Stability of X-ray Cellulose Crystallite Orientation Parameters in Native Cotton with Change of Location and Year of Growth

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ABSTRACT: In this article, data on cellulose crystallite orientation parameters measured in terms of the Hermans orientation factor, average angle of orientation (α_m), and 40, 50, and 75% X-ray angles in respect to the same 13 cotton cultivars grown at different agroclimatic locations and in different crop years in India are presented and discussed. It was observed that whereas the average values of the X-ray orientation parameters are different for different varieties they remain practically invariant within individual varieties with change of the location of growth. The orientation parameters, therefore, appear to be genetic in origin and independent of the agroclimatic conditions of growth. It is believed that these results can be suitably exploited by cotton breeders in evolving varieties with an increased strength of fibers. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 72: 269–276, 1999

Key words: cotton; cellulose; crystallite; orientation; location

INTRODUCTION

A cotton fiber is a tubular outgrowth of a single cell on the epidermis of the seed.[1–4] Chemically, cotton fiber constitutes about 94% pure cellulose and this cellulose is almost wholly crystalline. The degree of polymerization (DP) for native cotton cellulose has been estimated to the approximately 3000–5000[5–8] by some and as high as 5000–13,000 by others.[9–13] The molecular chains of cellulose aggregate in an extended and nonfolded[14] form into elementary fibrils, which, in turn, combine to form a microfibril. The fibrils are believed to be 4–30 nm wide,[5,14–19] although smaller fibrils of about 2 nm have also been reported.[20,21] The evidence in this respect is almost entirely electron microscopic.

However, it is stated that the lattice coherence along the elementary fibrils is intercepted at regularly spaced intervals with an average distance of about 50 nm, so that the fibrils contain a sequence of slightly mismatched crystal blocks with the same axial orientation of the cellulose chains but differing from each other in the orientation of the a- and the c-axes. On the basis of X-ray diffraction studies,[22,23] the unit cell of cellulose-I has been worked out to be monoclinic with a, b, and c dimensions of a = 8.35 Å, b = 10.30 Å, c = 7.9 Å, and b = 84°. It is generally believed that although one-third of the total molecules constitute the amorphous phase cotton is essentially crystalline and the disorder is due mainly to the fact that

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small crystalline units are imperfectly packed together. The structure of cotton cellulose is therefore considered to be paracrystalline.[24–29] The orientation of the cellulose crystallites with respect to the fiber axis is known to determine the intercotton differences and most of the technologically important properties of the fiber.[5,30,31] Both optical and X-ray methods are used to determine orientation.[23,32–34] Since the optical methods are generally very tedious and require a large amount of data for a representative value of the fiber, X-ray methods are generally favored in orientation studies.

The most widely accepted parameters for characterizing the orientation of the crystallites with respect to the fiber axis is the Hermans crystallite orientation factor[35] and this factor has been shown to correlate with important fiber properties, particularly the strength of the fiber.[35–38] Moharir et al.[20,39–43] in a series of publications identified the Hermans crystallite orientation factor and the average angle of orientation (α_m) derived from it to be the best indices for the characterization of fibers for strength both within varieties of individual species and within a mixture of varieties of different species taken together, as compared to the 40, 50, and 75% X-ray angles. Moharir et al.[30,39,40] also proposed to use the Hermans factor for screening genotypes in a cotton breeding program for evolving hybrids with an increased inherent strength of the fibers as demanded by the efficient, high-speed modern textile processing machinery.[44]

In this article, data on the Hermans orientation factor, the average angle of orientation (α_m), and 40, 50, and 75% X-ray angles in respect to the same 13 cotton varieties grown at different agroclimatic locations and in different crop years in India are presented and discussed. Also discussed are the correlations of these orientation parameters among themselves and with the bundle tenacity of the fibers.

