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MOISTURE RESPONSE OF THERMOMECHANICAL AND OTHER FIBRES

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Synopsis The objective of this study was to obtain a better understanding of the way in which the response of individual cellulose fibres to changes in relative humidity is relevant to the dimensional behaviour of paper and to the role of the fibres in composite materials, where dimensional instability is the main impediment to their wider use. The torsional response of individual pine tracheids to changes in relative humidity has been measured, and it is proposed that the observed angle of twist on drying a collapsed fibre is a function of the microfibril angle, the wall thickness, the fibre length and the fractional linear shrinkage across the microfibrils. A link between fibre twist and paper shrinkage is suggested. In thermomechanical pulps the temperature of defibration apparently affects the fibre twist in a way which is inversely related to the degree of fibrillation and fibre damage. The mechanical properties of wet fibre webs have been studied as a function of moisture content. The wet web strength and other properties of thermomechanical pulps depend on the fibrillation induced during refining which in turn depends on the relationship of the refining temperature to the lignin glass transition point.

Introduction

THIS investigation began as an attempt to obtain a better understanding of the response of individual cellulose fibres to changes in moisture content, with the ultimate objective of devising means of minimising the changes in those internal spatial relationships of constituent parts of the fibre which influence its overall dimensions and the stability of fibre assemblages. The problem is relevant not only to paper products in which dimensional stability is an important consideration, but to the role of cellulose fibres in composite materials, where changes in dimensions in response to varying atmospheric conditions constitute the main impediment to their wider use.

Influenced by the work of Mark and co-workers at Syracuse[1,2] we chose

the torsional response of individual fibres as the principal technique to be used in these studies. As the work progressed, a parallel study on factors determining the preparation of satisfactory thermomechanical pulps (TMP) led to our paying increasing attention to fibres of this type. We were also becoming increasingly interested in the wet web strength of thermomechanical and chemithermomechanical pulps (TMP and CTMP), and an attempt has been made to explore the extent to which the wet web behaviour is related to individual fibre properties. TMP and CTMP fibres are also of particular interest in composite materials other than paper, where the high pulp yields which can be obtained, coupled with the ease with which fibres may be separated in an undamaged state at temperatures above the lignin glass transition point, are important economic considerations.

Torsional response of fibres to humidity changes

WHEN a fully saturated cellulose fibre or tracheid is observed under a low power microscope it appears to have the form of a cylindrical tube. On drying without axial restraint to a moisture content in equilibrium with normal atmospheric conditions the cylinder collapses, unless the cell wall is thick in comparison to the lumen diameter, to a twisted ribbon-like shape with two or more complete twists per mm of its length. Raising or lowering the relative humidity of the surroundings between 30 and 70 per cent results in a repeatable untwisting or twisting of the fibre through an angle of a few degrees for each per cent change in relative humidity.

Cylindrical models have sometimes been used to describe the response of cellulose fibres to changing humidity, and while such an approach may be valid for wood, or even in the initial stages of the formation of the paper web, considerations based on the twisting behaviour of a flat ribbon are more closely related to paper performance. Using this approach, we have attempted—

(i) to measure the torsional response of single fibres under minimal tensile load over a range of relative humidities, and to develop a simple theoretical model to express this behaviour in terms of the geometry and microfibrillar organisation of the fibre, and
(ii) to correlate the dimensional instability of paper with fibre twisting.

Measurement of fibre twisting

THE torsional response of a single fibre was measured as the angle of rotation through which one end moved with respect to the other, when subjected to a known change in relative humidity. The apparatus consists of a Perspex cabinet fitted with a cylindrical fan providing a tangential air flow as shown in

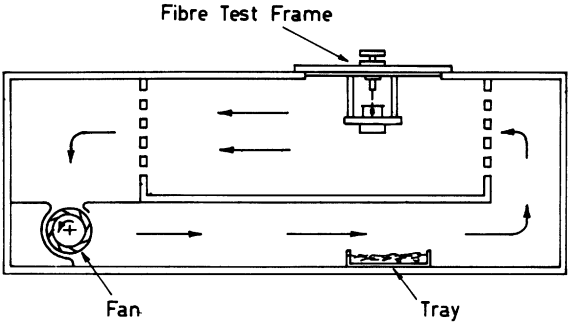

Fig. 1—Diagram of test cabinet

Fig. 1. A recirculating flow of air was maintained over a tray of either water, to increase the r.h., or lithium chloride, to decrease the r.h., which was measured on a Lambrecht hygrometer placed in the test area of the cabinet. This was calibrated to within two per cent r.h. by means of wet and dry bulb thermometers. Measurements were made for complete r.h. cycles by increasing the r.h. from below 30 per cent to above 70 per cent and then reducing it back to 30 per cent. The average time taken was 25 min for the increasing r.h. and 45 min for the decreasing part of the cycle. The work was done in an air-conditioned laboratory at *ca.* 20° C.

