The Correlation between X-Ray Orientation Parameters and Strength of Fibers in Native Cotton

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SYNOPSIS

This paper reviews the relationship between tensile strength of native cotton fibers and the various orientation parameters and the true spiral angle. It is concluded that the Hermans crystallite orientation factor and the average angle of crystallite orientation, α_m, deduced from it are the best parameters to characterize fiber strength. Moreover, α_m represents a closer approximation to the true spiral angle in cotton than do the 50% or 75% X-ray angles. It is recommended that cotton breeders characterize fiber strength and screen genotypes on the basis of the Hermans factor and α_m rather than the 40, 50, or 75% X-ray angles.

INTRODUCTION

A mature cotton fiber is a flat kidney-bean-shaped ribbon with characteristic twists along its length, called convolutions.[1-3] Chemically, a cotton fiber contains 95–97% pure cellulose.[3-8] The bulk of this cellulose lays within the secondary growth walls, as crystalline microfibrils that spiral around the axis of the fiber.[6,7,9-11]

Currently, there are two structural models for cotton: (i) constant spiral angle and (ii) constant gyre length.[12-18] There is considerable confusion about the details of the helical deposition of crystalline cellulose microfibrils within the successive secondary walls of developing cotton fibers.[6-11] Spiral orientation of crystalline cellulose microfibrils in cotton is measured both by X-rays and optical techniques,[4,6,7,9-18] and it is argued that the Becke-line optical method[19,20] measures only the refractive index of the outer layers of cotton.[18,21] However, Hebert et al.[22] demonstrated that both the X-ray and optical techniques give essentially similar results and reject the misconception about the Becke-line method.

It is generally believed that the strength of cotton fibers and the measure of spiral orientation by X-rays or optical techniques are affected or distorted by the presence of convolutions and the shrinkage within individual fibers.[6,11,19,23-26] Betrabet and Iyengar[19] obtained a significant correlation between the convolution angle and strength and showed that in spite of interspecies differences, a common regression line can be fitted. Meredith[12,20] demonstrated that the spiral angle deduced from refractive indices and corrected for convolution angles is constant (ca. 21–22°) for all varieties of cotton irrespective of species. Although several authors support Meredith's statement,[13,15-17,26] others[6,18,27-35] believe that the spiral angle is not necessarily constant. Duckett and Ramey[18] have shown a rapid decrease in spiral angle in the outermost secondary layers, whereas the spiral angle of the innermost secondary layers is nearly constant. They[18] suggest also that the extent of transverse shrinkage is an essential factor in the formation of the convolution angle but has no influence on the spiral angle of the cellulose fibrils. It is generally accepted that the X-ray orientation method gives a value that is a composite of true fibrillar orientation and convolution angle.[6,11,12,15,20,36] Recently Iyer et al.[26] showed a constancy of spiral angle in never-dried cotton irrespective of species and attributed many of the dif-

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ferences in orientation factor between cotton varieties in the air-dried state to the presence of convolutions.

In spite of these reports, the values of X-ray angles uncorrected for the contribution of convolutions are significantly correlated with mechanical properties of fibers.[6,11,37] Cellulose crystallite orientation in native cotton is defined mainly in terms of the Hermans factor and 40, 50, and 75% X-ray angles, and these are correlated with some of the other fiber properties.[6,11,37] Moharir[37,38] and Moharir et al.[36-43] postulated that the Hermans factor and the average angle of crystallite orientation α_m deduced from it is the best index for varietal characterization of cotton fiber strength and is recommended to be used in cotton breeding. Although there is some theoretical justification for the use of the 50% X-ray angle as the half-width of a spectral line intensity peak in spectroscopy, the choice of 40, 75, and other X-ray angles is purely arbitrary.[36,38]

In this paper, we attempt to settle the uncertainty regarding the choice of an orientation parameter for correlating with strength of cotton fibers. This is essential because the new spinning technology (open-end spinning) implies a shift in cotton breeding from increased length to increased strength of fibers.[44,45] Characterization of fiber strength is important for cotton breeders because strength of cotton is not only heritable but also environmentally the most stable of all fiber properties.[44]

