CHAPTER 2

MATERIALS AND METHODS

2.1 Raw material and sample preparation

The fronds were obtained from oil palm trees at the Bringham Estate in Damansara Utama, near Kuala Lumpur. The trees were cultivated in 1969 and reached a height of between 5.5 to 7.7 metre in 1987/88. The frond samples were taken from the lowest part of the trees. An average of 3 fronds were taken from each tree at random. The fronds were collected from time to time when the need arised.

The frond consists of leaflets, a central rachis to which the leaflets are attached and a spiny petiole (the part of the leaf stalk between the lowest leaflets and the trunk). Photographs of the tree and leaf can be seen in Plates 1 and 2.

As soon as the fronds were received at the laboratory, the leaflets were manually separated from the rachis and petiole. The leaflets were further separated into leaf blades and midrib, each cut into 5 cm pieces. The leaf blades and midrib were dried separately in the sun to prevent fungi attack, then placed into different plastic bags from which samples were taken from time to time. These samples were the raw material used throughout this work.

PLATE 1: An oil palm (Elaeis guineensis Jacquin) tree

PLATE 2: A close-up view of an oil palm leaf

Experiments were carried out separately on the midrib and leaf blade.

3.2 Determination of apparent density

The determination of apparent density followed the method of TAPPI T 258 in TAPPI (1978). An example of the calculation is shown in the Appendix (A1, p.144).

3.3 Moisture content

About 3 g of the raw material was placed into a previously weighed container and dried in an oven to a constant weight at 105°C. The moisture content was expressed as a percentage of the air dry weight of the raw material. The factor to convert the air dry weight (AD) to the oven dry weight (OD) equivalent weight was then calculated.

The moisture content (MC) of the raw material was not constant as the material was collected from time to time; thus the MC of each lot was separately determined.

3.4 Morphological study

The preparation of ultimate fibres was adapted from the method of Spearin and Isenberg (1947). The midrib was manually chopped into pieces the size of matchsticks, while

The leaf blades were manually shredded into pieces. This was well mixed and a representative taken for digestion. For comparison purposes, the morphology of the petiole was similarly determined.

An equal volume of glacial acetic acid (100 ml) and hydrogen peroxide (100 ml) was used to completely immerse the sample in a beaker. It was then covered and placed in a boiling water bath in a fume cupboard. Additions of glacial acetic acid and hydrogen peroxide were repeated hourly till the sample was bleached (white).

The bleached samples were then disintegrated by gentle shaking in a flask containing glass beads. The resulting fibres were filtered and washed with water. They were then stained with safranin-o and mounted onto a microscopic slide with a drop of glycerine. The dimensions of the projected images of the length of 200 fibres, the width and lumen of 100 fibres were measured by means of a Visopan projector. For the length, a magnification of 50x was used whereas a magnification of 500x was used for the measurement of lumen and width of fibre.

Calculation related to fibre morphology is found in the Appendix (A1, p.144).

2.5 Proximate chemical analyses

Prior to the chemical analyses the samples were ground. Proximate chemical analyses were carried out on material which passed through a BS 40 mesh sieve and retained on a BS 60 mesh sieve. Prior to the analyses, the material was oven dried to determine the moisture content. All analyses were carried out in duplicates.

2.5.1 Determination of moisture content

About 2 g of the sample was placed into a previously weighed bottle. This was dried to constant weight at 105°C. The moisture content was expressed as a percentage of the AD weight of the woodmeal. The factor to convert the AD weight to the OD equivalent weight was calculated. An example of the calculation for the moisture content is found in the Appendix (A3[a], p. 145).

2.5.2 Ash content

Ash content of wood is defined as the residue remaining after ignition at 575 ± 25°C for 3 hr or longer if necessary to burn off all the carbon.

The method is taken from "Ash in wood" described in TAPPI T 15 (TAPPI, 1978).

An example of the calculation for ash can be found in the Appendix (A3[b], p.145).

2.5.3 Solubility in 1% alkali solution

This method determines the resistance of a wood or lignocellulosic sample to a solution of hot dilute alkali. One application is in the determination of the degree of fungal attack that has taken place in wood. As the wood decays, the percentage of alkali soluble materials increases and the pulp yield as a result of the decay decreases.

This method was based on "1% caustic soda solubility of wood" as in TAPPI T 212 (TAPPI, 1978). Sodium hydroxide (1%) and acetic acid (10%) solutions were used.

An example of the calculation for 1% alkali solubles is found in the Appendix (A3[c], p.146).

2.5.4 Alcohol-benzene solubility

This is a measure of the waxes, fats, some resins and possibly some portions of wood gums.

