J. Bamboo and Rattan, Vol.8, Nos. 3&4, pp. 175-186 (2009)

Parameters affecting palasan cane (*Calamus merrillii* Becc.) flexibility and development of a cane flexibility equation

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Abstract: The effect of structure (ground parenchyma and vascular bundle distribution), physical traits (microfibril angle, degree of crystallinity and amorphous region), and cell wall chemistry (cellulose, hemicellulose and lignin) on the amount of compressive strain was evaluated to develop a mathematical model that could describe palasan cane flexibility. Image analysis, iodine staining method, x-ray diffraction technique and standard chemical analysis were performed. Finally, compressive strain was measured using the thermomechanical analyzer. Regression showed that among the different parameters, ground parenchyma percentage, microfibril angle, amount of amorphous region and hemicellulose content were positively correlated to compressive strain. Multiple regressions resulted to the mathematical equation: $y = 0.012x_2 - 0.001x_1 + 0.016x_3 + 0.017x_4 - 1.861$ where; y = compressive strain, x_2 is for the microfibril angle, x_1 is for the ground parenchyma, x_3 is for the amorphous region and x_4 is for the hemicellulose content. Among these parameters, hemicellulose content gave the most significant effect with $P = 0.050$.

Keywords: Palasan canes, compressive strain, mathematical model, image analysis, iodine staining, x-ray diffraction, chemical analysis, thermomechanical analyzer.

INTRODUCTION

Rattan cane is a unique biomaterial widely utilized in the furniture industry. Exceptional in the sense that it was able to combine two contrasting features particularly stiffness and flexibility. Normally, when a material is rigid it is inflexible. However with enough heat, rattan can be bent, molded, and curved into different shapes without compromising its mechanical attributes.

The mechanical traits of rattan canes have been extensively discussed in several reports (Abd Latif and Norralakmam, 1992; Bhat *et al.,* 1996). From this, strength was

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observed to be affected by species, age, stem position, fiber proportion, specific gravity and fiber wall thickness where the latter was the main contributor. All of these parameters are directly related to the distribution and occurrence of the vascular bundle (Siripatanadilok, 1996). It shows that cane stiffness is dependent on the vascular bundle due to the protected sheath of bast fibers (Chang *et al*.,1988) present within its structure.

In contrast, parameters affecting cane flexibility have been rarely discussed. It is normally associated with thermal treatment particularly how heat encourages molecular movement that allows cane reconfiguration (Abasolo *et al*., 2002a, 2002b). Hypothetically, if the bundle is for mechanical strength, the ground parenchyma cells that surrounds it is the most likely candidate that provides elasticity to the material. However, proof has yet to be solicited hence, such generalization is still invalid. This study was conducted to identify the different factors that influence cane elasticity. In addition, it attempted to combine all these parameters into a mathematical model that would best describe this flexible nature of palasan canes.

MATERIALS AND METHODS

Commercially important palasan canes (*Calamus merrillii* Becc.) growing naturally in the Makiling Forest Reserves, Los BaÒos, Laguna, Philippines, were collected. Two meter long samples were obtained from the base, middle and top portions of the cane. These portions were further reduced to peripheral, intermediate and core regions.

Sample preparations

Compressive creep strain

From the three regions 0.45 cm x 0.5 cm x 0.5 cm blocks were prepared. A thin cover glass was glued on the smoothened cross-sectional surface to evenly distribute the load.

Tissue distribution

Using a sliding microtome, 35 ñ 45 μm thick cross-sectional sections were dissected out from the blocks. Sections were stained with safranin, dehydrated with ethanol and treated with fast green. Stained sections were permanently mounted in a clean glass slide with Entellan binder.

Microfibril angle

From the different regions (peripheral, intermediate, core), 35 ñ 45 μm thick tangential sections were cut off using a sliding microtome. Samples were air-dried prior to measurements.

Degree of crystallinity, amorphous region and chemical analysis

Sixty ñ 80 mesh powder samples were prepared from the different regions using a Wiley mill. Samples were oven dried prior to measurements.

