JOURNAL OF MATERIALS SCIENCE LETTERS **20**, 2001, 2095–2096

The determination of porosity and cellulose content of plant fibers by density methods

L. Y. MWAIKAMBO
Department of Engineering and Applied Science, University of Bath, Bath BA2 7AY, UK
E-mail: msplym@materials.bath.ac.uk

M. P. ANSELL
Department of Engineering and Applied Science, University of Bath, Bath BA2 7AY, UK

Plant fibers often arranged in fiber bundles, for example hemp and jute are comprised of four main chemical components namely cellulose, hemicelluloses, lignin and pectin. These are chemical components, which make the solid morphological structure of plant fibers together with the air filled lumen. In some cases the lumen contains protoplasm for example in cotton single fibers [1].

The absolute diameter of plant fibers excludes the lumen but includes the pores trapped between the fibrils and lamellae. The absolute density excludes all the pores and lumen and is therefore a measure of the solid matter of the fibers. The bulk (apparent) density on the other hand includes all the solid material and the pores of the fibers. The bulk density is always less than that without pores hence less than the absolute density due to the buoyancy effect caused by the trapped air. The absolute density of pure cellulose is reported as 1592 kg/m^3 and that of regenerated cellulose as 1583 kg/m^3 [2]. The absolute density of most plant fibers is between 1400–1500 kg/m^3. This slight drop of density in plant fibers is due to the presence of porosity including the lumen and impurities such as waxes and oils.

The quantification of the plant fibers' constituents has traditionally been carried out by chemical methods using established standard methods such as the ASTM, BS and ISO standards. Sun *et al*. [3] used 6%NaOH and 17.5%NaOH to extract hemicellulose and produced a scheme for the fractionation of polysaccharides and lignin from oil palm trunk and empty fruit bunch (EFB). Mitchel and Winandy [4] used ethanol, acidic chlorite and then an alkaline to extract hemicellulose from holocellulose. Sugars, residual acid and Klason lignin were determined by Pettersen and Schmidt [5] to determine α-cellulose.

The literature available gives different cellulose contents for the same fiber and sometimes the difference between one result and the other can be of the order of 12% difference [6, 7]. In this work combining the Archimedes method with the technique of helium pycnometry makes unique accurate measurements of porosity and cellulose content.

The densities of hemp, sisal, jute and kapok fibers have been measured as part of a programme of work on plant fiber composites [8]. Kapok fibers are single seed fibers and the three other fibers are fiber bundles. Average diameter for 50 fibers evaluated by scanning electron microscope (SEM) and image analysis (IA) are presented in Table I.

Benzene with a density of 875 kg/m^3 was used as a solvent for the measurement of the bulk density of fibers and an electronic balance was used to weigh fibers. Weights were measured to the nearest 0.001 g. A tuft of fibers was first weighed on the pan and its weight recorded as W_{fa}. The fiber was then immersed in the benzene and made to rest on the weighing pan submerged in the solvent. The weight in kg of the immersed fiber was then recorded as W_{fs}. The bulk density (ρ_b) of the fibers was calculated using Equation 1.

$$\rho_b = \frac{\rho_s W_{fa}}{W_{fa} - W_{fs}} \tag{1}$$

Where ρ_s = the density of the solvent (benzene) (kg/m^3). All measurements were determined at 19 °C. Helium pycnometer model AccuPyc 1330 acquired from Micrometrics Instrument Corporation (USA) was used to measure the absolute density of fibers.

The sample was placed into the pycnometry sample holder and weighed. The sample and container were then placed into the cell chamber. The sample identification number was given and its weight recorded. The helium gas was then admitted into the cell chamber filling the open lumen and pores. This procedure was

TABLE I Some physical properties of hemp, sisal, jute and kapok fibers

Fiber type	Diameter (μm)	Absolute density ρ_a (kg/m^3)	Bulk density ρ_b (kg/m^3)	Porosity P (%)	Non-cellulose X (%)	Non-cellulose + air Y (%)	$P + X$ Y' (%)	$[(P + X) - Y]$ T_{air} (%)	Cellulose content Z (%)
Hemp	67.84	1505	1468	2.46	5.46	7.79	7.92	0.13	91.95
Sisal	167.85	1428	1273	10.85	10.30	20.04	21.15	1.11	77.74
Jute	45.44	1532	1358	11.36	3.77	14.70	15.13	0.43	84.44
Kapok	22.65	1474	384	73.95	7.41	75.88	81.36	5.48	13.16

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repeated according to the number of the pre-determined runs and ten were used in this work. At the end of the last run a summary report was printed out giving the volume of the sample and its weight for each run, the average sample volume and absolute density (ρ_a) and their standard deviations.