The method is adapted from "Alcohol-benzene solubility of wood" as in TAPPI T 212 (TAPPI, 1978). The solvent used contained 1 volume of ethyl alcohol and 2 volumes of benzene.

An example of the calculation for alcohol-benzene solubles is found in the Appendix (A3[d], p.146). The subsequent analyses were carried out using the air dry extractive free sample.

2.5.5 Hot water solubility

This procedure measures extraneous components such as tannins, gums, sugars, colouring matter and starches in wood and pulp.

The method used was based on "Water solubility of wood" as in TAPPI T 207 (TAPPI, 1978). An example of the calculation for hot water solubles can be found in the Appendix (A3[f], p.147).

2.5.6 Lignin

Lignin is the insoluble residue that is left when the carbohydrates of wood when treated with strong acids are hydrolysed.

The method was taken from "Acid-insoluble lignin in wood and pulp" as in TAPPI T 222 (TAPPI, 1978) using

sulphuric acid (72%). An example of the calculation for the acid-insoluble lignin is found in the Appendix (p. 5).

2.5.7 Pentosans

Pentosans are part of the non-cellulosic carbohydrates present in wood and plant materials and consist mainly of xylan and arabinan. Pentosans are hydrolysed by boiling dilute acid to pentoses and they then decompose into volatile furfural which is almost entirely distilled over under the condition of the experiment.

The determination of pentosan was based on the method by Savard *et al.* (1954). The chemicals used included hydrochloric acid (13.2%), potassium bromate (0.1N) and potassium iodide (10%). Sodium thiosulphate (0.1N) was used as the titrating solution.

An example of the calculation for pentosans is found in the Appendix (A3[h], p. 148).

2.5.8 Holocellulose

Holocellulose is the lignin free fibrous material containing all of the hemicelluloses and celluloses in wood.

The method used was taken from that of Wise *et al.* (1946) with slight modifications for routine analysis. Sodium chlorite and acetic acid (10%) were employed here. An example of the calculation for holocellulose can be found in the Appendix (A3[i], p.150).

2.5.9 Alpha-cellulose

Alpha-cellulose can be determined as the portion of the plant cellulose which is insoluble in 17.5% sodium hydroxide at 20°C under specified conditions described in TAPPI T 203.

The method used was an adaptation of that in "Alpha-, Beta- and Gamma-cellulose in Pulp", TAPPI T 203 (TAPPI, 1978). Chemicals used were sodium hydroxide (17.5%), sodium hydroxide (8.3%) and acetic acid (2N). An example of the calculation for alpha-cellulose is found in the Appendix (A3[j], p.151).

2.6 Pulping trials

The raw materials were pulped in three cooking processes, (i) the chemical soda, (ii) the sulphate cooking and (iii) the neutral sulphite semichemical (NSSC) process. Each cooking was carried out in a six litre MK steel

digester which operates by indirect heating via circulating cooking liquor.

2.6.1 Chemical soda process

The amount of raw material, the liquor:wood ratio, temperature and the length of time of pulping of the midrib were kept constant in each cooking. The concentration of sodium hydroxide, however, was varied from 14% to 18%; consequently the amount of water added was changed accordingly.

The fixed conditions were:

liquor to wood ratio = 5:1

time to max temp (160^{o}C) = 1.5 hr

time at max temp (160^{o}C) = 2 hr

Pulping of the leaf blades was also carried out by the conditions outlined above but at a higher ratio of liquor to wood of 7:1 had to be employed due to the bulky nature of the material. The pulping was carried out initially at 18% A.A., but since a very low yield (10.6%) was obtained, pulping of the leaf blades was discontinued.

Pulping of the leaves (whole) was also conducted at 20% A.A. and like the leaf blades, further pulping was not conducted due to the very low yield (9.3%) obtained.

2.6.2 Chemical sulphate process

Pulping by the chemical sulphate process was conducted under the following fixed conditions:

sulphidity = 25%

liquor to wood ratio = 5:1

time to max temperature (170°C) = 1.5 hr

time at max temperature (170°C) = 2 hr

The concentration of active alkali was varied from 14% to 18% Pulping of the leaf blades was carried out using 18% A.A. and with a slightly higher liquor volume. Further pulping was not considered since the yield was very low.

After each cook in the soda and sulphate processes, the pulp was removed, washed thoroughly and disintegrated for 5 minutes in a Thwing-Albert Lear Blend-A-Mill. Screening of the pulp was carried out on a Sommerville fractionator with the plate of 0.15 mm wide slits used. Water was removed as much as possible from the pulp by means of a water extractor (Neng Shin-Taiwan). The pulp was then blended in a Hobart blender, after which it was placed in an air-tight plastic bag and stored in the refrigerator to condition the pulp for at least a day before the determination of yield, kappa number and preparation of handsheets for evaluation of the properties of the pulp.