EXPERIMENTAL

The 13 cotton varieties belonging to all the four commercial species of cotton, namely, *Gossypium arboreum, Gossypium herbaceum, Gossypium hirsutum,* and *Gossypium barbadense,* were grown at four locations, namely, Sirsa, New Delhi, Nagpur, and Coimbatore, in India during the 1992, 1994, and 1995 crop seasons. Mature seed cotton fibers were harvested, ginned, and purified in the laboratory for removal of waxes, pectic materials, and protoplasmic residues by soaking the fibers for 6 h each in methanol and carbon tetrachloride and subsequent boiling of the fibers for 3 h in a 2% sodium hydroxide solution. The fibers were neutralized for 1 h in 0.1*N* HCl and washed free of acid with double-distilled water and dried at room temperature.[20,39] The purified fibers were combed and made into bundles of well-parallelized fibers. The bundle was mounted on an X-ray diffractometer holder and scanned in a transmission mode on a Philips Model PW-1720 X-ray generator, equipped with a chart recorder and a microprocessor controller. X-ray diffractograms were recorded at a 35 kV voltage at 20 mA current, using nickel-filtered CuKα radiation of wavelength of 1.5418 Å. The orientation scans were obtained by keeping the glancing angle fixed (viz., 22.6° for the 002 plane, 16.2° for the $10\bar{1}$ plane, and 14.8 for the 101 plane) and rotating the sample through 360° in a plane perpendicular to the radiation direction. The horizontal scale and the peak and rotation angles were kept common for all samples. The azimuthal scans of the 002 plane were normalized to equal curve heights, and from these, the values of 40, 50, and 75% X-ray angles were read. Likewise, the scans of the 101 and $10\bar{1}$ planes were also normalized, and using the graphical integration procedure due to Hermans,[35] the values of Hermans crystallite orientation factors and the average angle of orientation (α_m) were determined from the equations

$$f = 1 - \frac{3}{2}\,(\overline{\sin^2 \alpha_m})$$

where

$$\overline{\sin^2 \alpha_{hkl}} = \frac{\int_0^{\pi/2} I \sin^2 \alpha_{hkl} \cos \alpha_{hkl}\, d\alpha_{hkl}}{\int_0^{\pi/2} I \cos \alpha_{hkl}\, d\alpha_{hkl}}$$

Likewise, X-ray diffraction patterns were also separately recorded from well-parallelized bundles of fibers of cotton varieties and degummed-purified ramie fibers on a Siemens D-500 X-ray diffractometer using CuKα line at 35 kV, 15 mA, and scanning speed of 0.02°/s, in the 2θ range of 10°–40°. The 002, 101, and $10\bar{1}$ diffraction peaks were resolved by the FIT XDR data analysis software for a normalized area, and considering the area under the 002 peak for ramie fibers to be 100% crystalline, the area under the 002 peaks for all cottons were compared. The percent relative crystallinity with respect to ramie for all cotton varieties was thus computed. The data on the locationwise average values of bundle tenacity, crystallinity, and orientation parameters for all crop years and the number of replicate samples for the cotton varieties are given in Table I, columns 1–7. In Table II are given the correlation coefficients of the Hermans factor, α_m, and bundle tenacity with other orientation parameters.

RESULTS AND DISCUSSION

It can be observed from Table I, column 3, that the average values of bundle fiber tenacity within individual varieties vary marginally with the location. Likewise, the values of the Hermans crystallite orientation factor and the average angle of orientation (α_m), columns 5 and 6, Table I, also vary in a narrow range within individual varieties grown at different locations. The values of the relative crystallinity with respect to ramie in column 4, Table I, also show variation within individual varieties with the location of growth. However, in the variation of these parameters, there does not appear to be a distinct pattern of variation with a location, despite the fact that the locations from Coimbatore to Sirsa are spread between 11° to 29° north latitudes in India (Table III). Further, it may be mentioned that there are minor variations in agronomic practices for the cultivation of cotton at these locations, besides major variations of climate and soil types. This discussion can be seen in sharper focus from the data on locationwise average values of the Hermans factor and α_m for individual cotton cultivars, summarized in Table I. It may be observed that within individual varieties the average values of both the Hermans factor and α_m do not change drastically with the location and latitude of the location of growth, as is evident from the lower values of the standard deviations within individual varieties (sample nos. 7, 14, 21, 28, 35, 42, 48, 55, 62, 68, 74, 79, and 84, Table I). However, the average values of both these parameters do indeed vary from one variety to the other and this variation is more significant.