The manner in which the fibre was suspended for testing is shown in Fig. 2, which shows the testing frame attached to the cabinet lid. The arrangement allowed the fibre to be mounted in the frame with its axis horizontal, and then the complete assembly to be placed in the cabinet for testing, with the fibre axis vertical. The fibre was attached with epoxy adhesive between the tip of a sewing needle and the end of a 4·5 cm length of fine hypodermic tubing on which a small mirror had been fixed. The tubing and mirror were freely suspended from the lower end of the fibre during the test. The needle was

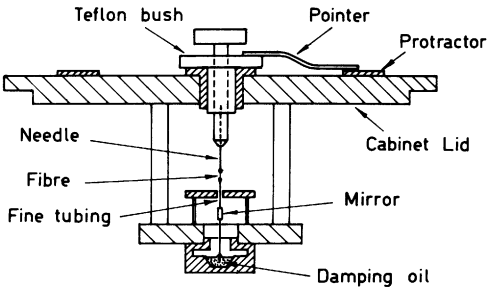

Fig. 2—Diagram of test frame

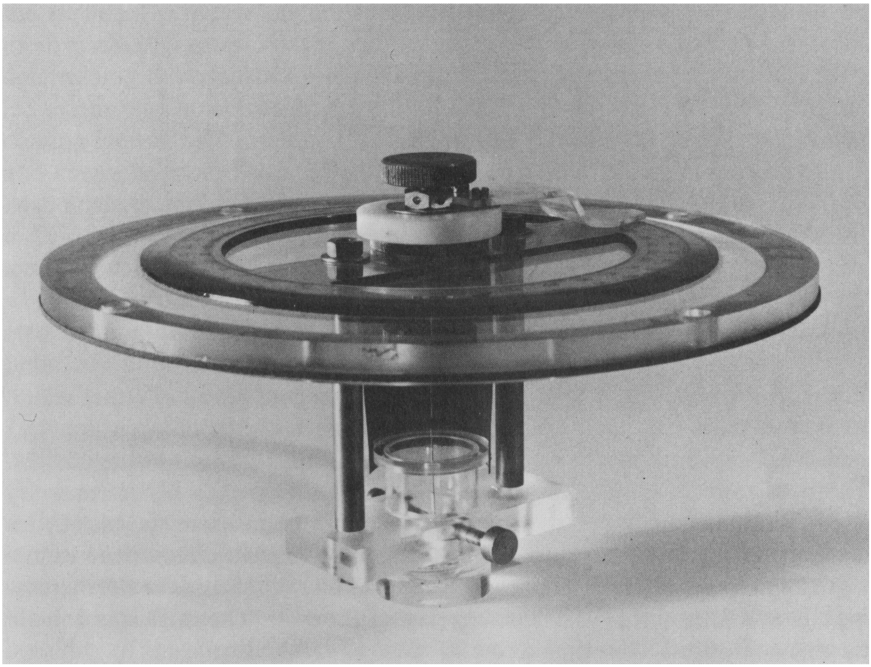

Fig. 3—Photograph of test frame

mounted in a Teflon bush in the cabinet lid so that the assemblage of needle, fibre, tubing and mirror could be rotated from outside the cabinet, and the angle of rotation measured by a pointer attached to the bush so that it moved over a full circle protractor attached to the cabinet lid (Fig. 3). A narrow beam of light was directed through the end of the cabinet and on to the mirror, the angular position of which was adjusted by rotation of the bush and pointer so that a bright image of the light source could be positioned on a scale on the side of the cabinet. This arrangement provided for measurement of fibre twist as the angle through which the upper end of the fibre had to be rotated to maintain a fixed angular position of the lower end, as indicated by a constant angle of deflection of light by the mirror. A set of individual units consisting of tubing and attached mirror were made up so that the corresponding set of fibres could be preserved for microscopic examination. The mass of each unit was *ca.* 20 mg. As the optical system was very susceptible to vibration with such a small mass, the apparatus was arranged so that the lower end of the tubing dipped into a small container of oil of low viscosity, thus providing the requisite damping of unwanted oscillations.

