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A method for determining the fibrillar angle in wood tracheids

by D. H. PAGE, *Pulp and Paper Research Institute of Canada, Pointe Claire, Quebec, Canada*

SUMMARY

The predominant direction of fibrils in isolated walls of wood tracheids can be determined from observation of the extinction position in the polarizing microscope. This method cannot be used directly to obtain the fibrillar direction for separate intact cells, because the light path must traverse the opposite walls of the cell, the fibrillar angles of which are similar but of opposite sense.

This difficulty may be overcome by impregnating the cell lumens with mercury and using incident (epi-) illumination with crossed polars. The incident light now passes through the upper wall of each cell, suffers reflection at the mercury surface and returns through the same wall. The extinction position gives the predominant fibrillar direction of the upper wall.

The method is rapid, has been found suitable for tracheids in wood, wood pulp and paper, and should be of value for the study of other plant cells such as cotton.

INTRODUCTION

The walls of the fibres (the term 'fibre' will be used throughout to indicate any thickened elongated cell in accordance with technological rather than botanical practice) of woody plants are thickened by a series of concentric layers of microfibrillar cellulose, embedded in a matrix of hemicelluloses and lignin. Four principal layers, termed the primary (P), outer secondary (S_1), middle secondary (S_2) and inner secondary (S_3) layers are distinguished. Each has a distinctive arrangement of microfibrils as shown in Fig. 1. The majority of the cell wall material, perhaps 80–95%, is contained within the middle secondary layer within which the microfibrils are locally parallel and trace a steep spiral around the fibre axis. The angle between the fibrillar direction and the fibre axis (termed the fibrillar, micellar or spiral angle) varies between fibres within trees and between trees depending on growth mechanisms that are incompletely understood.

Because the cellulosic fibrils have a high longitudinal modulus compared with their transverse moduli and the moduli of the encrustants, the mechanical properties of fibres are controlled to a large extent by the magnitude of the micellar angle. Fibres with high micellar angles have low tensile moduli and high breaking extensions (Spark *et al.*, 1958; Cowdrey & Preston, 1966; Tamolang *et al.*, 1967). These fibre properties appear to be reflected in paper properties, for a close correlation has been found between the micellar angle of fibres and the breaking extension of paper sheets prepared from them (Watson & Dadswell, 1964;
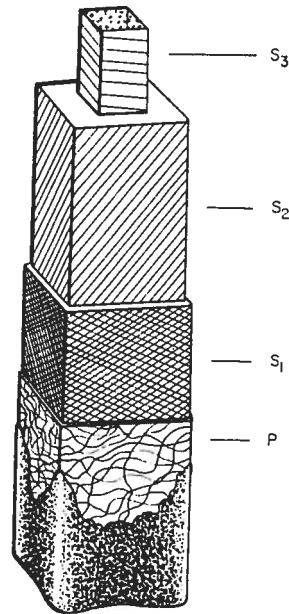
Fig. 1. Schematic representation of a single fibre of wood showing the arrangement of the cellulosic microfibrils in the various layers of the cell wall.
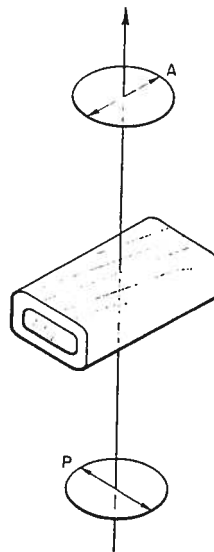

Fig. 2. In transmitted light, with crossed polars, an intact fibre is extinguished when it lies parallel to the plane of one of the polars. No information can be obtained on the magnitude of the micellar angle.

Guha, 1961). It is therefore of some technological importance that methods should exist for the determination of the micellar angle in wood and wood pulp fibres.

In a recent article Meylan (1967) described experimental comparisons of some of the presently available methods. These are:

(a) Measurement of the spread of the (002) arc in the X-ray diffraction pattern (Preston, 1946);

(b) Light microscopy of individual fibres stained with iodine to reveal the fibrillar direction (Bailey & Vestal, 1937);

(c) Polarized light microscopy of fibre sections cut at various known angles to the fibre axis (Wardrop & Preston, 1947, 1951; Manwiller, 1966);

(d) Light microscopy of longitudinal wood-sections or single fibres by observation of such features as striations, cracks and pits that align with the fibrillar direction (Bailey & Vestal, 1937);

(e) Polarized light microscopy of macerated fibres sectioned longitudinally (Preston, 1934).