From the data on the average values of the 40, 50, and 75% X-ray angles given in columns 7a, b, and c of Table I for individual locations for each variety, it is observed that there is no definite pattern in the variation of the X-ray angle values with the location of growth. In fact, in many cases, the values of the 40 and 50% X-ray angles for distant Coimbatore and Sirsa or Nagpur locations are nearly or almost the same. The values of the 75% X-ray angles, however, show some deviations with the location of growth. These observations bring home the fact that X-ray angles may also be genetic in origin, in principle, and the reasons for variation in the values of the X-ray angles with the location of growth may possibly be sought in the rate and amount of cellulose synthesis which varies within an individual variety with the location of growth of cotton and, consequently, with environmental conditions of growth as evidenced by the variation in the maturity of cotton with latitude of the place of growth.[45]

It may also be pertinent to mention here that genetic inheritance of the Hermans orientation factor and X-ray angles had been seen earlier by Moharir et al.[46] in the F1 hybrids of *G. barbadense* and *G. hirsutum* parents. From the values of the correlation coefficients and probability values, given in Table II, it may be observed that both the 40 and 50% X-ray angles correlate significantly with the Hermans factor although the values of the correlation with the 50% X-ray angle is slightly better. Likewise, the correlations of the 40 and 50% X-ray angles are equally significant with the average angle of orientation (α_m). However, bundle fiber tenacity (Table I, column 3) correlates best with the 40% X-ray angle than with the 50% X-ray angle (Table II). A relative orientation/crystallinity index with respect to ramie does not yield any significant correlation with the Hermans factor, α_m, and bundle tenacity. The correlations of the Hermans factor and α_m are far better with the true spiral angle (α_m-θ)[42,43] than with the other two measures of true spiral angles (40%-θ) and (50%-θ).[42,43] The correlations of α_m with the 40 and 50% X-ray angles are again equally significant and so also with the true spiral angles (40%-θ) and (α_m-θ). This indicates that α_m and the true spiral angle (α_m-θ) determine the spirality of cellulose crystallites in native cotton more faithfully than do the 40 and 50% X-ray angles and the true spiral angles deduced from them.[42,43]

In conclusion, it may be said that the Hermans crystallite orientation factor for an individual cotton variety does not drastically vary with year and the location of growth of cotton. However,

Table I Locationwise Average Values of Bundle Tenacity, Relative Crystallinity, and Crystallite Orientation Parameters for All Crop Years

