Orientation of Aqueous Stretched Cottons as Measured by X-ray Diffraction

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SYNOPSIS

The structural changes produced by aqueous swelling and stretching in cotton is found to be irreversible. This article presents the X-ray angle data of six varieties of cotton that belong to *Gossypium hirsutum*. A marked decrease in X-ray angles was found in all treated samples, thereby indicating better orientation. Improvement in the orientation is associated with increase in the stretch. The improvement is high in all tension-dried cottons, showing the impact of the tension drying. Similar improved orientation is found in all varieties, indicating the varietal response of *G. hirsutum* cottons. © 1996 John Wiley & Sons, Inc.

INTRODUCTION

Cotton fiber is known to have a very complicated structure. Its structure has continued to attract the interest of researchers who employ special instrumental techniques to study the morphology of the fiber. Even though cotton is highly crystalline, it owes its weakness to improper orientation and other structural flaws along its length. Chemical modifications have been suggested and performed but with little encouraging results. Therefore, a search has been taken up to overcome this problem by simple methods whereby the fiber attains better orientation and assumes a solid, cylindrical shape with a virtual absence of structural flaws and other imperfections along its length.

Water, the most common swelling agent accompanied by stretch, is found to produce irreversible changes in the morphology and structure of cotton fiber.[1,2] It is assumed that water causes plasticization in the fiber, and when it is accompanied by stretch, the fibrils tend to orient toward the fiber axis and thus assume a cylindrical shape and come closer to its own state before boll opening. The modified structure is set with the annealing action of water. Based on this assumption, a study was begun to investigate the effect of aqueous swelling and stretching on the cotton fiber structure.

EXPERIMENTAL

Cottons that belong to *Gossypium hirsutum* are known to have uneven orientation throughout their length. Hence, six varieties of cotton that belong to *G. hirsutum* were subjected to swelling in water and stretched to desirable levels using a stretching device. The fibers were given two levels of stretch, 8 and 10%, over the initial length. They were subjected to two types of drying: slack drying and tension drying. Slack-dried samples after swelling and stretching were removed from the stretcher and allowed to dry in the slack condition. Tension-dried samples were left on the stretcher under tension until dry.

Aqueous Swelling and Stretching

A fiber stretcher, a stretching device, was used to carry out the stretching operation in the fibers. A uniform tuft of fibers was taken from the previously prepared hand sliver. One end of the tuft of the combed fibers was cut and fixed in one of the jaws of the stretcher. The free edges were cut to uniform

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size and fixed into the second jaw. Both the jaws carrying the fiber were carefully transferred and fixed in the fiber stretcher. A mark was made on the fibers along the edges of the jaws to indicate the slippage of fiber, if any. The initial length of the fiber was noted on the main scale and the corresponding vernier reading was also noted. The stretcher was then placed in a shallow tray containing water at room temperature. After wetting and swelling for 30 min, the stretcher was removed from the water and a predetermined stretch was given over the initial length by drawing the second jaw. Four types of samples were thus prepared by varying the stretch and condition of drying. Samples were prepared with 8 and 10% stretch over their initial length. Under each stretch, one sample was removed from the stretcher after stretching and dried loose (slack-dried sample). Another sample was dried under the stretched condition on the stretcher (tension-dried sample).

Table I Orientation Parameters of Raw and Aqueous Swollen and Stretched Fibers Based on X-Ray Technique