Measurements

Compressive creep strain

Compressive creep was measured using a Hitachi Thermomechanical Analyzer (TMA). Thermal expansion of the glass cover was considered negligible because of its thickness. Instrument calibration was performed. Block was placed at the center of the probe. After determining the initial height, a 5 g compressive load was applied while the sample tube was flooded with water. Water was used as the heating medium to prevent sample shrinkage and moisture content fluctuation during measurement that could negatively influence the results. Thermal equalization between water and sample was performed for 10 min, after which the load was raised to 150 g. Sample was heated up to 50^{o}C to remove the possible influence of microstructural prestresses (Spatz *et al.,* 1999) and irreversible thermal expansion (Sasaki and Okuyama, 1983) on the accuracy of the measurements. Temperature was carefully monitored so that the softening temperature of the cane (Abasolo *et al.,* 2002a) was not attained. The amount of compressive strain was determined for 30 min. Average of two measurements was used in the analysis.

Tissue distribution

Ground parenchyma, vascular bundle and fiber area percentage were measured using the permanently mounted cross-sectional samples. Digital images were taken using a light microscope with a Nikon digital camera attachment. Using the latest in digital imagery *e.g*., J Image software, tissue distribution along the length (base, middle, top) and across the radius (periphery, intermediate, core) of the canes was evaluated (Abasolo *et al.,* 2005).

Microfibril angle

Iodine staining technique was used to determine the microfibril angle of the samples (Meylan, 1967). The slices were soaked in Schultzís solution for 10 min with occasional shaking. After washing with distilled water and 100 per cent ethyl alcohol, they were airdried for 2-3 min. Samples were soaked in potassium iodide followed by nitric acid treatment. Pictures of the fiber wall surface were taken using an ordinary photo-microscope and the angle by which the crystal grain makes with respect to the longitudinal axis of the fibers was measured. A total of 35 pictures per site were used.

Degree of crystallinity and amorphous region

The X-ray diffraction technique was used to determine the percent crystallinity and amorphous region of the individual samples. The powdered specimen was bombarded by X-ray beam which was subsequently diffracted by the crystalline region of the microfibrils to the detector. Beginning at 40°, the detector absorbed the beam up to an angle of 5° at a scanning speed of 2°/min. Beam intensity was recorded and plotted against the angle traveled by the detector. The computer automatically computed for both peak and background intensities that represent the crystalline and amorphous region, respectively. Four measurements per sample were taken.

Chemical components

The major cell wall components of the individual sections were analyzed following the ASTM (1975) standards (D 1104-56, D 103-60, D 1106 -56). Average of two measurements was used in the evaluation.

Statistical analysis

Using the SPSS statistical software, regression analysis was performed to determine the effect of the individual parameters to the amount of compressive strain. Likewise, a multiple regression analysis was conducted to formulate a model that could predict the amount of compressive strain.

RESULTS AND DISCUSSION

Compressive creep strain

Flexibility is a reflection of how a material can permit large deformations during loading without causing a significant reduction in strength. It could be measured through bending, tensile and compression tests. Small samples are more prone to slippage when clumped to the TMA machine. Thus, the latter was selected because this was the only method that eliminates the possibility of sample slippage during testing.

A typical strain curve was observed characterized by an instantaneous creep for the first 5 min followed by a steady but gradual increase in strain up to the 30 min loading time (Fig. 1). The amount of creep strain varied between position along the length (base, middle, top) and across the radius of the cane (periphery, intermediate, core). Regardless of where the samples were obtained, the core exhibited the highest strain (0.30μ - 0.41μ) values while the peripheral region gave the lowest (0.11μ - 0.24μ). This showed that the core was more susceptible to compressive load whilst the peripheral region was more resistant.