2.6.3 NSSC pulping process

Pulping by the NSSC process was conducted under the following fixed conditions:

sodium carbonate = 6%

liquor to wood ratio = 7:1

time to max temp (160°C) = 1.5 hr

time at max temp (160°C) = 2 hr

Rubbing plates: (i) = 0.625 mm clearance

(ii) = 0.25 mm clearance

(iii) = 0.125 mm clearance

NSSC pulping was carried out only on the midrib.

The main chemical used was sodium sulphite buffered with sodium carbonate. Upon completion of each digestion, the black liquor was collected for the determination of the final pH. The partially digested samples were washed and passed through a Bauer refiner with the rubbing plate attached. After washing, the pulps were screened by means of a Sommerville fractionator with a plate of 0.15 mm wide slits to remove the shives. The screened pulp was then treated similar to those obtained from soda and sulphate cooks.

Calculations related to NSSC pulping are found in the Appendix (A5, p. 158).

2.7 Analysis of pulping liquor

At the end of each pulping trial, a sample of the black liquor was collected for analysis.

2.7.1 Analysis of pH

The pH value of the black liquor was measured by a pH meter.

2.7.2 Chemical consumed in a sulphate digestion

A sample of the black liquor from each pulping trial by the sulphate method was analysed for chemical consumed. Calculations on the chemical consumed in a sulphate digestion can be found in the Appendix (A4[f], pp. 155-156).

2.7.3 Estimation of total alkali

A 5 ml black liquor sample was ashed in a platinum crucible at 600° to 700°C. The white ash was then dissolved in water and titrated to pH 4.2 with HCl (0.1N), using methyl orange as indicator.

2.7.4 Estimation of active alkali remaining

A 25 ml black liquor sample was placed in a measuring cylinder and 50 ml of barium chloride (10%) added to precipitate the carbonate ion. This was made up to 250 ml using distilled water, mixed and filtered.

2.7 Analysis of pulping liquor

At the end of each pulping trial, a sample of the black liquor was collected for analysis.

2.7.1 Analysis of pH

The pH value of the black liquor was measured by a pH meter.

2.7.2 Chemical consumed in a sulphate digestion

A sample of the black liquor from each pulping trial by the sulphate method was analysed for chemical consumed. Calculations on the chemical consumed in a sulphate digestion can be found in the Appendix (A4[f], pp. 155-156).

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2.7.4 Estimation of active alkali remaining

A 25 ml black liquor sample was placed in a measuring cylinder and 50 ml of barium chloride (10%) added to precipitate the carbonate ion. This was made up to 250 ml using distilled water, mixed and filtered.

A 100 ml filtrate was then removed and titrated to pH 8.3 with HCl (0.1N), using phenolphthalein indicator.

2.7.5 Yield of pulp

The yield of pulp was calculated after the moisture content was determined. The method used to determine the moisture was similar to that described in Section 2.3.1. The yield of pulp was expressed as a percentage on the OD pulp.

2.7.6 Screenings

The uncooked material was collected and dried in the oven at 105°C. From the oven dry weight the percentage of screenings was calculated.

Calculations related to alkaline pulping are found in the Appendix (A4, pp. 151-157).

2.7.7 Kappa number of pulp

The Kappa number is the number of millilitres of tenth normal potassium permanganate solution which would be consumed by one gramme of moisture free pulp under the conditions specified in this standard. The results are corrected to 50 per cent consumption of the

permanganate. This method is based on "kappa number of pulp" in TAPPI T 236 (TAPPI, 1978).

Prior to weighing the pulp, it was conditioned in the atmosphere for 20 min. The amount of pulp, which is estimated by trial and error to consume approximately 50% of the permanganate was calculated. The permanganate consumption must be between 30 - 70% to comply with the standard.

The solutions required for the titration were potassium permanganate (0.1 ± 0.005N), sulphuric acid (4N) and sodium thiosulphate (0.2 ± 0.005N).

An example of the calculation for Kappa number is found in the Appendix (A4[h], pp. 157-58).

Bleaching

Bleaching trials were carried out on the sulphate pulp of the midrib, with a kappa number of 21.4, by a three-stage method using the CEH (Chlorination-Alkali extraction-Hypochlorite) sequence.

The conditions for chlorination were determined by the method described in "Raw Materials for More Paper" in F.A.O. (1953). Calculations related to bleaching are found in the Appendix (A6, pp. 159-165).

