A Study of X-Ray Orientation in Never-Dried Cotton Fibers

Investigations in the recent past have led to the opinion that the spiral angle of fibrils in different genetic varieties of cotton is more or less the same and that the differences found in X-ray orientation angle measured on fiber bundles are due largely to the influence of convolutions formed during the initial desiccation of fibers in the cotton bolls. It was Meredith,[1,2] who observed for the first time that the spiral angle deduced from refractive indices is nearly the same (ca. 21°) for all varieties of cotton when the effect of convolutions is corrected for. An extension of this work by Betrabet et al.[3] to Indian cottons also led to the same conclusion.

More recently, Hebert[4] measured the spiral angle by Hartshorne's optical method[5] and confirmed the earlier observation that spiral angle of cotton is a constant at around 21°. Duckett and Tripp[6] using a single-fiber X-ray technique showed that the variation in spiral angle among different varieties is small. Morosoff and Ingram[7] examined the interconvolution tracts by X-ray diffraction as well as by Hartshorne's method and found that the spiral angle is a constant in the range 20–23°. De Luca and Orr,[8] on the other hand, have reported a range of values for the sprial angle (12–18°) among different varieties on the basis of a modified X-ray method.

One thing common to all earlier investigations on the true spiral angle of cotton discussed above has been the use of naturally dried, convoluted fibers for the measurement. A more direct approach would be to make measurements on fibers in which convolutions are absent. The use of mercerized fibers[9,10] would appear promising insofar as the alkali treatment renders the fibers nearly convolution-free, but other structural changes likely to accompany swelling would make the results difficult to interpret.

The alternative would be to study fibers in which convolutions are prevented from forming. It is well known that cotton fiber develops convolutions during the initial desiccation at the time of boll opening. Fiber collapse and formation of convolutions could be prevented by dehydrating the fiber by solvent exchange. Orientation measurement on such solvent-exchanged (never-dried) unconvoluted fibers as well as on their air-dried convoluted controls would give direct information about the influence of convolutions on fibrillar orientation indices. In the present study, 21 varieties of cotton, drawn from all four major species, were studied for X-ray orientation in the nature-dried (convoluted) and solvent-dried (convolution-free) states. It has thus been possible to minimize, if not altogether eliminate, the effect of convolutions on the interpretation of results.

EXPERIMENTAL

Unopened but fully mature bolls, collected on the basis of the flowering date and the average maturation period for the variety and stored in 5% aqueous formaldehyde, formed the starting materials. Fibers from a particular boll were carefully separated from the seeds while in water, and were washed thoroughly. A portion of fibers was dried in air while the rest was stored in 70% aqueous methanol and used for solvent exchange.

Solvent Exchange

This comprised exchanging the water in the fiber with methanol and then exchanging the methanol with benzene. The fibers stored in aqueous methanol were first transferred to 100% methanol. After several exchanges with dry methanol followed by dry benzene, the fibers were left exposed to air at a low humidity (ca. 35% RH) to allow evaporation of benzene. The solvent-exchanged fibers were stored at nearly the same low RH till measurements were made.

The solvent-exchanged fibers were not entirely devoid of convolutions. For practical purposes, however, the treatment was assumed to be effective if the convolutions were fewer than

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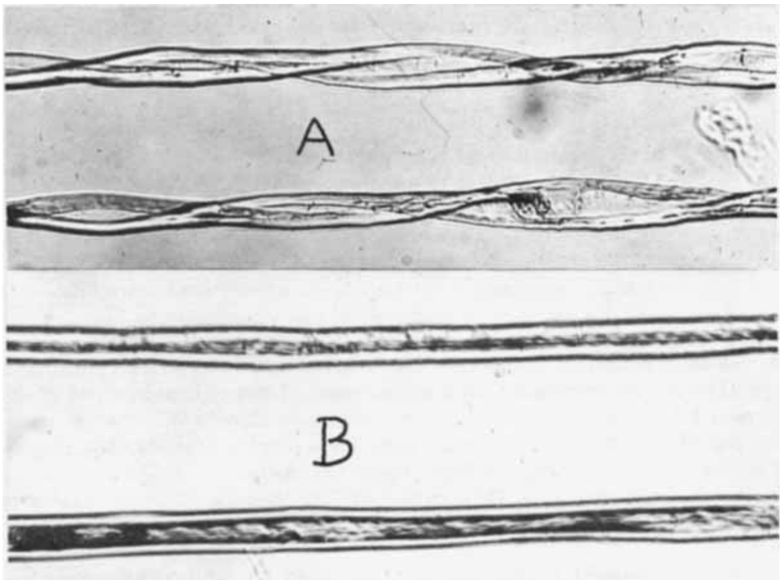

Fig. 1. Photomicrographs of air-dried (A) and solvent-exchanged (B) fibers from a particular cotton variety (IAN. 579).