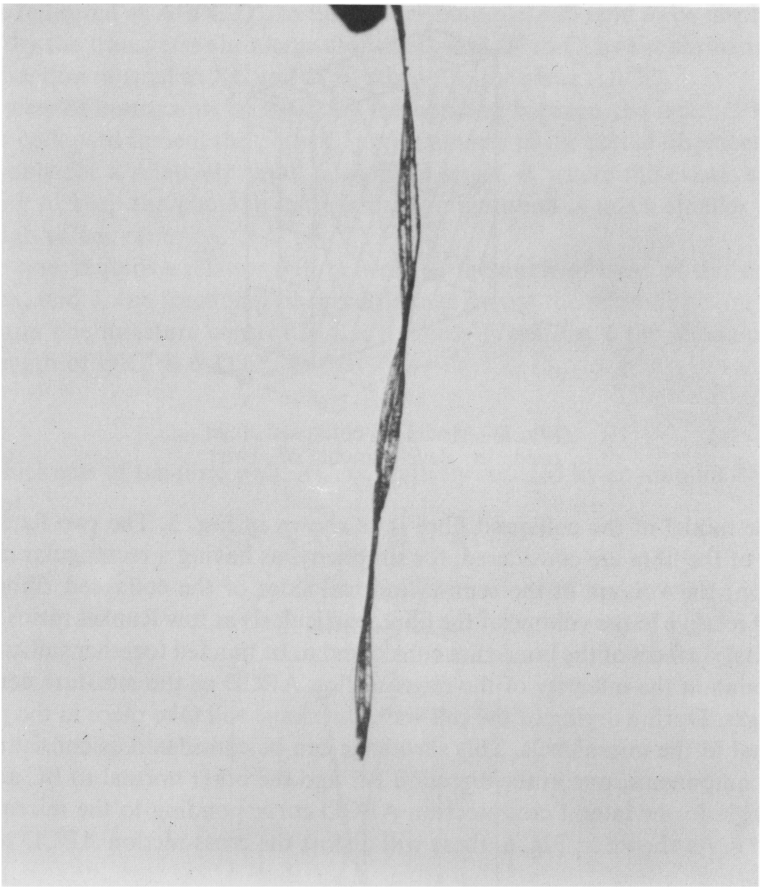

Fig. 4—A tested *Pinus radiata* kraft fibre. The free length is about 1·5 mm

Theory of twisting in collapsed fibres

THE loss of water from the lumen of a drying fibre can be seen under the microscope as a retraction of the meniscus from the end of the fibre, accompanied by collapse, and followed by twisting of the fibre. A twisted pine tracheid is shown in Fig. 4. On the basis of a collapsed fibre model de Yong[3] has proposed a geometrical theory of fibre twist, which predicts the amount of twist per unit length of fibre in terms of the linear shrinkage across the microfibrils, the thickness of the fibre wall and the microfibril angle. The essential features of this approach are as follows.

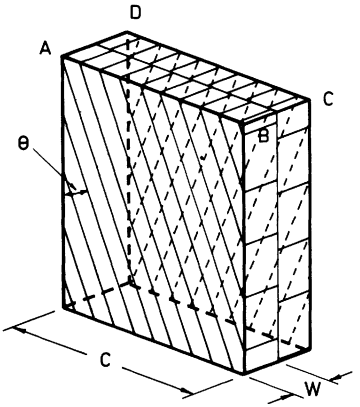

Fig. 5—Model of collapsed fibre used in development of twist theory

The model of the collapsed fibre is as shown in Fig. 5. The two flattened sides of the fibre are considered, for simplicity, as having a rectangular cross-section; the volume in the semi-cylindrical sides of the collapsed ribbon is small relative to the volume of the fibre, particularly at low Runkel ratios. The internal surfaces of the lumen are considered to be bonded together sufficiently to maintain the integrity of the cross-section ABCD as the moisture content changes. During drying of the cell wall, shrinkage will take place in the plane normal to the microfibrils. This shrinkage can be considered as consisting of two components, one in the direction BC and the other normal to BC and at an angle to the lateral cross-section ABCD corresponding to the microfibril angle θ. As shown in Fig. 6, these will distort the cross-section ABCD to the

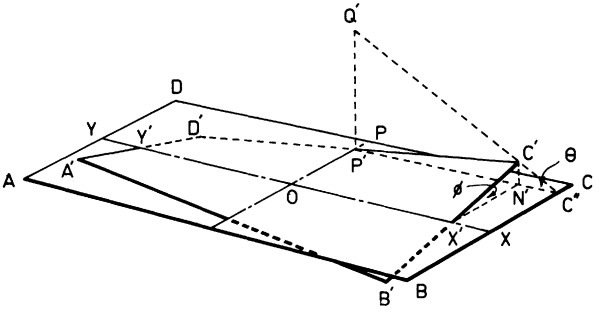

Fig. 6—Twist produced in cross-section of collapsed fibre by shrinkage across the microfibrils

shape indicated as A′B′C′D′, so that C, for example, will tend to be displaced to C″ by the transverse shrinkage along XC, and C″ to C′ by the shrinkage in the direction normal to XC and at an angle θ to the plane ABCD.