2.9 Preparation of handsheets

Handsheets were prepared according to the procedure described in the Second Report of the Pulp Evaluation Committee to the Papermakers' Association (Papermakers' Association, 1936).

The handsheets were dried in a constant humidity room maintained at 25°C and 80% relative humidity for at least 24 hours, in contrast to the standard conditions of 20°C and 65% relative humidity.

Calculations on trial sheets are found in the Appendix (A7, pp. 165-166).

2.10 Physical properties of handsheets

All sheet properties mentioned previously, with the exception of tearing resistance and folding endurance, were measured according to the appropriate TAPPI standards (TAPPI, 1978).

All mathematical conversions of test readings from the existing instruments to SI units are in accordance with ISO format.

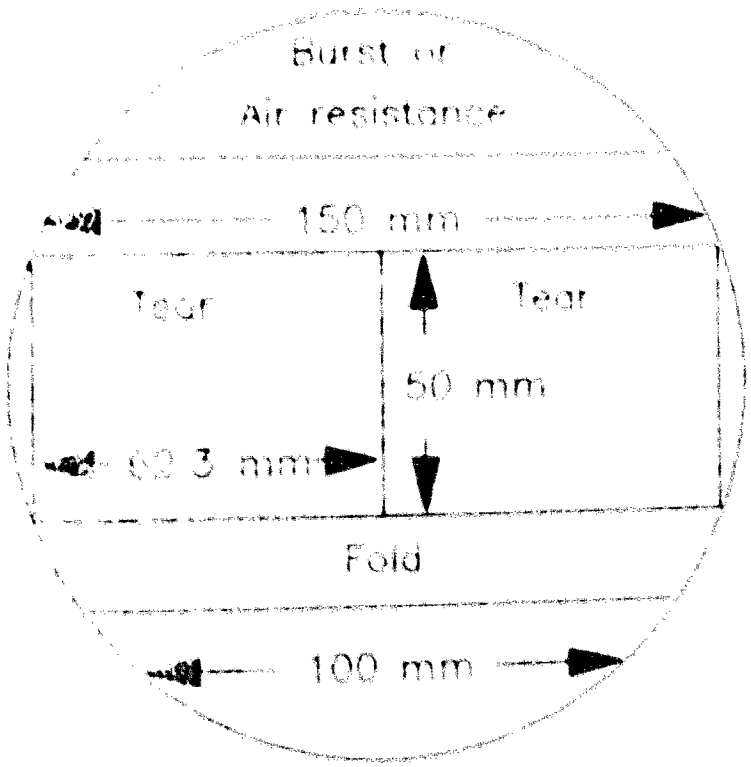
A set of at least eight acceptable handsheets of 60 ± 2 g/m^2 oven-dry grammage is required for testing.

2.10.1 Thickness

The rough edges of the sheets were firstly trimmed to avoid irregularities. The thickness tester was switched on for an hour prior to testing. A total of ten readings were taken. The mean thickness of a single sheet was then calculated in microns.

Preparation of test pieces for strength evaluation

Each handsheet sample was cut singly on a paper cutting as shown in the diagram below.



Accordingly each sample should give:

(1) Eight similar sectors (six for bursting strength and two for air resistance).
(2) Eight similar sectors for tensile strength tests.
(3) Eight similar sectors for four tearing strength tests in four-sheet piles.
(4) Eight similar strips for double fold tests.

2.10.2 Tearing index

The tearing index was measured on an Elmendorf tester according to TAPPI (1978).

2.10.3 Basis weight

After completing the above test, the torn pieces from the sample were placed in a tared weighing bottle and dried to constant mass in an oven maintained at 105 ± 2°C. The basis weight was then calculated.

2.10.4 Bursting strength

Bursting strength was carried out on an L&W electronic Mullen burst tester according to TAPPI T 403 os-74 (TAPPI, 1978).

2.10.5 Folding endurance

The folding endurance was measured with a Kohler-Molin fold tester using an applied load of 800 g (TAPPI, 1978).

2.10.6 Tensile strength

The Intellect 500 tensile machine was used for the testing according to TAPPI T 404 os-74 (TAPPI, 1978). A load cell of 100N was used for testing. The rate of break was maintained at 15 to 25 sec.

From this test, tensile index, breaking length, tensile energy absorption (TEA), TEA index and stretch values were calculated.

Calculations related to the physical tests of the handsheets can be found in the Appendix (A8, pp. 166-168).

2.10.7 Brightness

The brightness of the bleached handsheets was measured on a Technibrite Opacity and Brightness Tester. The readings were based on the absolute reflectance of the ceramic standard at 557 nm.