1/mm. Where this number was exceeded, the treatment was repeated with fibers from a fresh boll. However, in case of a few cottons, even after repeated trials, the convolutions could not be limited to within 1/mm.

Tests

The air-dried and solvent-dried fibers were subjected to tests for convolution angle (θ) and X-ray orientation indices. The convolution angle was determined by the procedure followed

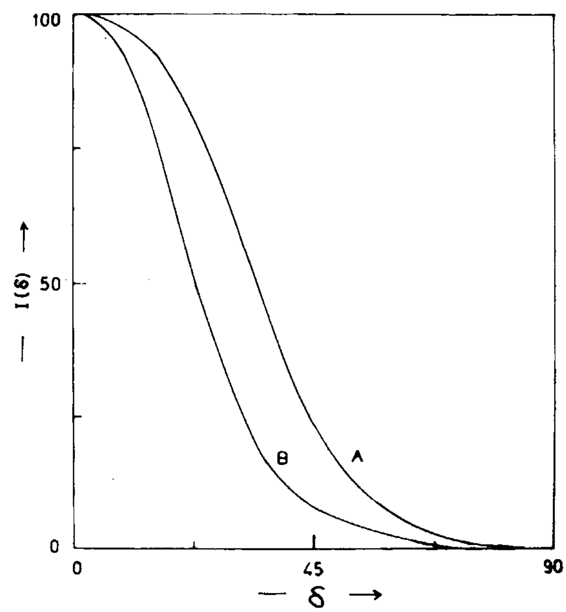

Fig. 2. Profile of X-ray intensity diffracted from (002) plane shown as a function of azimuthal angle (δ) measured from the equator for air-dried (A) and solvent-exchanged (B) fibers (variety = IAN. 579).

at CTRL.[11] The azimuthal intensity profile of the 002 diffraction are from the fiber bundle was obtained by using a Philips stabilized X-ray generator fitted with diffractometer and recording accessories. The 50% X-ray angle (ψ) and Hermans' orientation factor f_x were computed from the intensity profile.

RESULTS AND DISCUSSION

Figure 1 shows the photomicrographs of air-dried (A) and solvent-dried (B) fibers from a certain variety of cotton. Fibers in (A) show a number of convolutions typical of an air-dried sample while those in (B) are free from convolutions. Figure 2 shows the peak-normalized orientation profiles of the air-dried and solvent-dried fibers for the same sample. The absence of convolutions in the latter has considerably sharpened the orientation profile thereby reducing the 50% X-ray angle from about 35° to 23° for this variety.

Table I gives the 50% X-ray angle (ψ and ψ') and the convolution angle (θ and θ') for the air-dried as well as the solvent-dried samples. For the 21 cottons included in the tables, a high correlation ($r = 0.82$) has been found between ψ and θ for the air-dried samples, and this result is in line with the observation made by earlier workers.[12,13]

It is interesting to compare the ranges of X-ray angle in the two sets of samples (see bottom of Table I). From a value of 12.5° in the air-dried fibers, the spread of X-ray angle has come down to 5.7° in the solvent-dried samples in which the convolutions are almost completely absent. It would, therefore appear that convolutions are largely responsible for the variation in the observed fibrillar orientation index.

It is not possible to say in quantitative terms how the X-ray angle would be influenced by the few convolutions which still remain in the solvent-dried sample. The effect of convolutions could be eliminated by subtracting the convolution angle from the value of spiral angle cal-

TABLE I
50% X-Ray Angle (ψ) and Convolution Angle (θ) for the Air-Dried and Solvent-Dried Cotton Fibers