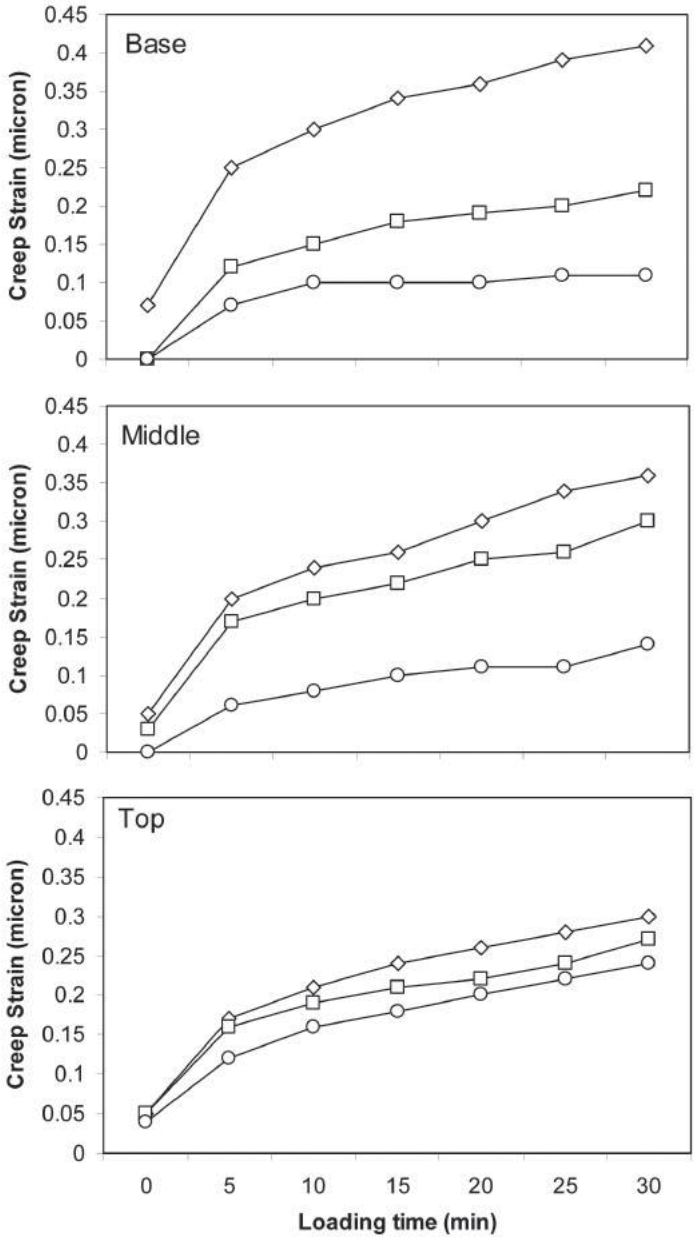

Figure 1. The amount of strain along the length (base, middle, top) and across the radius of the cane. ○ = periphery, □ = intermediate, ◊ = core.

Tissue distribution

Table 1 provides the percentage distribution of the ground parenchyma, vascular bundle and fiber elements within the rattan cane. Ground parenchyma percentage ranged

Table 1. Tissue distribution of the individual portions and analysis of variance

	Ground parenchyma (%)	Vascular bundle (%)	Fiber (%)
Base core	48.93 (4.54)	51.07 (5.65)	17.71 (2.70)
Base intermediate	56.14 (5.25)	43.83 (4.30)	17.27 (3.11)
Base periphery	33.78 (4.75)	66.22 (5.75)	38.36 (4.50)
Middle core	44.77 (6.51)	55.23 (6.70)	14.81 (2.33)
Middle intermediate	46.66 (5.13)	53.34 (7.23)	15.51 (2.11)
Middle periphery	31.07 (4.69)	68.93 (8.62)	14.58 (1.55)
Top core	43.24 (3.59)	56.76 (7.98)	15.57 (3.46)
Top Intermediate	40.22 (3.42)	59.78 (5.67)	16.72 (2.34)
Top periphery	40.66 (2.69)	59.34 (5.43)	20.41 (1.26)

Values in parentheses = standard deviation

from 31.07 to 56.14 vascular bundle percentage varied from 43.83 to 68.93, whilst, fibers ranged from 14.58 to 38.36 per cent. The trend along the length and across the radius of the cane was not discussed nonetheless several reports (Abasolo *et al.,* 1999; Siripatanadilok, 1996; Bhat *et al.,* 1990) have tackled this topic in detail.

Correlating the observed tissue percentage to the measured compressive creep strain showed that the ground parenchyma, vascular bundle and fiber elements gave moderate relationships with the amount of compressive strain (Fig. 2). Both vascular bundle and fiber percentage inhibited strain generation, whereas the ground parenchyma

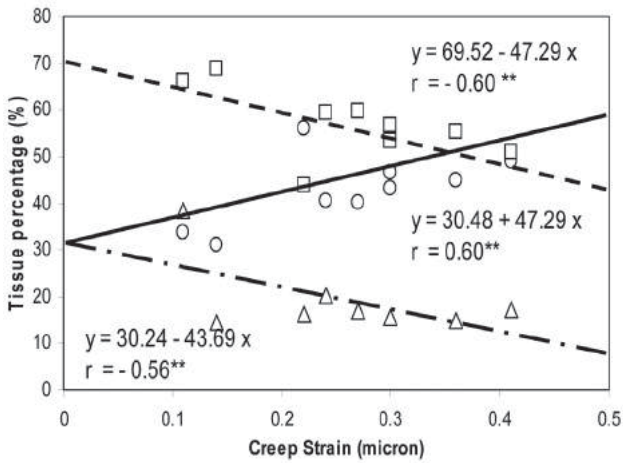

Figure 2. Effect of the different tissues on the amount of creep strain. ○ = ground parenchyma, □ = vascular bundle, Δ = fiber elements. ** = significant at α = 5%.