In view of constraints imposed by the bonding between the internal walls of the collapsed lumen, the point C′ approximates to the actual displacement of C only for a relatively small microfibril angle, θ, where the elastic stress tending to keep the plane in its original configuration is much smaller than for high values of θ.

We now require a relationship between ϕ, the angle of twist of this cross-section, and δ, the fractional linear shrinkage across the microfibrils on drying from one moisture content to a lower one. From Fig. 6 the reduction in the length of Q′C″ is $\delta \cdot$Q′C″, so that

$$C'C'' = \delta c/2 \cos\theta$$
$$\text{and } C'N' = C'C'' \cdot \sin\theta = (\delta c \tan\theta)/2.$$

The thickness of the fibre wall, XC, is similarly reduced by an amount $\delta \cdot$XC, so that

$$X'N' = XC(1-\delta) = w(1-\delta).$$

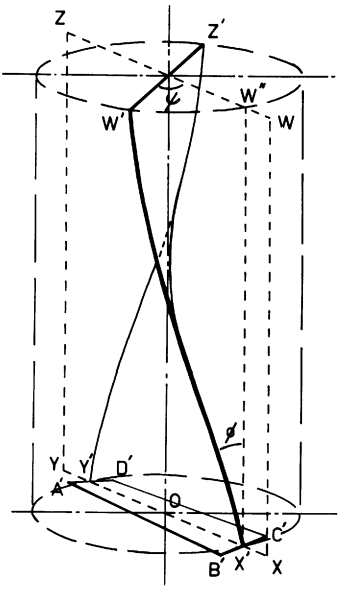

Fig. 7—Relationship between twist of cross-section and axial fibre twist

Thus,

$$\tan \phi = C'N'/X'N' = (\delta c \tan \theta)/2\, w\, (1-\delta), \quad . \quad . \quad (1)$$

where ϕ is the angle of twist (C'X'N' in Fig. 6).

We now have to relate this twist of the cross-section to the complete fibre twist, i.e. the rotation of one end of the fibre with respect to the other. In Fig. 7, the edges of the untwisted fibre are XW and YZ. As the cross-section ABCD twists, the fibre edges remain at right angles to the edges of the cross-section, B'C' and A'D', and so form two helices X'W' and Y'Z' with spiral angle ϕ.

As a consequence of shrinkage the radius of the cylinder of rotation decreases from c/2 to c(1 − δ)/2. If the angle of fibre twist is ψ radians, as shown, then the distance through which the radial projection of W moves around the shrunken cylinder is given by

$$W''W' = c(1-\delta)\psi/2.$$

If the twisted fibre lies in a cylinder of length L, then

$$\tan \phi = W''W'/L$$
$$= c(1-\delta)\psi/2L. \quad . \quad . \quad . \quad . \quad (2)$$

Combining equations (*1*) and (*2*), the specific fibre twist per unit length of fibre, ψ_s is given by

$$\psi_s = \psi/L = \delta \tan \theta/w(1-\delta)^2. \quad . \quad . \quad . \quad (3)$$

Equation (*3*) was tested for *Pinus radiata* RMP fibres over the relative humidity range 30–70 per cent, where the twist *vs* r.h. curve was sufficiently linear, after the first cycle, to permit the calculation of twist per per cent r.h. A value for δ of 0·0006 per per cent relative humidity change was used, as given by the observed fractional change of thickness of a paper sheet made from similar fibres. The microfibril angle θ was measured as 16° by X-ray diffraction,[4] and the cell wall thickness as 0·005 mm, from microscopic data.

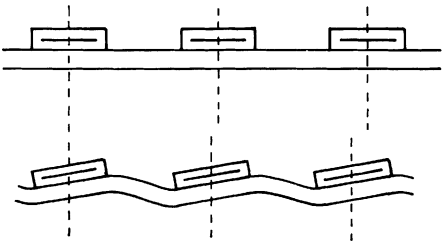

Fig. 8—Idealised scheme for relating fibre twist to lateral shrinkage of a paper sheet

This gives a value for ψ_s per per cent change in relative humidity of 2·0°, which can be compared with the value of 2·3° found experimentally.

In some instances individual cell wall thickness was estimated from microscopic examination of fibres after the twist had been measured. The microfibril angle was calculated from X-ray diffraction data.[4] The following results were obtained—

Wall thickness (μm)	3·8	4·5	5·6	6·3
Fibre type	earlywood	intermediate wood	latewood	latewood
Microfibril angle (°)	17	15	14	13
Twist angle (ψ_s per per cent r.h.)				
calculated	2·8	2·0	1·5	1·3
measured	2·1	1·9	2·7	1·3

More refined experiments along these lines are still in progress.