Sample no.	Variety of cotton	Air-dried (°)			Solvent-dried (°)		
		ψ	θ	ψ-θ	ψ'	θ'	ψ'-θ'
G. barbadense							
1.	Giza 7	33.0	10.65	22.35	24.8	1.33	22.47
2.	ERB.4600	30.5	6.57	23.93	23.6	1.38	22.22
3.	Suvin	27.9	7.63	20.27	23.4	1.63	21.77
4.	IBSI.25	28.0	6.97	21.03	23.8	0.90	22.90
5.	IBSI.53	24.0	7.78	16.22	21.4	1.25	20.15
G. hirsutum							
6.	IAN.579	34.5	12.35	22.65	23.2	1.41	21.79
7.	MCU.5	33.8	12.10	21.70	25.2	1.17	24.03
8.	HH.35	31.6	11.13	20.47	25.5	1.60	23.90
9.	Hybrid 4	31.5	8.68	22.82	24.8	1.33	24.47
10.	Hybrid 5	29.5	6.40	23.10	23.2	1.91	21.29
11.	G. Cot- 11	32.5	9.58	22.92	26.7	1.95	24.75
12.	G. Cot- 10	27.5	7.28	20.22	24.2	2.65	21.55
13.	IAN.4975	28.2	8.23	19.97	23.7	2.63	21.07
G. herbaceum							
14.	Suyodhar	28.5	6.47	22.03	25.1	1.60	23.50
15.	Jayadhar	28.7	7.27	21.43	23.6	1.38	22.22
16.	Sujay	23.0	4.40	18.60	21.6	1.05	20.55
17.	Digvijay	27.2	6.80	20.40	23.2	1.11	22.09
G. arboreum							
18.	Sanjay	22.0	4.70	17.30	21.0	0.70	20.30
19.	K.9	27.6	5.41	22.19	25.2	2.13	23.07
20.	AKH.4	28.2	5.50	22.70	25.5	0.83	24.67
21.	AK.235	27.5	6.47	21.03	26.2	1.28	24.92
Range		12.5	—	7.7	5.7	—	4.7

TABLE II
Hermans' Orientation Factor (f_x) for the Air-Dried and Solvent-Dried Cotton Fibers

Sample no.	Variety of cotton	Hermans' orientation factor f_x	
		Air-dried	Solvent-dried
G. barbadense			
1.	Giza 7	0.63	0.70
2.	ERB.4600	0.66	0.71
3.	Suvin	0.69	0.71
4.	IBSI.25	0.69	0.71
5.	IBSI.53	0.72	0.75
G. hirsutum			
6.	IAN.579	0.60	0.73
7.	MCU.5	0.63	0.72
8.	HH.35	0.62	0.71
9.	Hybrid 4	0.64	0.72
10.	Hybrid 5	0.69	0.73
11.	G. Cot-11	0.61	0.70
12.	G. Cot-10	0.68	0.70
13.	IAN.4975	0.67	0.75
G. herbaceum			
14.	Suyodhar	0.63	0.72
15.	Jayadhar	0.64	0.72
16.	Sujay	0.71	0.73
17.	Digvijay	0.65	0.71
G. arboreum			
18.	Sanjay	0.74	0.75
19.	K.9	0.63	0.70
20.	AKH.4	0.68	0.69
21.	AK.235	0.65	0.69
Range		0.14	0.06

culated with the help of refractive index.[2] The difference would give an estimate of the true spiral angle. Such an elimination, strictly speaking, is not possible with the present data since the orientation parameter used here is the X-ray angle. Nevertheless, since the X-ray angle ψ is closely related to the angle of spirality ϕ and since the two are also numerically close to each other, the substraction of θ from ψ would yield spiral angle measures valid at least for the sake of comparison.

Following the above reasoning, the values of $(\psi - \theta)$ and $(\psi' - \theta')$ for the air-dried and solvent-dried samples respectively have been calculated, and these values have also been included in Table I. It is clear from the data that while the initial spread in the X-ray angle is about 12.5°, it reduces to about 7.7° after subtraction of convolution angle (see ψ and $\psi - \theta$). The corresponding ranges for the solvent-dried samples are 5.7° and 4.7° (see ψ' and $\psi' - \theta$). One may expect the ranges of $\psi - \theta$ and $\psi' - \theta'$ to be equal since both the indices represent the true spiral angle unaffected by convolutions. However, the determination of θ involves errors which would increase with the increase in the frequency of convolutions and hence $\psi' - \theta'$ must be treated as more reliable than $\psi - \theta$. Thus the spread of 12.5° in the observed X-ray angle ψ of the air-dried fibers comes down to 4.7° when the effect of convolutions is totally eliminated (see $\psi' - \theta'$).

Table II gives values of Hermans' orientation factor for the air-dried and solvent-dried fibers. The f_x values lie between 0.60 and 0.74 in the case of the former set, while in the latter, they are confined to the range 0.69–0.75. The dispersion of f_x among varieties has come down from 0.14 in air-dried samples to 0.06 in the unconvoluted samples.

The narrow ranges of orientation indices in the solvent-dried samples shows that much of the differences in orientation factor found among different cotton varieties in the air-dried state is attributable to the presence of convolutions as indeed reported by Hebert et al.[13] A nearly constant X-ray angle or orientation factor for the solvent-dried samples may be taken to indicate nearly equal values of spiral angle in different varieties of cotton because, besides convolutions, only the spirality of fibrils can affect orientation measurements. Though some differences still persist, considering the errors likely in the measurements these differences could be largely ignored.

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