. Its fiber saturation point was about 20-24%. FSP was highly influenced by its fiber distribution as well as the compactness or the relative packing density of its cell wall. Although there were a lot of potential adsorption sites within the chain owing to its low degree of crystallinity that indirectly indicated the abundance of amorphous region, water adsorption of the cane was still lower to wood which has lesser amorphous region. This showed that the moisture adsorption phenomenon of the canes was more affected by the overall fiber wall characteristics particularly its compactness.

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