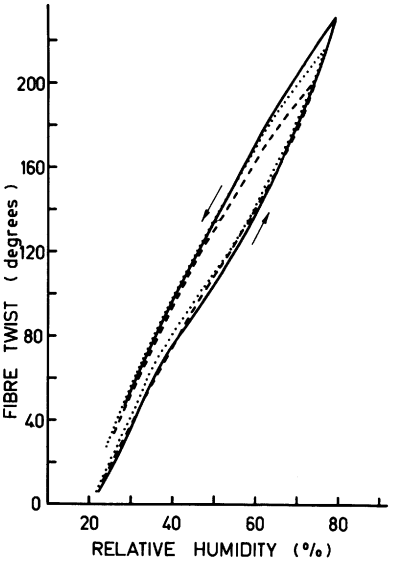

Fig. 9—Change in specific twist of a *P. radiata* RMP fibre during successive relative humidity cycles.

Cycle 1: – – –, Cycle 2: · · · · · ·, Cycle 3: — — —

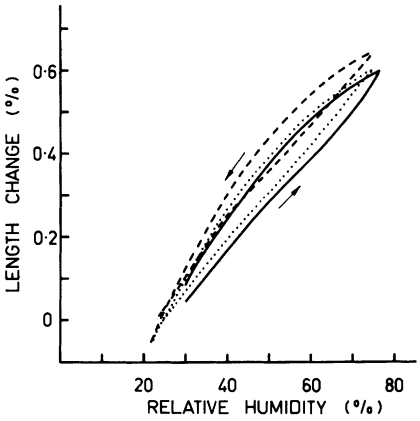

Fig. 10—Change in length of a strip from a *P. radiata* RMP handsheet during successive relative humidity cycles. Cycle 1: – – –, Cycle 2: · · · · · ·, Cycle 3: — — —

Analogy between fibre and paper properties

THEORIES of the mechanism of the dimensional instability of paper based on lateral swelling or shrinkage of the individual fibres are in a rather unsatisfactory state; in particular an acceptable mechanism is required to account for the change in effective fibre length between interfibre bonding sites. Steenberg[5] has suggested that 'the fibres may be creased between the joining points' and Page and Tydeman[6] have associated the change in length with 'kinks or microcompressions'. Perkins and Mark,[7] in developing their theory of the elastic behaviour of paper, have found it necessary to assume that the fibre segments are not straight. These suggestions seem to us to be consistent with the proposition that in layered structures a somewhat restricted but nonetheless significant twisting of fibres on drying will lead to bending in the fibre segments which cross them, as shown in an idealised way in Fig. 8.

In Figs. 9 and 10 it can be seen that the form of the hysteresis loop for fibre twist resulting from experiments in which the relative humidity is increased and decreased in discrete steps of 2 per cent resembles that for changes in strip length of paper made from the same pulp. In similar experiments the changes in thickness (Fig. 11) do not appear to be so closely related to fibre twist, and a complex mechanism may operate, involving perhaps both the direct effect of transverse fibre shrinkage, or swelling, and twisting of the fibres. In the experiments illustrated in Figs. 9 to 11 a period of several hours elapsed between the cycles.

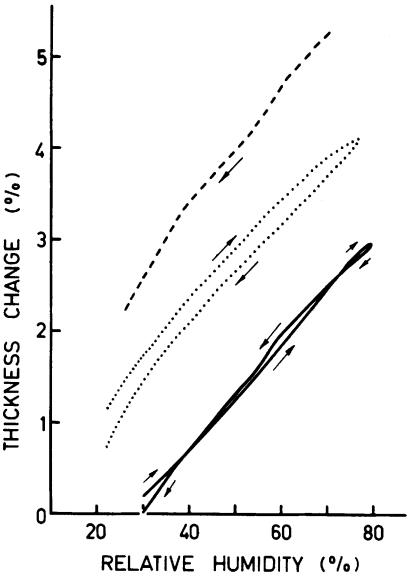

Fig. 11—Change in thickness of a strip from a *P. radiata* RMP handsheet during successive relative humidity cycles. Cycle 1: – – –, Cycle 2: · · · · · ·, Cycle 3: — — —

Effect of TMP temperature on torsional humidity response

A LABORATORY study has been made of the effect of the first stage refining temperature on the properties of *Pinus radiata* TMP pulps.[8] Saturated chips (64 per cent moisture content, green basis) were pre-steamed for 1½ min and defibred for 3 min in an Asplund Laboratory Defibrator, followed by washing of the pulp and post-refining in a Bauer Laboratory Refiner (two passes at 5 per cent stock concentration with the rubbing plates at 0·18 mm clearance).