MATERIALS AND METHODS

Cotton Samples

The 24 *Gossypium hirsutum* cotton varieties listed in Table I were studied for a PhD thesis.[38] Serial (Sr.) nos. 3 and 7 were collected from the Tamilnadu State Agricultural University, Coimbatore, and Sr. nos. 5 and 17, from the Gujrat Agricultural University, Junagarh and Surat centers, respectively. The other 20 varieties were grown and collected from the Division of Genetics, Indian Agricultural Research Institute (IARI), New Delhi, India. All the varieties were grown in the crop year 1975. Most of the results and data on physical parameters and correlations between various fiber properties have been extensively reported and discussed.[38-40] Only the data on orientation parameters (Hermans factor f; α_m; 40, 50, and 75% X-ray angles), convolution angle θ, and correlations of single-fiber and bundle tenacity with orientation parameters and with the computed true spiral angles are reported in Tables I and II and are discussed below. A recent report on the true spiral angle in never-dried cotton[26] prompted us to reexamine and analyze the data on convolution angles[38] in relation to true spiral angles in cotton and, by doing so, to arrive at the best choice of an orientation parameter to characterize strength.

Convolutions

Ginned unpurified cotton fibers of the 23 (out of 24 listed in Table I) cotton varieties[38] were mounted parallel on glass slides with the help of a quick-fix adhesive and a closely ruled paper as a guide below the glass slide. The number of convolutions was counted and fiber ribbon width measured along the entire length of fibers, using a Carl-Zeiss optical microscope. The number of convolutions/cm on 200 fibers for each variety was counted and average convolution length determined. Knowing the average convolution length and ribbon width, the convolution angle was determined using Meredith's expression[46,47] given in eq. (1):

$$\text{Convolution angle } (\theta) = \arctan\left[\left(\frac{\pi}{2}\right)(\overline{D/C})\right] \quad (1)$$

where D is the ribbon width and C is the pitch of the convolution; $(\overline{D/C})$ represents the average value of (D/C). The convolution angles so obtained are reported in column 5, Table I.

Single-Fiber and Bundle Tenacity

Details of characterization techniques have been described.[38-40] Briefly, single-fiber tenacity was determined on an Instron strength tester with 1 cm gauge length, using pneumatic jaws. Average tenacity of 180 fibers of each variety was computed from the stress–strain curves. Bundle tenacity in eight replications was determined on a Preseley strength tester using nominal zero-gauge length. The single-fiber and bundle tenacity data are reported in columns 7 and 8 of Table I, respectively.

X-Ray Orientation

All X-ray diffractograms were recorded from purified cotton fibers. Details of the purification procedure for removal of waxes, pectic materials, and protoplasmic residues have been described.[36,38-40] Flat-plate X-ray diffractograms from bundles of well-parallelized fibers were recorded on a Philips X-ray diffractometer. A Joyce–Loeble microdensitometer was used for mapping radial intensity at each 5°

Table I Data on Various Orientation Parameters, Convolution Angle, and Single-Fiber and Bundle Tenacity of Cotton Fibers Studied