Cotton Fiber Variety/ State	X-ray Angles (Degrees)			Mean Orientation Angle α (Degrees)	Hermans Orientation Factor (f_x)
	40%	50%	75%		
SRT-1, raw	29.33	26.90	18.00	18.15	0.708
8% S.D.	26.47	23.85	14.62	16.85	0.747
8% T.D.	21.54	18.25	11.75	15.12	0.798
10% S.D.	26.15	23.06	14.32	16.85	0.796
10% T.D.	17.14	14.36	9.00	12.41	0.861
10% T.D. rewetted	17.50	14.60	9.50	12.30	0.859
Deviraj, raw	39.50	35.85	27.00	22.30	0.568
8% S.D.	32.00	28.85	21.00	19.19	0.675
8% T.D.	28.85	25.38	17.69	17.68	0.723
10% S.D.	31.00	27.45	19.00	18.53	0.698
10% T.D.	22.69	20.00	12.69	15.68	0.782
10% T.D. rewetted	23.1	20.00	13.10	15.00	0.798
Jayalakshmi, raw	35.00	32.00	23.46	19.90	0.651
8% S.D.	28.46	25.00	17.70	17.26	0.737
8% T.D.	18.85	16.15	10.00	13.18	0.845
10% S.D.	23.00	21.00	14.50	15.01	0.799
10% T.D.	15.00	12.69	6.92	11.93	0.872
10% T.D. rewetted	16.20	13.10	6.70	11.63	0.878
B-1007, raw	33.00	29.00	20.00	19.73	0.659
8% S.D.	25.78	22.30	13.85	16.43	0.759
8% T.D.	22.00	19.00	12.00	14.35	0.816
10% S.D.	26.14	22.71	14.36	16.58	0.756
10% T.D.	20.87	18.50	11.74	12.92	0.849
10% T.D. rewetted	21.20	19.00	11.85	13.20	0.852
Sarada, raw	34.67	31.00	21.00	20.36	0.636
8% S.D.	28.33	24.67	17.00	17.68	0.723
8% T.D.	24.00	20.75	13.33	16.43	0.759
10% S.D.	26.33	23.33	15.00	17.26	0.737
10% T.D.	20.00	17.00	10.00	14.18	0.820
10% T.D. rewetted	20.78	17.50	10.30	13.85	0.826
Bicanaire Narma, raw	31.00	27.00	18.50	18.42	0.701
8% S.D.	24.00	21.00	14.00	15.18	0.794
8% T.D.	18.00	16.00	10.00	12.90	0.849
10% S.D.	25.33	22.00	15.00	16.22	0.766
10% T.D.	19.33	17.00	10.00	13.81	0.829
10% T.D. rewetted	19.62	17.35	10.80	13.52	0.831

S.D. = slack-dried fibers; T.D. = Tension-dried fibers.

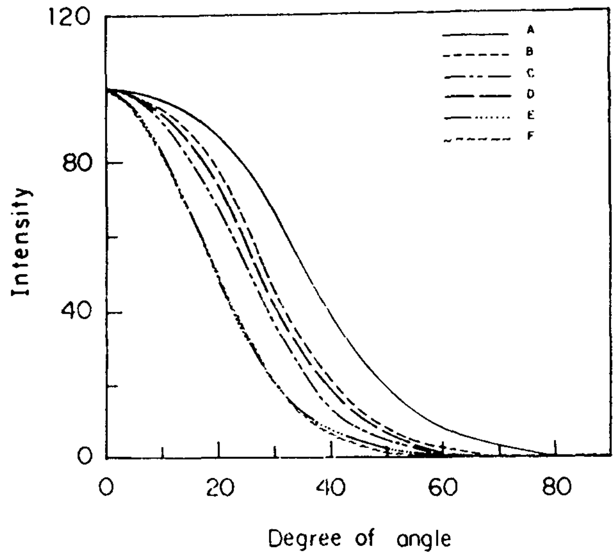

Figure 1 Peak-normalized orientation profiles of the 002 arc from fibers of the Deviraj variety: (A) control; aqueous stretched to (B) 8% and slack-dried, (C) 8% and tension-dried, (D) 10% and slack-dried, (E) 10% and tension-dried, and (F) 10% tension-dried and rewetted.

Measurement of Orientation by X-ray Diffraction

The Philips stabilized X-ray generator (PW 1130) set up with a diffractometer (PW 1050) recording accessories using Ni-filtered CuKα radiation was used for conducting these studies. The sample prepared as above was fixed on a face plate of the X-ray diffractometer. The plate was positioned such that the Ni-filtered CuKα X-ray beam emerging from a 0.5 mm collimator was incident on it. The face plate, which has a central hole to let the beam pass through, rotates in its own plane about the X-ray beam. The diffracted X-rays were received by a proportional detector placed in the horizontal plane and set at the 2θ value of 22.8°. The detector was coupled through a rate meter to a strip chart recorder. The speed was set to one revolution per hour for the face plate rotation during which various portions of the 002 diffraction arc would sweep across the counter window. The diffracted intensities were continuously recorded on the chart. Two intensity profiles were taken for each sample.

The recorded intensity profiles were smoothed out and a back ground line was drawn for the diffuse scatter. The intensity of these equatorial reflections was measured proceeding along radial lines at angular intervals of 3°. The average of the two intensity profiles was obtained. The curves normalized to equal peak heights were taken for all the samples. The azimuthal intensity distribution curve refers to the intensity distribution information over the azimuthal range 0° to 90°. From these curves, the values for 40, 50, and 75% X-ray angles were estimated.