In Fig. 12 the specific fibre twist is plotted against defibration temperature. Each vertical bar represents the range of six readings on an individual fibre, and the point marked on the bar is the mean value. As is usual with single fibre measurements the values show considerable scatter but three apparent effects can be discerned—

(i) an overall increase in twist with defibration temperature;
(ii) a lower twist value in the vicinity of the lignin glass transition point;
(iii) a possible grouping of the obervations for each temperature into high, low and intermediate values.

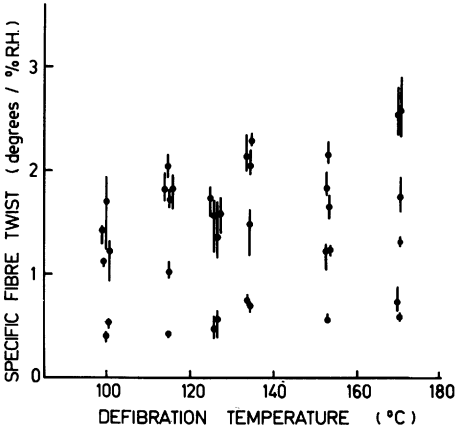

Fig. 12—Effect of defibration temperature on specific fibre twist for *P. radiata* TMP. Bars represent the range of values obtained in six tests on each fibre, and the mean is represented by a dot

The overall temperature effect could be connected with the degree of fibrillation and fibre damage, which is lower for pulps prepared at the higher temperature.[8] The fibre is protected from fibrillation by its lignin sheath, and the intact microfibrillar structure should lead to a greater twist response than in the case of a fibrillated fibre, where the spiral structure has been partially disturbed. A complication in this picture would be the decrease in collapsibility of the fibres separated at higher temperature, which is reflected in high bulk and poor bonding in the corresponding paper sheets.

The low twist near the glass transition point could also be a consequence of the high degree of fibrillation attained under those conditions.[8]

It was thought that the grouping of the observations for each temperature might be interpreted in terms of the fibre being derived from early, late or intermediate wood, with consequent differences in wall thickness and collapsibility. However attempts to eliminate variability by studying free fibres derived from a particular growth ring still yielded results with a significant scatter. It therefore seems likely that the main cause of variability may again be connected with the degree of fibre damage. If this were so, significance could still be attributed to the trend shown by the upper recorded values of twist, particularly where they tend to be grouped together for a particular temperature.

Wet web properties

THE effect of moisture content on fibre interactions can be conveniently studied by measuring wet web properties. Since the pioneering work of Brecht and Volk[9] and of Lyne and Gallay,[10] who measured the tensile strength of the wet paper web as an index of the behaviour of a particular pulp or furnish at the wet end of the paper machine, several groups of workers have attempted to determine which rheological properties of the web are the most significant. It has been realised that both the stress and the strain which the web can sustain are important. These quantities both contribute to the work to rupture (wet rupture energy) which Stephens and Pearson[11] have used to obtain a correlation with machine performance; the theory proposed is that, given a particular draw, the ability of the machine to run without excessive wet end breaks is dependent upon the energy capacity available to offset the effect of shives, and other discontinuities in the web, which lead to stress concentrations. On the other hand, Mardon and co-workers[12] have concluded that the energy to a given strain, taken as $3\frac{1}{3}$ per cent, is often a better parameter against which to correlate machine performance.

Details of the method of sample preparation and wet web strength measurement used in our work have been given in laboratory reports.[13] Briefly, 60 g/m^2 handsheets are formed normally on a British standard sheet machine up to and including the couching stage (dry blotters), and then pressed lightly between water-saturated blotters to reach a moisture content of 400–200 g per 100 g oven dry solid (the exact figure depending on the nature of the pulp). Test strips are then cut from several sheets, and tested by loading to rupture over a range of moisture contents on a rheometer operating at a constant loading rate, the standard rate adopted being 24 g/s.

The rheometer is a modified version of the microrheometer developed by de Yong[14] for tensile tests on single wood fibres, particularly those of *Eucalyptus* species, in which the fibre length rarely exceeds 1 mm. In order to avoid the problem of developing a highly precise driving mechanism for producing the very small extensions characteristic of such fibres the usual procedure of measuring the loads produced at given extensions was passed over in favour of a design in which the load is generated electromagnetically, and the resulting extension is detected with an inductive displacement transducer. The strip-holding section is closely modelled upon that used by Stephens and Pearson.[11] The strip is clamped and tested horizontally, to avoid plastic flow under the weight of the strip alone. A number of other measures have been developed to ensure accuracy and reproducibility.[13]

Strength and rheological properties of the wet web measurable in this way are—

1. Maximum Tension (MT), or load to rupture, expressed in g/cm (i.e. of strip width).
2. Maximum Stretch (MS), or extension at rupture (per cent).
3. Wet Rupture Energy (WRE), or work to rupture—the area under the load–extension curve, up to the point of rupture.
4. Energy to a pre-determined strain—e.g. the Wet Web Strength (WWS), the energy or work to 3½ per cent strain, as used by Mardon *et al.*[12]
5. The Tensile Modulus—the initial slope of the load–extension curve. In dry paper the initial part of the curve is commonly linear, but this appears to be rarely so for the wet webs we have investigated.