Sr. No.	*G. hirsutum* Variety	Hermans Factor f	Avg. Angle of Crystallite Orientation α_m (°)	X-Ray Angles (°) 40% (A)	50% (B)	75% (C)	Convolution Angle (°) θ	Different Measures of True Spiral Angle (°) α_m—θ	A—θ	B—θ	Single-Fiber Tenacity (N/tex)	Bundle Tenacity (N/tex)
	1	2	3	4a	4b	4c	5	6a	6b	6c	7	8
1	B-1007	0.592	31.4	38.7	34.0	22.5	13.6	1.78	25.1	20.4	0.22(L)	0.47
2	H-14	0.598	31.1	35.5	31.5	20.0	13.8	17.3	21.7	17.7	0.24	0.48
3	Laxmi	0.606	30.8	34.7	29.5	19.5	10.9	19.9	23.8	18.6	0.23	0.47
4	D-33	0.576	32.1	43.0	36.5	23.5	14.7	17.4	28.3	21.8	0.25	0.50
5	Deviraj	0.542	33.5	46.0(H)	41.5	27.5	12.1	21.4(H)	33.9(H)	29.4(H)	0.27	0.44(L)
6	Acala 4-42	0.591	31.4	37.5	33.0	21.5	—	—	—	—	0.21	0.49
7	MCU-8	0.580	31.9	30.0	26.0	18.0	13.8	18.1	16.2	12.2	0.28	0.48
8	Reba B-50	0.711	26.0	27.0	24.0	16.0	14.1	11.9(L)	12.9	9.9	0.31	0.53(H)
9	SV-213	0.621	30.1	30.5	27.0	19.0	13.6	16.5	16.9	13.4	0.24	0.48
10	K-2421	0.607	30.8	32.5	29.0	20.0	14.7	16.1	17.8	14.3	0.28	0.47
11	Cl_{20}	0.618	30.2	28.5	24.5	15.5	16.0	14.2	12.5	8.5	0.25	0.48
12	Am. Nectariless	0.640	29.3	31.5	27.0	18.5	11.6	17.7	19.9	15.4	0.26	0.49
13	Lankart-57	0.637	29.4	32.0	28.0	21.0	15.8	13.6	16.2	12.2	0.28	0.49
14	D-40	0.670	27.9	31.0	27.0	18.0	13.8	14.1	17.2	13.2	0.29	0.49
15	Bikaneri Narma	0.628	29.8	28.0	24.0	17.0	14.7	15.1	13.3	9.3	0.30	0.49
16	LH-299	0.604	30.9	24.5(L)	21.5(L)	14.5	12.8	18.1	11.7	8.7	0.23	0.47
17	SRT-1 G Cot. 100	0.673	27.8	27.0	23.5	16.0	15.8	12.0	11.2(L)	7.7(L)	0.28	0.48
18	Am. Nect. X SV-213	0.568	32.5	35.5	31.0	21.0	13.7	18.8	11.8	17.3	0.26	0.46
19	SV-213 X Am. Nect.	0.612	30.5	30.5	27.0	17.5	15.3	15.2	15.2	11.7	0.30	0.49
20	Cl_{20} X B-1007	0.628	29.8	33.0	28.0	19.0	9.5(L)	20.3	13.5	18.5	0.30	0.49
21	Acala 4-42 X Lankart-57	0.663	28.2	29.0	25.0	15.0	14.4	13.8	14.6	10.6	0.31	0.51
22	Reba B-50 X Cl_{20}	0.597	31.2	32.0	27.0	17.5	13.6	17.6	18.4	13.4	0.27	0.48
23	B-1007 X Lankart-57	0.664	28.2	28.0	25.0	16.5	15.2	13.0	12.8	9.8	0.31	0.48
24	K-2421 X SV-213	0.645	29.1	30.5	27.0	18.5	9.5(L)	19.6	21.0	17.5	0.34(H)	0.50
25	Range of values Max. − Min.	0.169	7.5	21.5	15.0	12.5	6.5	9.5	22.7	21.7	0.12	0.09

(H) highest value within varieties; (L) lowest value within varieties.

Table II Correlation Coefficients and Probability Levels between Different Orientation Measures with Single-Fiber and Bundle Tenacity of Cotton Fibers

Sr No.	Orientation Parameter	Single-Fiber Tenacity (1 cm Gauge Length)	Bundle-Fiber Tenacity (Nominal Zero Gauge Length)
1	Hermans factor f	$r = .552$	$r = .712$
		$P = .01$	$P = .001$
2	Average angle of crystallite orientation (σ_m)	$r = -.568$	$r = -.727$
		$P = .005$	$P = .001$
3	40% X-ray angle (A)	$r = -.324$	$r = -.413$
		$P = .20$	$P = .05$
4	50% X-ray angle (B)	$r = -.349$	$r = -.442$
		$P = .10$	$P = .05$
5	75% X-ray angle (C)	$r = -.342$	$r = -.476$
		$P = .01$	$P = .02$
6	(α_m—θ)	$r = -.318$	$r = -.534$
		$P = .139$	$P = .008$
7	(A—θ)	$r = -.315$	$r = -.337$
		$P = .143$	$P = .115$
8	(B—θ)	$r = -.259$	$r = -.440$
		$P = .231$	$P = .035$
Correlation coefficients for 20 IARI varieties			
9	(α_m—θ)	$r = -.282$	$r = -.434$
		$P = .242$	$P = .06$
10	(A—θ)	$r = .309$	$r = .003$
		$P = .197$	$P = .891$
11	(B—θ)	$r = -.254$	$r = -.155$
		$P = .292$	$P = .525$

angle for the equatorial (002) and (101, 101) reflections combined. From the series of azimuthal intensity curves, the scattered intensity distribution was computed as a function of the angular distance from the equator. The curves were normalized to equal peak heights for all the varieties. From the normalized curves for (002) reflections, the values for 40, 50, and 75% X-ray angles were measured and are reported in columns 4a, b, and c of Table I.