Sample No.	Cotton Variety	Location of Growth	Bundle Tenacity (gf/tex)	Relative Crystallinity with Respect to Ramie	Hermans Orientation Factor	Av Angle of Orientation α_m (°)	X-ray Angles (°) 40%	X-ray Angles (°) 50%	X-ray Angles (°) 75%
	1	2	3	4	5	6	7(a)	7(b)	7(c)
1	AKH-4	Sirsa	39.94	0.309	0.4662(L)	36.60(H)	29.50(H)	25.62(H)	15.75
2	AKH-4	New Delhi	49.84(H)	0.301(L)	0.4876	35.80	25.00(L)	22.00(L)	13.00(L)
3	AKH-4	Nagpur	33.85(L)	0.352(H)	0.4832	35.92	29.00	25.00	16.50(H)
4	AKH-4	Coimbatore	36.52	0.319	0.5022(H)	35.18(L)	28.20	24.33	16.00
5	Av within variety for all locations and crop years		40.05	0.320	0.4856	35.87	27.92	24.23	15.31
6	Range of variation (H–L)[a]		15.99	0.051	0.036	1.42	0.50	3.62	3.50
7	Standard deviation		6.99	0.022	0.0148	0.58	2.02	1.58	1.57
8	AC-738	Sirsa	43.95(H)	0.358(H)	0.4771(H)	36.20(L)	28.00(L)	25.00	15.00(L)
9	AC-738	New Delhi	42.61	0.327(L)	0.4690	36.45	29.00	25.50	16.00
10	AC-738	Nagpur	40.99(L)	0.357	0.4607(L)	36.82(H)	28.75	24.25(L)	15.25
11	AC-738	Coimbatore	43.06	—	0.4611	36.80	31.00(H)	27.00(H)	18.00(H)
12	Av within variety for all locations and crop years		42.65	0.347	0.4669	36.56	29.18	25.43	16.06
13	Range of variation (H–L)		2.96	0.031	0.0164	0.62	3.00	2.75	3.00
14	Standard deviation		1.24	0.017	0.0007	0.308	1.28	1.16	1.36
15	B.N.	Sirsa	39.39(L)	0.323(L)	0.4618(L)	36.80	29.25(H)	26.25(H)	18.25(H)
16	B.N.	New Delhi	49.31(H)	0.356	0.4961(H)	35.45(L)	26.50(L)	22.00(L)	12.50
17	B.N.	Nagpur	47.73	0.360	0.4765	35.84(H)	28.00	24.40	12.80
18	B.N.	Coimbatore	43.82	0.393(H)	0.4864	35.82	28.83	24.66	15.25
19	Av within variety for all locations and crop years		45.06	0.358	0.4802	35.98	28.14	24.32	14.70
20	Range of variation (H–L)		9.92	0.070	0.0343	0.39	2.75	4.25	5.75
21	Standard deviation		4.43	0.028	0.0140	0.58	1.21	1.75	2.66
22	Y-1	Sirsa	44.27	0.365	0.5151	34.70	25.00	21.50	13.00
23	Y-1	New Delhi	40.56(L)	0.270(L)	0.5187	34.50	25.00(L)	21.00(L)	13.00
24	Y-1	Nagpur	47.36(H)	0.389(H)	0.5007	35.22(H)	26.87(H)	24.37(H)	14.75(H)
25	Y-1	Coimbatore	44.75	—	0.5246(H)	34.30(L)	25.00	21.50	12.50(L)
26	Av within variety for all locations and crop years		44.23	0.341	0.5147	34.68	25.46	22.09	13.31
27	Range of variation (H–L)		6.80	0.119	0.0239	0.92	1.87	3.37	2.25
28	Standard deviation		2.80	0.063	0.0101	0.39	0.93	1.53	0.98
29	Maljari	Sirsa	37.07(L)	0.318(L)	0.4735	36.31	29.50(H)	25.87(H)	16.62(H)

Table I *Continued*

Sample No.	Cotton Variety	Location of Growth	Bundle Tenacity (gf/tex)	Relative Crystallinity with Respect to Ramie	Hermans Orientation Factor	Av Angle of Orientation α_m (°)	X-ray Angles (°) 40%	X-ray Angles (°) 50%	X-ray Angles (°) 75%
	1	2	3	4	5	6	7(a)	7(b)	7(c)
30	Maljari	New Delhi	45.20(H)	0.347	0.4699(L)	36.50(H)	20.00(L)	19.00(L)	10.00(L)
31	Maljari	Nagpur	43.76	0.427(H)	0.5014	35.20(L)	26.12	22.25	13.50
32	Maljari	Coimbatore	42.40	0.407	0.5224(H)	35.69	27.45	22.83	14.08
33	Av within variety for all locations and crop years		42.10	0.374	0.4918	35.92	25.76	22.48	13.55
34	Range of variation (H–L)		8.13	0.109	0.0525	1.30	9.50	6.87	6.62
35	Standard deviation		3.54	0.050	0.024	0.59	4.08	2.81	2.72
36	AKA-5	Sirsa	40.55	0.273(L)	0.4909	35.62	30.25(H)	26.75(H)	13.73(H)
37	AKA-5	New Delhi	45.38(H)	0.409(H)	0.5160(H)	34.60(L)	23.00(L)	20.00(L)	12.00(L)
38	AKA-5	Nagpur	45.31	0.315	0.4758(L)	36.20	26.00	22.33	14.00
39	AKA-5	Coimbatore	37.63(L)	0.348	0.4825	36.45(H)	28.16	24.00	15.08
40	Av within variety for all locations and crop years		42.21	0.336	0.4848	36.00	26.85	23.27	13.70
41	Range of variation (H–L)		7.75	0.136	0.0402	1.85	7.25	6.75	5.37
42	Standard deviation		3.80	0.049	0.017	0.82	3.09	2.84	1.27
43	LH-900	Sirsa	40.02(L)	0.472(H)	0.5338(H)	33.83(L)	27.00	22.75(L)	14.25(L)
44	LH-900	Nagpur	44.64(H)	0.381(L)	0.4817(L)	35.98(H)	27.60(H)	23.40(H)	14.40
45	LH-900	Coimbatore	42.39	0.389	0.5029	35.14	26.25(L)	22.82	14.75(H)
46	Av within variety for all locations and crop years		42.35	0.414	0.4979	35.31	26.95	22.99	14.46
47	Range of variation (H–L)		4.62	0.091	0.0521	2.15	1.35	0.65	0.50
48	Standard deviation		1.88	0.041	0.0218	0.89	0.55	0.35	0.25
49	LRA-5166	Sirsa	39.40(L)	0.313(L)	0.5625(H)	32.64(L)	28.75(L)	24.87	16.87
50	LRA-5166	New Delhi	44.84	0.365(H)	0.4900	35.70	27.50(L)	24.50(L)	15.00(L)
51	LRA-5166	Nagpur	47.21(H)	0.316	0.5356	34.65	28.00	24.50	15.75
52	LRA-5166	Coimbatore	40.31	—	0.4845(L)	35.88(H)	28.75	25.29	17.04
53	Av within variety for all locations and crop years		42.94	0.331	0.5183	34.81	28.25	24.79	16.16
54	Range of variation (H–L)		7.81	0.052	0.078	3.24	1.25	0.79	2.04
55	Standard deviation		3.71	0.029	0.0374	1.48	0.61	0.37	0.83
56	SRT-1 G.Cot. 10	Sirsa	39.35	—	0.4487	37.31	28.50	24.75	15.75
57	SRT-1 G.Cot. 10	New Delhi	47.88	0.384	0.4807(H)	36.00(L)	26.00(L)	23.00(L)	14.50(L)
58	SRT-1 G.Cot. 10	Nagpur	49.92(H)	0.377(L)	0.4414(L)	37.57(H)	29.50(H)	25.75(H)	16.75(H)
59	SRT-1 G.Cot. 10	Coimbatore	35.64(L)	0.404(H)	0.4733	36.33	29.25	25.50	15.75