percentage promoted it. Structural analysis showed that the load bearing unit of rattan stem is the vascular bundle particularly the fibers due to its polylamellated wall (Paremeswaran and Liese, 1985). As such, several papers have indicated its direct impact on cane stiffness (Abasolo *et al.,* 2000; Bhat and Mathew, 1995), thus, it is but

normal that these elements would limit cane flexibility. Ground parenchyma cells are thin walled unlignified cells composed basically of primary wall which derived their mechanical strength from its hydraulic properties (Romberger *et al.,* 1993). This makes it very pliable when load is applied and hence would likely promote cane flexibility.

Microfibril angle, degree of crystallinity and amorphous region

Microfibril angle (MFA) on the fiber wall surface ranged from 24.65° to 33.60° (Table 2). Analyzing further the characteristics of individual fibrils, degree of crystallinity was ranged from 12.50 to 26.40 per cent which was quite lower to that of normal wood fibers (Lee, 1961). Likewise, the amorphous region comprises 73.60 to 87.50 per cent of the fibrils evidently dominating the whole chain.

Table 2. Microfibril angle, degree of crystallinity and amorphous region of the microfibrils

	Microfibril angle (°)	Crystallinity (%)	Amorphous (%)
Base core	29.50 (2.35)	12.50 (1.65)	87.50 (11.34)
Base intermediate	26.50 (3.24)	12.56 (1.46)	87.44 (8.79)
Base periphery	24.65 (4.71)	20.45 (5.66)	79.55 (12.54)
Middle core	28.80 (4.66)	19.50 (3.99)	80.50 (12.56)
Middle intermediate	26.65 (3.79)	18.88 (2.31)	81.12 (15.32)
Middle periphery	26.25 (5.46)	24.60 (1.55)	75.40 (14.65)
Top core	33.60 (4.62)	19.50 (2.78)	80.50 (15.79)
Top Intermediate	32.00 (5.66)	26.40 (4.77)	73.60 (15.78)
Top periphery	30.90 (5.33)	23.80 (4.31)	76.20 (12.65)

Values in parentheses are standard deviation

Figure 3 depicts the relationship between MFA and compressive creep strain. Although the regression showed moderate correlation, the relationship was significant and

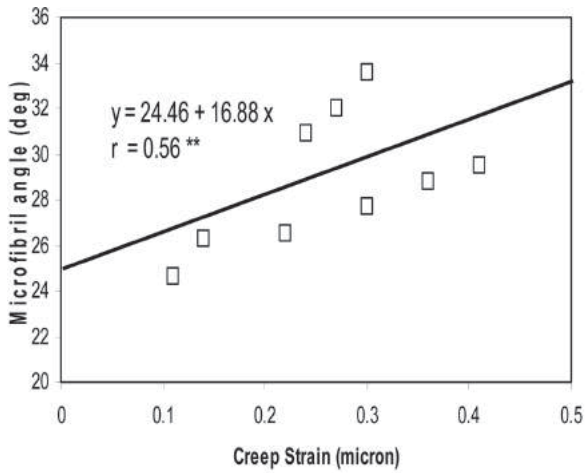

Figure 3. Effect of microfibril angle on the amount of creep strain. ** = significant at $\alpha = 5\%$.

positive. Opposite to flexibility, stiffness is reflection of the materialís ability to resist deformation. Microfibril alignment within the cell wall is directly related to material stiffness (Abasolo *et al.*, 2000; Cave and Walker, 1994). A decrease in microfibril angle towards the vertical axis of the cell would result in increase in stiffness and *vice versa* (Tsoumis, 1991). In the same manner, it could be deduced that large MFA would promote cane flexibility. In fact, the extent to which fibers can be stretched without breaking is dependent on its microfibril angle (Stamm, 1964) because of its ability to reorient itself to the direction of the load. The large MFA of rattan fibers would promote reorientation during loading and thus, would encourage cane flexibility.