Correlations which have been suggested between runnability and wet web strength appear to rely invariably upon data obtained from handsheets tested on laboratory equipment at necessarily somewhat arbitrary rates of loading or extension. There still appears to be considerable uncertainty as to which of the above properties is the most significant in paper machine operation, and in the absence of mill data on our pulps we shall give considerable weight to MT, together with the more variable, but obviously important, MS.

Effect of moisture content on wet web properties

THE tension developed upon drying a kraft paper strip to an arbitrary moisture content rises with the degree of beating.[15] Can we reconcile this with the suggestion made earlier that the twist of contiguous fibres could play a significant part in the longitudinal shortening of the free length between bonding sites? Certainly if the more highly fibrillated fibres show less twist, as we have also suggested, this, in itself, would tend to reduce the contraction, but insofar as the contiguous fibre is also highly fibrillated it is thereby rendered more flexible and hence more prone to accept creases, kinks and microcompressions. So the resolution of this question would depend on a rather complex stress analysis.

In the present work the change in wet web properties as drying proceeds has been measured on many pulps.[13] The observations in the second stage of drying, when air intrusion becomes marked, support the view of Robertson[16] that 'although interfibre bonds are presumably increasing, the ability of the stressed webs to maintain sliding contacts or to reform contacts decreases as the water is removed. This is shown by the rapid decreases in stretch in this region and the observed losses or relatively modest gains in strength'.

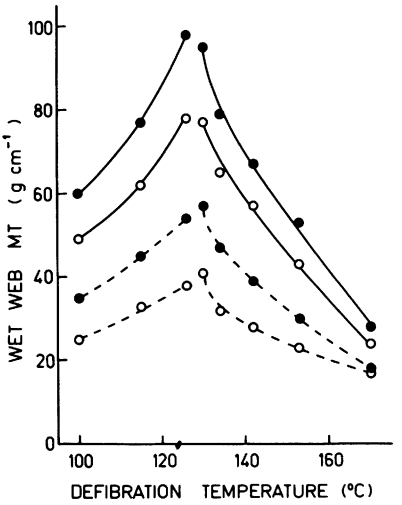

Fig. 13—Effect of defibration on wet web maximum tension of *P. radiata* TMP sheets. From the bottom, the curves represent moisture contents of 400, 300, 200 and 150 g per 100 g oven dry solid, respectively

Effect of TMP temperature on wet web properties

THE wet web properties were measured on the *Pinus radiata* TMP pulps prepared at various temperatures, as described earlier. As shown in Figs. 13 and 14, both MT and MS pass through sharp maxima just below 130° C. The curves given apply to critical regions on, say, a newsprint machine, where the strength of the wet web becomes of vital importance to the mill performance at moisture contents of about 350–400 g per 100 g o.d. pulp for an open couch transfer machine, and at about 150–200 g for a pick-up machine. Pulp and paper properties such as freeness, bulk and dry strength show cusps in the curves representing their dependence on defibration temperature (Fig. 15) in a similar way to the wet web properties.[8] Scanning electron micrographs of these pulps, kindly prepared by Professor A. B. Wardrop, showed very distinct differences in fibre structure at temperatures below, at and above the cusp, which is regarded as being close to the lignin glass transition point under the defibration conditions used.[8] The first régime (100–125° C) showed fibrillation accompanied by fibre damage; in the second (transitional) régime (*ca.* 125–135° C) fibrillation was more pronounced, leading to an almost

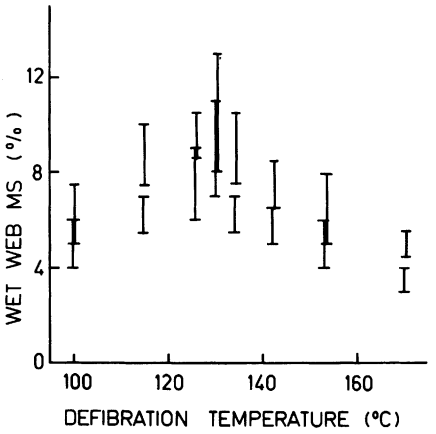

Fig. 14—Effect of defibration temperature on wet web maximum stretch of *P. radiata* TMP sheets. As experimental scatter is high, MS values at each temperature are shown as ranges rather than as single points. Wet web moisture contents are respectively 400 g and 150 g per 100 g oven dry solid for the upper and lower ranges at each temperature

colloidal system of dispersed surface microfibrils near the refining temperature corresponding to optimum properties; and in the third régime (135–170° C) we find the smooth, lignin-encased, unfibrillated fibres characteristic of higher refining temperatures.