For general use all the above methods have certain restrictions. Methods (a) and (c) require perfect alignment of the fibres and are applicable, therefore, only to wood; methods (b) and (d) are both tedious and are not applicable with certainty to all fibres; method (e) while most general is not applicable to many wood pulp fibres the lumens of which are partially collapsed or distorted from their original shape. The present report suggests a method that is more general and less tedious than those mentioned above, and has the further advantage that it is non-destructive.

METHOD

The polarizing microscope is a powerful tool for obtaining the predominant fibrillar direction in fragments of cell walls. The fibrillar direction in a cell-wall fragment coincides with the direction of maximum refractive index and this can be determined with great accuracy from the position at which the image extinguishes under crossed polars. However, this principle cannot be applied directly to determine fibrillar directions in the walls of intact fibres. For a fibre lying in the plane of the microscope slide the illumination must traverse both the lower and upper wall. The fibrils of the middle secondary layer spiral around the fibre so that on opposite walls the angle between the fibrils and the fibre axis is in the opposite sense. Thus, under examination in transmitted light (Fig. 2) the birefringences of the two opposite walls tend to compensate one another and to a first approximation the fibre behaves as a uniaxial crystal with the direction of maximum refractive index along the fibre axis and independent of the micellar angle. (This is not true for fibres with appreciable wall thickness; the optical theory of the system is then more complex (Preston, 1947). However, it remains true that information on the magnitude of the micellar angle is not readily attainable by direct observation of intact fibres in polarized light.)

It was to overcome this difficulty that Preston (1934) proposed the method of longitudinal sectioning of macerated fibres. In this way one of the opposite walls of the fibres is removed and light passes only through the other wall. It occurred to the author that the same principle could be applied without the need for sectioning. The method requires that mercury should be introduced into the lumens of the fibrous sample. When a fibre so impregnated is examined in a polarizing microscope using *incident* (epi-) illumination, the light passes through the upper wall of the fibre, suffers reflection at the mercury surface and exits through the *same wall*. With the polarizer and analyser crossed, extinction occurs

when the fibre is inclined at the fibrillar angle to one of the polars. The method is made clear from Fig. 3. The fibrillar angle is the angle between the axis of the fibre in its extinction position and the direction of one of the polars. Since micellar angles rarely reach 45°, ambiguity about the complementary angle to be chosen rarely arises but when it does it may be resolved by use of a compensator.
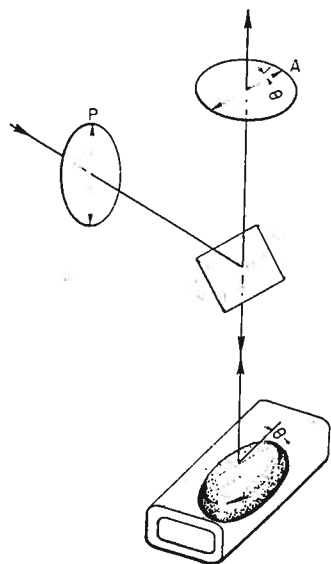

Fig. 3. Under epi-illumination with crossed polars a fibre with a mercury-filled lumen has an extinction position when the fibrils of the upper wall are parallel to the plane of one of the polars. The micellar angle is determined as the angle between this plane and the axis of the fibre in the extinction position.

In practice the method works with simplicity and reliability. Mercury is introduced into the fibre lumen under pressure. For this purpose a mercury porosimeter has been available, but it would seem that a much simpler system could be constructed and operated satisfactorily. The sample of fibres is inserted in the porosimeter cell, mercury is introduced and the pressure is raised to about 1000 psi. (It is established that, at this pressure, the lumens of the fibres are totally penetrated (Stone *et al.*, 1966).) The pressure is then released. Some of the mercury escapes, but the columns of mercury in the lumens frequently break at points of constriction, in the same way that the column breaks in a clinical thermometer, so that most fibres retain an appreciable amount of mercury in the form either of droplets or of entire columns. The sample is then ready for micr-scopical examination. The method can be used for samples of wood, wood pulp or paper. In the case of wood the only prerequisite is that a face be exposed that has been split along the middle lamella. It is necessary, therefore, to subject the sample to a mild delignifying action prior to splitting. Pulp fibres may be examined by impregnation in a fluffed state or after drying onto a glass slide. A paper sample may be impregnated and examined directly without need for separation of the fibres. It must be recognized though that, in this case, some of the fibres