The microfibril chain is divided into two regions (Panshin and de Zeeuw, 1978); one where the cellulose chains are arranged in a regular compact pattern (crystalline region) and the other where the chains are arranged in an irregular loosely manner (amorphous region). When the quantity of these regions was correlated to the amount of compressive strain, a moderate relationship was noticed (Fig. 4). The degree of

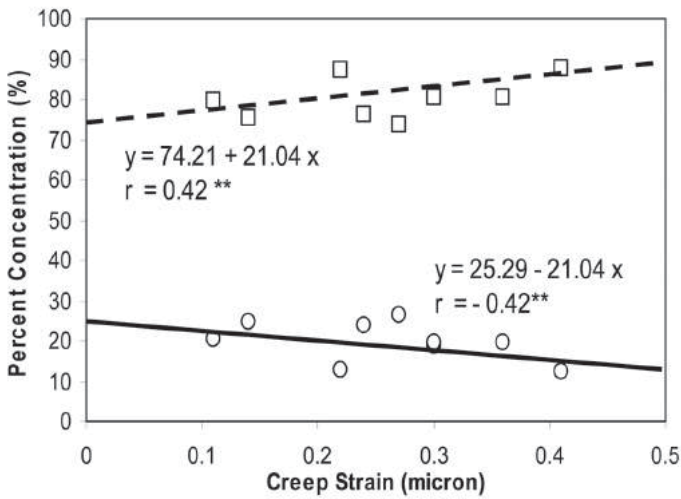

Figure 4. Effect of the concentration of crystalline and amorphous region on the amount of creep strain. ○ = crystalline, □ = amorphous. ** = significant at α = 5 per cent.

crystallinity negatively affected the amount of compressive strain, while the amorphous part promoted sample deformation. Formation of the crystalline region involves a high degree of lateral order between crystals (Kubler, 1987) that imparts stiffness to the cell wall, hence, would inhibit sample deformation. Contrary to this, the irregular and loose arrangement of the cellulose chain in the amorphous part would permit compression and extension of the fibrils, thus promoting sample deformation.

Chemical composition

Cellulose percentage by weight ranged from 34 to 53 (Table 3), hemicellulose

Table 3. Chemistry of the rattan cell wall

	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Base core	34.00 (3.55)	28.50 (1.56)	24.00 (2.34)
Base intermediate	52.50 (5.26)	25.00 (2.65)	22.00 (3.22)
Base periphery	53.00 (9.30)	23.50 (3.16)	24.00 (3.33)
Middle core	38.00 (7.98)	35.00 (5.40)	25.00 (4.31)
Middle intermediate	41.00 (5.13)	33.00 (7.23)	27.00 (2.11)
Middle periphery	46.00 (6.59)	30.00 (6.79)	23.50 (2.60)
Top core	37.00 (5.79)	32.00 (5.68)	25.00 (4.67)
Top Intermediate	39.00 (5.31)	32.50 (4.65)	24.00 (1.35
Top periphery	44.50 (4.98)	30.00 (5.31)	24.00 (2.98)

Values in parentheses are standard deviation

percentage ranged from 23.5 to 35 and lignin content varied from 22 to 27 per cent. Compared to wood, the chemical components of rattan cell were was more or less the same (Haygreen and Bowyer, 1982).

Figure 5 shows the relationship between the individual chemical components and the compressive strain. Cellulose content inhibited sample deformation, while the other

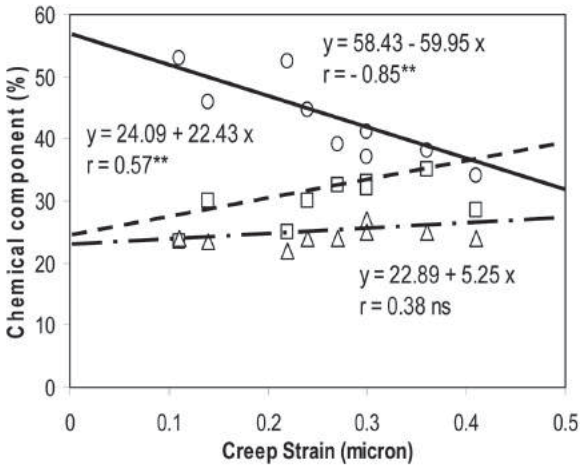

Figure 5. Effect of the individual chemical components on the amount of creep strain. ○ = cellulose, □ = hemicellulose, Δ = lignin. ** = significant at α = 5 per cent.