Relationship between fibre response and wet web behaviour

HIGH wet web strength has been shown[17] to be associated with the presence of fibre kinks, and a slight increase in kinking was observed on drying and rewetting a pine kraft pulp. However it is not known whether the fibre twisting effect is related in any significant way to wet web properties. It seems rather unlikely that it would affect the behaviour at the moisture contents encountered at the wet end of the paper machine, but at a later stage of drying it could well influence cockling and associated effects.

Acknowledgements

The authors are indebted to Mr R. McNamee, who assisted in the construction of the apparatus for measuring fibre twist and made many of the measurements, and to Dr V. Puri who prepared the thermomechanical pulps.

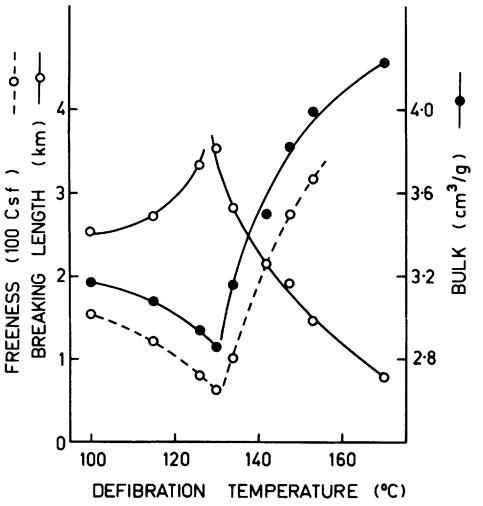

Fig. 15—Effect of defibration temperature on Canadian standard freeness, bulk and breaking length for *P. radiata* TMP, showing the cusps in the vicinity of the lignin glass transition point

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Discussion

Prof. B. K. Steenberg Dr Higgins, as you come from Australia you probably know that you can break a ship's mast if you tighten the ropes in dry weather and then get them wet. The twisting forces are considerable. You showed the specific twist plotted against the temperature of defibration. There was some scatter. Have you applied any statistical test to this to show that there is really a change with temperature?

Higgins No.

Steenberg You have plotted several variables against the temperature of defibration. Is this the important variable? I have data that lead me to believe that power consumption is. How long were the chips at the specified temperature and what was the actual temperature in the defibration zone?

Higgins In our experiments we had constant refining conditions, namely three minutes in a laboratory defibrator, type D, preceded by $1\frac{1}{2}$ minutes steaming. I would agree that it is a matter of energy input. In one way you could look at the properties shown on my last slide as dependent on freeness which falls more rapidly at the critical temperature.

Dr A. de Ruvo We at STFI have some experience in this field and we would like to compliment you on your experiments. Have you tested your fibres as a function of temperature and have you done any mechanical tests?

Higgins I will pass your question on to Mr de Yong if I may.

de Yong We have not done any mechanical tests on individual fibres. The wet tester described was a modification of our original single fibre tester. We believe that the testing of individual fibres is very time consuming and not too rewarding for the effort involved.

Under the chairmanship of B. W. Attwood

Dr D. H. Page In paper the picture is so complex that no new hypothesis can be ruled out. However, I must say that I believe that fibre twist is of minor importance for paper shrinkage for two reasons. Firstly, the tendency of fibres to twist would oppose one another. Secondly, I calculate a tilt angle of 8 degrees is necessary for a 1 per cent shortening. If this were the case it would have been observed in our microscopic work on fibre–fibre contact areas. Since we only had a depth of field of 1 μm any twist at the bond sites would have caused them to go out of focus, and we could not have taken the photographs. So I believe that while twisting cannot be ruled out, it is not important in most papers.

Higgins Thank you. I agree that the twisting mechanism could be regarded only as a perturbation on your mechanism.

Dr E. L. Back I would like to add some information on the dimensional stability of these fibres in the temperature range 100–180° C. We know that the hemicellulose content of these fibres will be reduced. There might be a dissolution of $\frac{1}{2}$ per cent at 100° C to 6–8 per cent at 180° C in 3 minutes treatment time of pinus wood. This will improve the dimensional stability considerably. This is general experience in hardboard production.