The porosity of fibers was then determined using the absolute and bulk density of the fibers using Equation 2.

$$P = \left(1 - \frac{\rho_b}{\rho_a}\right)100 \tag{2}$$

Where P = percent porosity (%). The percentage of non-cellulose binding materials (X) is calculated from the absolute density, ρ_a, and a data value for the density of cellulose, ρ_{cell}, according to Equation 3.

$$X = \left[1 - \frac{\rho_a}{\rho_{cell}}\right]100 \tag{3}$$

The percentage of non-cellulose binding material plus pores (trapped air) (Y) is calculated from the bulk density, ρ_b, and the density of cellulose, ρ_{cell}, according to Equation 4

$$Y = \left[1 - \frac{\rho_b}{\rho_{cell}}\right]100 \tag{4}$$

The porosity (P) and percentage of non-cellulose materials (X) are added to give the quantity Y' (Equation 5).

$$Y' = P + X \tag{5}$$

Values for ρ_a, ρ_b, P, X, Y and Y' are presented in Table II.

One would expect that Y values would be equal or close to Y' values. However, this is not the case as the values in column Y are less than the values in Y' and an equation is developed to determine the Y' values using densities (Equation 6) as follows.

$$Y' = \left(2 - \frac{\rho_b}{\rho_a} - \frac{\rho_a}{\rho_{cell}}\right)100 \tag{6}$$

The decrease in the values in column Y indicates that tiny air pores exist in addition to the porosity obtained by Equation 2, the P values. These micropores are represented by the T_{air} values in Table I. Equation 7 is used to calculate this microporosity.

$$\begin{aligned} T_{air} &= [(P + X) - Y] \\ T_{air} &= \left[\left(1 - \frac{\rho_b}{\rho_a}\right) + \frac{\rho_b - \rho_a}{\rho_{cell}}\right]100 \end{aligned} \tag{7}$$

The microporosity is very small for the fibre bundles but is significant for kapok fiber (Table I). This is considered to be caused by the loose packing structure of the constituents of kapok fiber.

An equation is developed using the bulk and absolute density of the fiber and the density of pure cellulose to determine the amount of cellulose a particular fiber contains and is developed as follows.

Let Z be the amount of cellulose in a fiber and using the expression in the last column of Table I, the expression for Z (Equations 8 and 9) is given as follows.

$$\begin{aligned} Z &= [1 - (P + X + T_{air})]100 \\ Z &= [1 - (P + X + P + X - Y)]100 \\ Z &= [1 - (2P + 2X - Y)]100 \end{aligned} \tag{8}$$

$$\begin{aligned} Z &= \left[1 - \left(3 - 2\frac{\rho_b}{\rho_a} - 2\frac{\rho_a}{\rho_{cell}} - \frac{\rho_b}{\rho_{cell}}\right)\right]100 \\ Z &= \left[2\left(\frac{\rho_b}{\rho_a} + \frac{\rho_a}{\rho_{cell}}\right) - \frac{\rho_b}{\rho_{cell}} - 2\right]100 \end{aligned} \tag{9}$$

The cellulose content of plant fibers can be calculated using the absolute, bulk and cellulose densities (Equation 9) and the values are given in the last column in Table I).

Factors such as cellulose microfibril angle have a strong influence on the mechanical properties of plant fibres, however, it can be seen that hemp fiber has strong potential as natural fiber reinforcement for polymeric materials. On the other hand kapok fibers are not only hollow but also have very low cellulose content in the cell wall.

It can be concluded that the combination of the classical bulk density measurement and the pycnometry method for measuring absolute density and porosity enable very accurate determination of true plant fiber porosity and cellulose content to be evaluated.

Acknowledgments

The first author wishes to thank the Sokoine University of Agriculture for financial support in the form of a scholarship under the Tanzanian-Norwegian technical co-orporation program. The authors wish to thank Dr. Garath Neighbour for providing the helium pycnometer.

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Received 10 August
and accepted 1 October 2001