Taking the measurements of the equatorial angle (α 002), $\mathrm{Sin}^2\,\alpha$ was estimated using the following expression suggested by Hermans[3] for native cotton:

$$\mathrm{Sin}^2\,\alpha \frac{\int_0^{\pi/2} I(\alpha)\sin^2\alpha\cos\alpha\,d\alpha}{\int_0^{\pi/2} I(\alpha)\cos\alpha\,d\alpha}$$

where α is the angle from the position of maximum intensity, and $I(\alpha)$, the intensity at α in the intensity curve (002).

The Hermans[3] crystallite orientation factor was then computed from

$$f_{(x)} = 1 - 3\sin^2\alpha$$

A random sample of the representative cotton was taken and combed thoroughly to remove loose fibers. After repeated combing to make the fibers as parallel as possible, one end of the tuft was trimmed and pasted together with a thinned quick-drying adhesive. Again, after thorough combing, the second end of the tuft was pasted together with the adhesive.

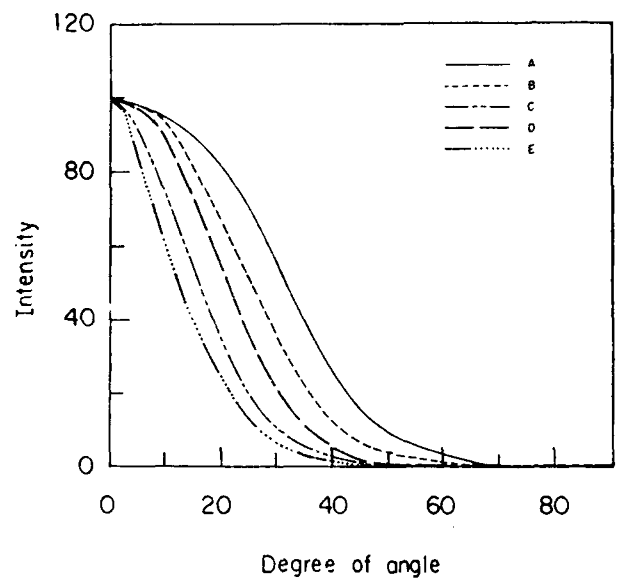

Figure 2 Peak-normalized orientation profiles of the 002 arc from fibers of Jayalakshmi variety: (A) control; aqueous stretched to (B) 8% and slack-dried, (C) 8% and tension-dried, (D) 10% and slack-dried, and (E) 10% and tension-dried.

Care was taken to make the fibers absolutely parallel to each other. Two such samples were prepared for each type of cotton.

RESULTS AND DISCUSSION

Table I gives the X-ray data, i.e., 40, 50, and 75% X-ray angles, mean fiber orientation (α), and the Hermans orientation factor $f_{(x)}$ of six varieties of untreated, water-swollen, and stretched cotton fibers at two levels of stretch, measured from normalized azimuthal intensity curves. The peak-normalized intensity curves of raw and aqueous swollen and stretched fibers from the Deviraj and Jayalakshmi variety are presented in Figures 1 and 2. Among the various X-ray angles, the variation is minimum in the mean orientation angle and it may be a better parameter to represent the orientation, as indeed was suggested by Moharir et al.[4]

It may be observed that the X-ray angle decreases as the percentage of stretch increased, indicating the improvement in orientation. The decrease is sharper in all tension-dried samples than in slack-dried samples. This trend was observed in all six varieties of cotton studied. The Jayalakshmi variety, however, registered a higher decrease in the X-ray angles and a higher increase in the Hermans orientation factor than did other varieties.

The slack-dried samples, irrespective of the stretch, show almost the same X-ray angle in all varieties except the Jayalakshmi variety, where the 10% stretched slack-dried sample shows a greater decrease in the X-ray angle than does the 8% slack-dried sample. A 10% stretch in the tension-dried samples lowers the X-ray angle to a noticeable extent and increases the Hermans orientation factor.

At each level of stretch, both the slack-dried and tension-dried samples are given the same amount of stretch, but drying under tension seems to bring more considerable changes in orientation than does slack drying. This may be because drying in a slack condition contributes to the regain of the spiral structure (stress-relaxation). On rewetting and drying, the samples gave nearly the same X-ray angle, suggesting that the structural change brought about by the treatment is quite stable. It is interesting to observe that the 10% tension-dried samples of all six varieties of hirsutum cottons showed similar orientation irrespective of their orientation at the raw state.

The above clearly indicates the amazing effect of simple aqueous swelling and stretching on cotton fibers. Moreover, these structural changes are brought in without altering the chemical structure of the cotton fibers, like mercerization. These structural changes need to be thoroughly investigated at the yarn and fabric stages to bring about a revolutionary change in the cotton textile industry.

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