two encouraged compressive strain although the impact of lignin was only minimal. Related to the effect of microfibrils on the amount of strain, cellulose provides the main framework of the cell wall. Being such, it imparts rigidity to the wall and would therefore limit deformation during loading. Opposite to the former, hemicellulose and lignin are more pliable because of their instability especially when heated (Hillis and Rozsa, 1985). In fact, only if cellulose starts to soften beyond 230^{o}C (Fengel and

Wegener, 1984), the hemicellulose-lignin matrix softens at a low temperature of 80°C (Abasolo *et al.,* 2002b). This clearly showed how this matrix encourages molecular movement within the walls and hence, cane flexibility.

Model development

The study evaluated the parameters that influenced cane flexibility at three different levels *e.g*., microscopic level (structural design), cell wall level (fiber architecture *i.e.*, MFA, crystallinity and amorphous), and at the nanoscopic levels (chemical make up of the walls). From these three levels, ground parenchyma, MFA, amorphous region and hemicellulose component, respectively, gave significant relationships with compressive strain. This means that in all aspect of properties of cane flexibility was promoted. Starting from the way the different cellular elements are arranged within the structure up to the very minute details of how the cell wall was built, pliability of the material is being promoted. This is why palasan cane can be bent, molded, curved or changed in shape indefinitely without causing any major reduction in stiffness.

Consolidating all these parameters and subjecting them to a multiple regression analysis, the resulting model was generated:

$$y = 0.012\ x_2 - 0.001\ x_1 + 0.016\ x_3 + 0.017\ x_4 - 1.861$$

where: x_1 = Ground parenchyma percentage
x_2 = Microfibril angle (MFA)
x_3 = % Amorphous region
x_4 = Hemicellulose content

To verify the applicability of this model in predicting cane flexibility, the predicted value was correlated with the actual observed values of compressive strain. It showed that the two were highly correlated with r = 0.93** at $P = 0.0002$ (Fig. 6). This proved

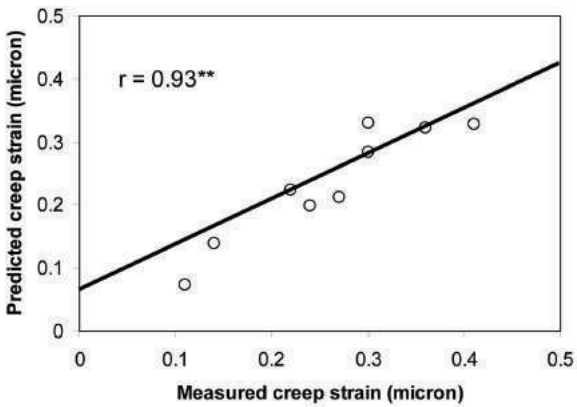

Figure 6. Correlation between the predicted creep strain and the actual creep strain. ** = significant at $\alpha = 5$ per cent.

that the generated model was able to accurately estimate the actual strain values. Furthermore, analysis revealed that among the four parameters, hemicellulose gave the most significant effect on the model (Table 4) with a $P = 0.05$. This was probably due to the fact the hemicellulose via the 5-Carbon xyloglucan side chain (Passioura, 1994) allows cell wall expansion. Thus, the collective effect of wall expansion of the different cells would result in the overall flexibility of the rattan canes.

Table 4. Multiple regression results

Parameters	Coefficients	Standard Error	*t*- Stat	*P*-Value
Ground parenchyma (%)	-0.001	0.004	-0.277	0.795
MFA (°)	0.012	0.008	1.572	0.191
Amorphous (%)	0.016	0.007	2.194	0.093
Hemicellulose (%)	0.017	0.006	2.770	0.050

CONCLUSION

Palasan cane is a unique biomaterial that can withstand extreme deformation during processing without significantly compromising its mechanical properties. This excellent attribute could be traced back to its structural design, fiber architecture and chemical make up of its cell wall. The combination of these individual parameters makes palasan cane a superb raw material for the furniture industry because it mixes strength with flexibility. The present study was able to identify the different parameters that affected cane flexibility and a mathematical model was developed. It accurately predicted actual deformation behaviour of the material. The study was also able to show that among the different parameters, hemicellulose content gave the most significant contribution to cane flexibility.

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