Measuring the wetting angle and perimeter of single wood pulp fibers: a modified method

John H. Klungness
Forest Products Laboratory, Forest Service, U.S. Department of Agriculture, Madison, Wis. 53705

KEYWORDS: **Pulps • Fibers • Wetting • Contact angle**

In pulp processing development it is often necessary to measure theeffect of a process variable on individual pulp fiber wettability. Such processes would include drying of market pulps, recycling of secondary fibers, and surface modification of fibers as in sizing. However, if wettability is measured on a fiber sheet surface, the results are confounded by variations in fiber network morphology.

These variations may be avoided by measuring directly individual pulp fiber wettability. Wilhelmy's formula (*1*) shows the relationship between the pull (F) exerted on a solid rod inserted into a mass of liquid and the wetting angle to be:

$$F = P\gamma_{LV}\cos\theta \qquad (1)$$

where

P = perimeter of the rod along the three-phase boundary line

γ_{LV} = surface tension of the liquid

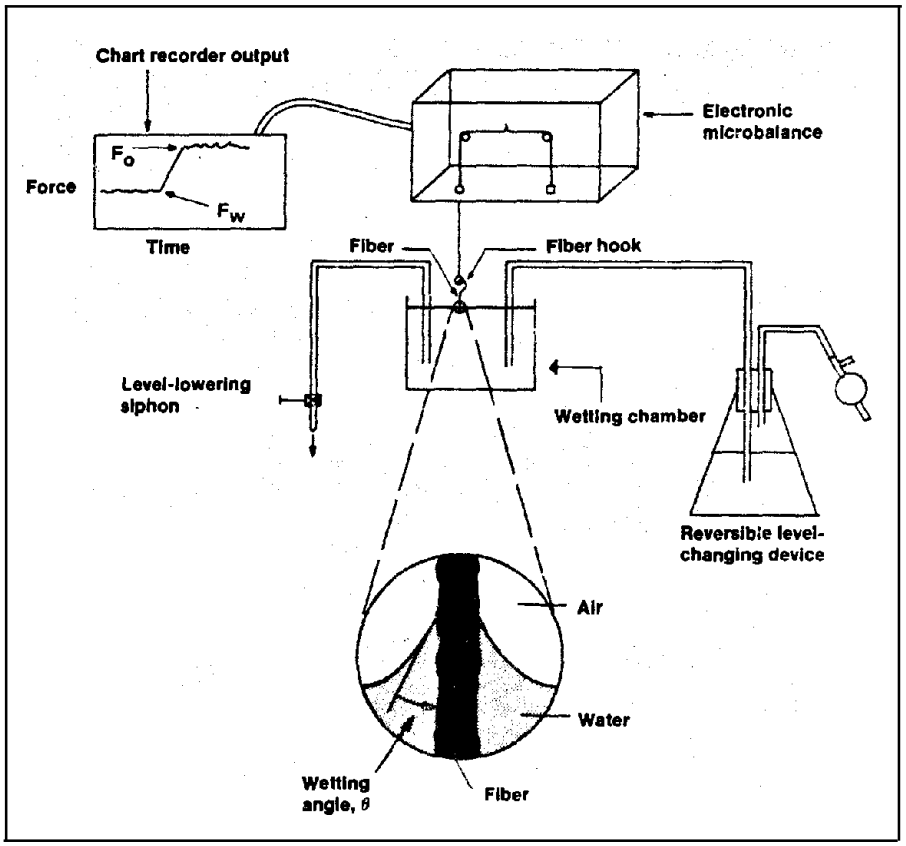
θ = contact angle.

Miller and Young (*2*) have used this relationship to determine the wettability (W) of uniform cross section synthetic filaments that has been defined as (F/P). With circular filaments, the wettability can be easily determined directly from a plot of F vs. the depth of immersion of vertical filaments, an approach that eliminates the buoyancy effect. The perimeter is obtained from diameter measurements for round cross sections; for noncircular filaments the perimeter is more difficult todetermine and requires cross-sectioning of the filament and measurement of the perimeter by microscopic techniques. When F and P are determined and γ_{LV} obtained from the literature, the contact angle θ can be calculated.

Young (*3*), in his work with single wood pulp fibers, used a light microscope equipped with a calibrated eyepiece to measure the fiber perimeter. He has determined that the buoyancy effect of pulp fibers may be neglected; it is small compared with the attraction force because wood pulp fibers are usually much smaller in diameter than synthetic filaments. The buoyancy force decreases with the square of the fiber radius while the force of liquid attraction to the fiber only decreases in direct proportion to the fiber radius. However, these results depend on accurate measurement of the perimeter of the fiber at the precise location of the liquid surface on the fiber where F is measured. This measurement is extremely difficult to make since the pulp fiber is nonuniform in cross section along its entire length.

In this article we report a much simpler method for establishing the perimeter at the desired location on the pulp fibers. A slightly modified device originally described by Miller and Young (*2*) was used (Fig. 1). In place of the reversible elevator for raising or lowering the liquid level, a less expensive but effective siphon arrangement was substituted.