Table I *Continued*

Sample No.	Cotton Variety	Location of Growth	Bundle Tenacity (gf/tex)	Relative Crystallinity with Respect to Ramie	Hermans Orientation Factor	Av Angle of Orientation α_m (°)	X-ray Angles (°) 40%	X-ray Angles (°) 50%	X-ray Angles (°) 75%
	1	2	3	4	5	6	7(a)	7(b)	7(c)
60	Av within variety for all locations and crop years		43.19	0.388	0.4561	36.99	28.31	24.75	15.68
61	Range of variation (H–L)		14.28	0.027	0.0393	1.57	3.50	2.75	2.25
62	Standard deviation		6.80	0.011	0.0189	0.75	1.59	1.24	0.92
63	Suvin	New Delhi	49.67	—	0.5197	34.50	24.00(L)	20.00(L)	12.00(L)
64	Suvin	Nagpur	45.34(L)	0.357(L)	0.5425(H)	33.50(L)	25.00(H)	22.00(H)	15.00(H)
65	Suvin	Coimbatore	53.59(H)	0.375(H)	0.4827(L)	35.95(H)	25.00	21.25	13.00
66	Av within variety for all locations and crop years		49.53	0.366	0.5149	34.97	24.66	21.08	13.33
67	Range of variation (H–L)		8.25	0.018	0.0598	2.45	1.00	2.00	3.00
68	Standard deviation		3.36	0.009	0.0300	1.23	0.57	1.01	1.52
69	Jyoti	Sirsa	—	0.417(L)	0.4866	35.80	27.00	23.00	14.00
70	Jyoti	Nagpur	—	0.422	0.4858(L)	35.80(H)	27.00(H)	23.00(H)	15.00(H)
71	Jyoti	Coimbatore	43.41	0.437(H)	0.5110(H)	34.80(L)	25.50(L)	22.50(L)	13.50(L)
72	Av within variety for all locations and crop years		43.41	0.425	0.4944	35.46	26.50	22.83	14.16
73	Range of variation (H–L)		—	0.020	0.0252	1.00	2.50	1.50	2.50
74	Standard deviation		—	0.010	0.0143	0.57	0.86	0.28	0.76
75	G.Cot. 13	Dhandhuka	42.16(H)	—	0.4866(L)	35.80(H)	27.00(L)	23.00(L)	14.00(L)
76	G.Cot. 13	Chharodi	40.89(L)	—	0.5005(H)	35.20(L)	28.00(H)	24.00(H)	15.00(H)
77	Av within variety for all locations and crop years		41.52	—	0.4912	35.60	27.50	23.50	14.50
78	Range of variation (H–L)		1.27	—	0.0139	0.60	1.00	1.00	1.00
79	Standard deviation		0.89	—	0.0007	0.42	0.70	0.70	0.70
80	G.Cot. 100	Nagpur	42.80	0.344	0.5072(H)	35.00(L)	29.00(H)	25.00(H)	16.00(H)
81	G. Cot. 100	Coimbatore	—	—	0.4970(L)	35.35(H)	27.00(L)	23.00(L)	14.50(L)
82	Av within variety for all locations and crop years		42.80	0.344	0.5004	35.23	28.00	24.00	15.25
83	Range of variation (H–L)		—	—	0.0102	0.35	2.00	2.00	2.25
84	Standard deviation		—	—	0.0009	0.25	1.41	1.41	1.06