will be in a collapsed state and hence will not be impregnated. Examples of the method are given in Figs. 4 and 5. Fig. 4 (a) shows a radial wood surface with the polarizer parallel to the axes of the fibres. Three latewood fibres are seen at the top of the field and below them four earlywood fibres of the following annual
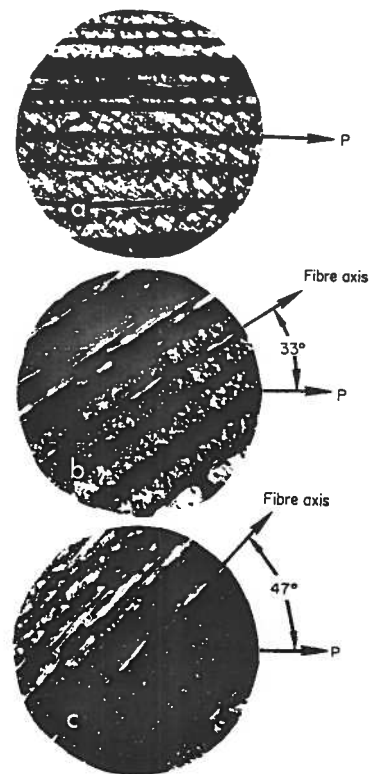

Fig. 4   Fig. 5

Fig. 4. Radial surface of wood with lumens impregnated with mercury. Epi-illumination with crossed polars (×176). The impregnated fibres are bright when the fibre axes coincide with the plane of polarization (a). The micellar angles are, for the three latewood fibres, 33° and, for the four earlywood fibres, 47° as shown from their extinction positions (b) and (c).
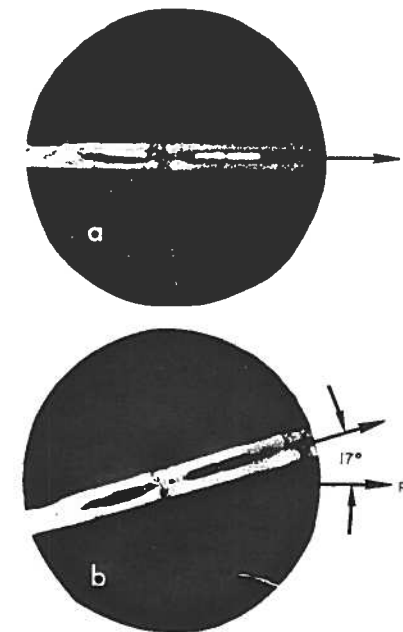
Fig. 5. Commercial unbleached kraft fibre, mercury impregnated and wet mounted. Epi-illumination with crossed polars (×176). The extinction position (b) indicates a micellar angle, in the water-swollen state, of 17°.

ring. A rotation of the specimen by 33° with respect to the polarizer and analyser causes extinction of the three latewood fibres as shown in Fig. 4 (b). The four earlywood fibres, however, remain bright and are not extinguished until rotation of the specimen has reached 47°, as in Fig. 4 (c). Fig. 5 shows the method applied to single pulp fibres.

Using this method it is possible to obtain rapidly a mean value of micellar angle and its distribution for samples of pulp and paper. An illustration is given in the histogram of Fig. 6. This is typical of the distribution of micellar angles
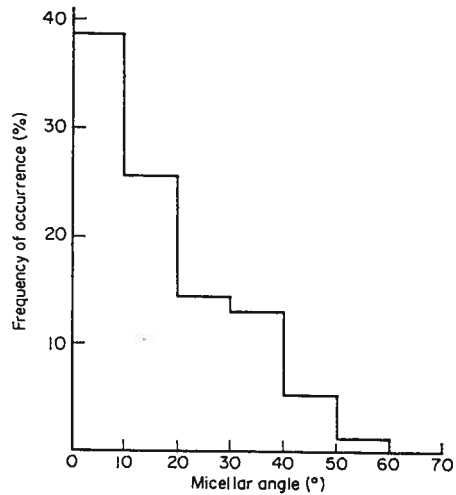

Fig. 6. Frequency distribution of micellar angle in fibres from a commercial kraft pulp from eastern Canada. Mean micellar angle 16·5°; $N = 78$.

in the few east Canadian pulps examined by the author. West Coast pulps tend to have lower micellar angles. This difference between pulps from different origins indicates that the measurement of micellar angle may indeed have appreciable technological significance.

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