The technique hasevolved from work by Miller (*4*) that has shown that if a uniform synthetic filament is cut squarely at the end and immersed in water,



1. Apparatus for measuring single-fiber wettability.

I. Circumference of rayon fibers by cross-section measurement vs. wettability technique

Fiber type	Strand number	Fiber circumference: Cross section measurement, mm × 10^{-2}	Fiber circumference: Wettability method, mm × 10^{-2}	Difference, %
A	1	8.69	8.41	-3.2
	2	9.40	10.86	15.5
B	1	6.23	5.70	-8.5
	2	6.97	5.82	-16.5
	3	6.94	5.75	-17.1
C	1	3.36	2.95	-12.1
	2	3.52	2.82	−19.9
D	1	4.12	3.96	-3.9
	2	4.13	3.8	-6.1
Average				-8.0

II. Wetting angle for oven-dried loblolly pine kraft at three lignin levels

Lignin content of pulp, %	Wetting angle (degrees) sample mean	Number of fibers	Number of measurements[a]	Standard deviation between fibers	Standard deviation within fiber
16.2	48.75	19	91	21.29	14.16
4.8	43.32	23	110	22.54	12.93
0.2	20.82	19	98	23.83	9.13
Overall		61	299	22.58	

[a]One to nine measurements per fiber.

III. Wetting angle differences between three lignin levels of once-dried loblolly pine kraft pulps (in degrees)

Pulp comparison	Differences between means: Wetting angle measurement	65% confidence limits[a]	95% confidence limits[a]
16.2-4.8%	5.3	±5.25	±8.35
16.2-0.2%	27.93	±5.39	±8.58
4.8-0.2%	22.5	±5.14	±8.19

[a]Multiple comparisons based on variance between fibers.

the wetting angle will equal zero just prior to breaking contact with the liquid. On a recording device, the force just before breaking will then register maximum, because cos θ increases to its maximum of one. Thus, Eq. 1 may be written:

$$P = \frac{F_o}{\gamma_{LV}} \qquad (2)$$

where F_o = force at θ = 0°.

It also follows then that

$$\cos\theta = \frac{F_w}{\frac{F_o}{\gamma_{LV}} \cdot \gamma_{LV}} = \frac{F_w}{F_o} \qquad (3)$$

where F_w = the force recorded at equilibrium just prior to lowering the level.

After immersing a single fiber into water, the force F_w is then equal to the equilibrium force recorded before the water level is lowered. F_o is the first force indicated on lowering the water level. This force will not necessarily stabilize or remain constant if the water level continues to recede but will instead trace a profile of the perimeter of the fiber at the line of contact between the surface of the water and the fiber.

We have found with wood pulp fibers that F_o at θ = 0° can be measured (by continuous movement of liquid across the fiber) because F_o is not rate-dependent over a wide range of level range change. Thus, the ratio of the two wetting forces gives an accurate determination of the wetting angle, since the circumference is measured at exactly the location of both wetting force measurements. It is more rapid because no fiber cutting is required. Also, repeated attempts to cut fibers squarely resulted in distorted fiber ends.

The circumferences of various, types of commercial rayon fibers were measured by this method (wettability method) and compared with those measured by observation after using the cross-sectioning method described by Horn (5). The circumference values by the wettability method (Table I) averaged 8% lower, and the difference was assumed to be the result of distortion of the fibers while imbedding them between the two cellulose acetate sheets for cross-sectioning with a microtome.

Wetting angles of three loblolly pine kraft pulp fibers varying in lignin level were also determined by this method (Table II). The wetting angle decreased with lignin level, which was expected since lignin is more hydrophobic than cellulose. The values also did not deviate significantly from any of the few previously reported contact angles (*3, 6*).

The reduced variability of wetting angle within a fiber as the lignin level decreased is expected because with less lignin a mechanically smoother and less chemically variable surface results. But, when comparing pulps, two types of variability must be considered: The variability of measurements within a fiber and the variability between fibers. To compare pulps statistically, one must use the variability between fibers, which is largely caused by the heterogeneous nature of wood pulp. [Young (*3*) discusses the factors affecting the surface chemistry and smoothness of pulp fibers in detail.] Measurements on approximately 20 fibers of each pulp showed significant differences between 4.8% and 0.2% lignin at the 0.0001 level, between 16.2% and 0.2% lignin at the 0.0001 level, and between 16.2% and 4.8% lignin at the 0.315 level. To obtain a significant difference between the 16.2% and 4.8% lignin at the 0.05 level would have required measurements on approximately 60 fibers (with five measurements per fiber). Table III gives 65% and 95% confidence intervals for the difference between lignin contents.

We believe the modified method developed is more rapid and at least as accurate as any known method for measuring perimeters and determining wettability of pulp fibers.

Literature cited

1. Wilhelmy, J., *Ann. Physik* **119:** 177(1863).
2. Miller, B., and Young, R.A., *Textile Research Journal* **45**(5): 359 (1975).
3. Young, R.A., *Wood and Fiber* **8**(2): 120 (1976).
4. Miller, B., Textile Research Institute, Princeton, N.J., personal communication, 1980.
5. Horn, R.A., and Ellickson, S.C., USDA For. Serv. Res. Note FPL-022, 1964.
6. Valko, E.I., *in* "Chemical After-Treatments of Textiles," (N.S. Wooding, and S.M. Atlas, eds.), Wiley-Interscience, New York, 1971, p. 91.

The author gratefully acknowledges the laboratory work performed by James A. Harloff.

The Forest Products Laboratory is maintained at Madison, Wis.. in cooperation with the University of Wisconsin.

Received for review June 9, 1981.

Accepted June 17, 1981.