[a] H: highest value within variety; L: lowest value within variety.

Table II Correlations of Hermans Factor, Average Angle of Orientation α_m, and Bundle Fiber Tenacity with Other Orientation Parameters

Parameter	Hermans Factor	Average Angle of Orientation α_m	Bundle Fiber Tenacity
α_m	$\gamma = -0.911$ $P > 0.001$	1.00	$\gamma = -0.280$ $P > 0.01$
40% X-ray angle	$\gamma = -0.283$ $P > 0.001$	$\gamma = 0.456$ $P > 0.001$	$\gamma = -0.421$ $P > 0.001$
50% X-ray angle	$\gamma = -0.340$ $P > 0.001$	$\gamma = 0.409$ $P > 0.001$	$\gamma = -0.320$ $P > 0.01$
75% X-ray angle	$\gamma = -0.286$ $P > 0.01$	$\gamma = 0.268$ $P > 0.01$	$\gamma = -0.151$ N.S.
ROI[a] with respect to ramie	$\gamma = 0.158$ N.S.	$\gamma = -0.225$ N.S.	$\gamma = 0.105$ N.S.
True spiral angle[b] (40%-θ)	$\gamma = -0.314$ $P > 0.01$	$\gamma = 0.325$ $P > 0.01$	$\gamma = -0.348$ $P > 0.01$
True spiral angle[b] (50%-θ)	$\gamma = -0.341$ $P > 0.01$	$\gamma = 0.238$ $P > 0.05$	$\gamma = -0.295$ $P > 0.05$
True spiral angle[b] (α_m-θ)	$\gamma = -0.370$ $P > 0.01$	$\gamma = 0.318$ $P > 0.01$	$\gamma = -0.123$ N.S.

[a] ROI: relative orientation/crystallinity index.
[b] For definition, see refs. 42 and 43.

there are significant differences from one variety to the other. This conclusion is again in contradiction to that of Hebert et al.,[47] who concluded from electron diffraction studies that the degrees of orientation of crystallites within cotton varieties did not deviate appreciably from one another.

The above conclusion apparently suggests that the Hermans factor appears to be genetically inherited and is not drastically influenced by the location or by the year of growth.[46] The conviction and recommendation[39,40] in using this parameter for the characterization of cotton genotypes for an increased strength of the fibers and its use in cotton breeding programs for evolving strains with an improved strength of the fibers[30,42,43] through hybridization is thereby further strengthened. In view of the demand for increased strength of cotton fibers for modern textile processing machinery,[44] it becomes imperative for progressive cotton breeders to take advantage of these findings. This situation is more relevant to the Indian cotton scenario, where a bulk of cotton production falls in the category of the medium and short staple range and fall short in adequate strength of the fibers.

Table III Latitude and Longitude of the Locations of Growth of Cotton in India

Name of Location	Latitude	Longitude
SIRSA (North India)	29°-10′north	75°-44′east
NEW DELHI (North India)	28°-39′north	77°-13′east
NAGPUR (Central India)	21°-10′north	79°-12′east
COIMBATORE (South India)	11°-00′north	76°-58′east

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