The Hermans crystallite orientation factor f is defined as

$$f = 1 - \frac{3}{2}\langle \sin^2 \alpha \rangle \qquad (2)$$

where α is the angle made by the molecular chain in the crystallite with the fiber axis and $\langle \sin^2 \alpha \rangle$ is the average value of $\sin^2 \alpha$. If α_{hkl} is the angle between (hkl) and the equator, then, according to Hermans,[41]

$$\langle \sin^2 \alpha \rangle = \langle \sin^2 \alpha_{002} \rangle + \langle \sin^2 \alpha_{101,10\bar{1}} \rangle \qquad (3)$$

The average values of the distribution factor on the right-hand side of eq. (3) were determined from the azimuthal intensity scans of (002) and (101, $10\bar{1}$) combined reflections and using the following relationship and graphical integration procedure described by Hermans[5]:

$$\langle \sin^2 \alpha_{hkl} \rangle = \frac{\int_0^{\pi/2} I(\alpha_{hkl}) \cdot \sin^2 \alpha_{hkl} \cdot \cos \alpha_{hkl} \cdot d\alpha_{hkl}}{\int_0^{\pi/2} I(\alpha_{hkl}) \cdot \cos \alpha_{hkl} \cdot d\alpha_{hkl}} \qquad (4)$$

Having calculated this function, the Hermans crystallite orientation factors were determined using eq. (2). Also from the values of $\langle \sin^2 \alpha_{hkl} \rangle$, the average value of α, designated as α_m in the text, was calculated. The computed data on the Hermans factor and the average angle of crystallite orientation α_m are given in columns 2 and 3 of Table I, respectively.

Experimental and Data Analysis

Meredith[12,20] eliminated the effect of convolutions by substracting the convolution angle from the value of spiral angle calculated with the help of the refractive index. The difference gave the measure of the true spiral angle in cotton. Strictly speaking, such elimination is not possible with the data presented (Table I). However, since the X-ray angle is closely related to the angle of spirality ϕ and the values of the two are numerically close to each other, in general, the substraction of the convolution angle from the X-ray angle would yield a close measure of the true spiral angle. The true spiral angles in never-dried cotton have also been calculated in this way previously.[26]

Both 40 and 50% X-ray angles and α_m have been widely used as measures of spirality[6,11,37] to correlate with fiber properties, but with varying success. Therefore, following the above reasoning,[26] at least three different close measures of true spiral angle can be obtained by substracting the values of the convolution angle from the values of α_m and 40 and 50% X-ray angles. The close values of true spiral angles so obtained for 23 of the 24 cotton varieties are presented in columns 6a, b, and c of Table I, respectively.

RESULTS AND DISCUSSION

It is evident from Table I, Sr. no. 25, that, corresponding to a range of 0.169 for the Hermans factor (column 2), the range (maximum value minus minimum value) for the X-ray angles (columns 4a, b, and c) progressively decreases from 21.5° for the 40% angle, 15.0° for the 50% angle, to 12.5° for the 75% X-ray angle, respectively. This range is only 7.5° in the values of α_m (column 3). The convolution angles (θ) (column 5) vary within a range of 6.5°. Likewise, the range in the computed true spiral angles (columns 6b and c) from 40 and 50% X-ray angles is considerably higher than the range in true spiral angles deduced from the average angle of crystallite orientation α_m (column 6a).

Single-fiber and bundle tenacity (Sr. no. 25, columns 7 and 8) values range between 0.21–0.34 and 0.44–0.53 N/tex, respectively, for the 24 varieties studied.

Table II clearly demonstrates that both single-fiber and bundle tenacity values show the best correlations with the Hermans factor f and α_m. Although the correlations of f and α_m with both single-fiber and bundle tenacity are significant, the values for bundle tenacity are considerably higher. Both 40 and 50% X-ray angles correlate significantly better with bundle tenacity than with single-fiber tenacity, and the values of the correlation coefficient for the 50% X-ray angle are marginally higher than those with 40% X-ray angles. Seventy-five percent X-ray angles have been shown to be very close approximations of the true spiral angle in cotton,[48] and the correlation coefficients between 75% X-ray angles and both the single-fiber and bundle tenacity are more significant than they are between the 40 and 50% X-ray angles.

Comparing the correlations of the three different measures of true spiral angle in cotton, it is obvious that the true spiral angle deduced from the α_m correlates best with both the single-fiber and bundle tenacity values. The correlation of the true spiral angle deduced from α_m with bundle tenacity is significantly better with greater probability than those obtained with 75% X-ray angles. Further, the true spiral angle deduced from the 50% X-ray angles are correlated better with bundle tenacity than is the true spiral angle deduced from the 40% X-ray angles.

Environmental conditions considerably influence fiber properties.[1–3,49,50] Correlation coefficients for 20 cotton varieties, grown under identical agroclimatic conditions on the same farm at IARI, in the same crop year, were therefore computed separately (see Table II, Sr. nos. 9–11). It was expected that the correlations would either improve or at least exhibit faithfully the relationship between the true spiral angle and the tenacity of fibers. Only the true spiral angle deduced from α_m correlated significantly with bundle tenacity, but not with single-fiber tenacity, whereas the correlations of true spiral angles deduced from 40 and 50% X-ray angles with single-fiber and bundle tenacity are erratic and insignificant.

Determination of single-fiber tenacity in cotton is very tedious and time-consuming as compared to the determination of bundle tenacity. Therefore, the bundle tenacity of fibers is considerably more important for all practical purposes. All correlations between bundle tenacity and various orientation

parameters listed in Table II for cotton varieties within a genetic species and also for varieties grown under identical agroclimatic conditions are statistically significant although the probability levels differ. But the correlations between bundle tenacity and f and α_m and the true spiral angle deduced from α_m are uniformly more significant than for the other orientation parameters.

Therefore, the Hermans crystallite orientation factor f and the average angle of crystallite orientation α_m are the best parameters to characterize fiber strength. Moreover, α_m measures the average spirality in cotton more faithfully than do any other X-ray orientation angles.

Differences between opinions on the "constant spiral angle" and the "varying spiral angle" in cotton are not yet settled satisfactorily. However, it is certain that, even if the spiral angle varies from one variety to another, this variation cannot be very wide. From Table I, Sr. no. 25, it can be seen that the values of the true spiral angle deduced from 40 and 50% X-ray angles show very wide variations (22.7° and 21.7°, respectively) and that the true spiral angles deduced from α_m vary within just 9.5°. This range is comparable to the range of values of true spiral angles of 12.5° for air-dried cotton[26]: 7.9° and 7.5° from X-ray and optical angles, respectively, in cotton of the fourth picking, as reported earlier.[18,48]

Several workers have pointed out[6,7,11,19,37,51-53] that a lower spiral angle corresponds to increased orientation and, consequently, to higher tenacity. It is clear that the true spiral angle deduced from α_m, which correlates best with both single-fiber and bundle tenacity values, thus represents a better measure of spirality than do the 40, 50, or 75% X-ray angles. The lowest value of true spiral angle deduced from α_m corresponds also to the highest value of bundle tenacity (see Sr. no. 8, columns 6a and 8 of Table I). This relationship is not faithfully represented by the lowest values of true spiral angles deduced from 40 and 50% X-ray angles (see Sr. no. 17, columns 6b and c and 8 of Table I).

CONCLUSION

It is concluded that the Hermans crystallite orientation factor f and the average angle of crystallite orientation α_m deduced from it are the best parameters to characterize cotton fiber strength. Also, α_m is the best measure of average spirality for computing the true spiral angle in cotton. It is recommended that characterization of cotton for strength for hybridization should only be done on the basis of the Hermans factor and α_m and not on the basis of 40 or 50% X-ray angles. Incidently, the present data demonstrate that the spiral angle within different varieties of cotton is not constant.

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