NOTES

X-Ray Diffraction Studies on Cotton/Jute Blends

This communication is concerned with some exploratory studies conducted to assess whether x-ray diffraction (XRD) methods can be applied for analyzing the blend compositions (BC) in yarns spun from blends of cotton and jute fibers.

EXPERIMENTAL

Ni-filtered CuK_α radiation from a Philips stabilized x-ray generator with diffractometer and recording accessories was used for obtaining the XRD patterns. Fibers weighing about 150 mg were cut into fine powder, passed through a 150-mesh screen, and filled in the Philips rectangular specimen holder. The sample was then subjected to a pressure of 25,000 lb/in.^{2} for 5 min and its XRD pattern recorded.

For obtaining the (002) azimuthal intensity profiles, a bundle of well-parallelized fibers was mounted on a face plate kept normal to the incident x-ray beam collimated by a 0.5-mm collimator. A hole in the center of the plate allowed the beam to fall on the bundle. The diffracted x-rays were received by a Geiger counter placed in the horizontal plane and set at 20° (002) with respect to the direction of the incident beam. The counter was coupled through a rate meter to a strip-chart recorder. The face plate was rotated in its own plane at the rate of 1 rev/hr and the (002) azimuthal intensity profile recorded.

RESULTS AND DISCUSSION

The x-ray diffractograms of cotton and jute fibers are shown in Figure 1. The significant differences between the patterns pertain to (i) the resolution of the (101), ($10\bar{1}$) peaks; (ii) the breadth of the reflections, particularly of (002); and (iii) the general "amorphous" scatter, noticeable in the 2θ range of 17–20°. Out of these, it was found that (i) was best suited to easily differentiate the patterns. Therefore, a parameter to quantify the resolution of the (101), ($10\bar{1}$) peaks was defined as

$$\text{resolution parameter (RP)} = \frac{(h_1 + h_3)}{h_2}$$

where h_1, h_2, and h_3 are the heights at 2θ values of 14.8°, 15.8°, and 16.5°, respectively, in the scan above a background line drawn connecting the points in the scan at 2θ values of 10° and 18° (Fig. 1). This method of expressing the resolution is somewhat similar to that used by earlier workers.[1] The above 2θ angles were chosen after detailed considerations of the positions of the (101) and ($10\bar{1}$) reflections and the minima in between in the XRD scans of cotton and jute. It may be noticed that the RP has been defined in such a way that it increases with increasing resolution of the peaks.

The RP values thus measured for jute and cotton were 1.8 and 2.5, respectively. Even though a calibration curve of RP versus BC could be drawn using the above values, it was found that the BC could be estimated with less than 5% error only by carrying out at least four tests per sample. Therefore, a method to enhance the resolution of the (101) and ($10\bar{1}$) peaks of the XRD patterns of cotton and jute was developed. It consisted of treating the powdered sample with $3.1 \pm 0.05N$ aqueous NaOH solution at 25°C for 10 min in a flask, transferring the contents to a sinter, washing and neutralizing the alkali with 1% acetic acid, and washing repeatedly till the sample was free of alkali. The sample was then dried in an oven set at 105°C until completely dry and a pellet made as described. The XRD patterns of the treated samples are also shown in Figure 1. The increase in RP values for jute and cotton on giving the above treatment to the fibers were found to be 13% and 30%, respectively. These conditions of treatment removed impurities from jute only marginally

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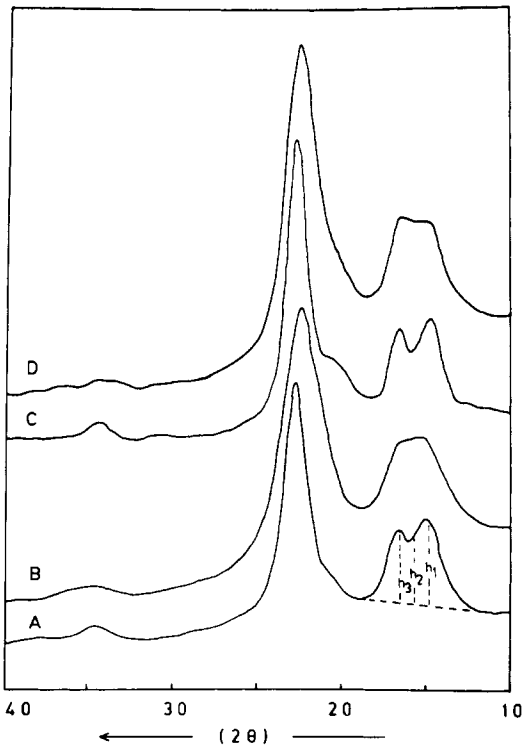

Fig. 1. X-Ray diffractograms of (A) untreated cotton, (B) untreated jute, (C) cotton treated with 3.1 ± 0.05N alkali, and (D) jute treated with 3.1 ± 0.05N alkali.

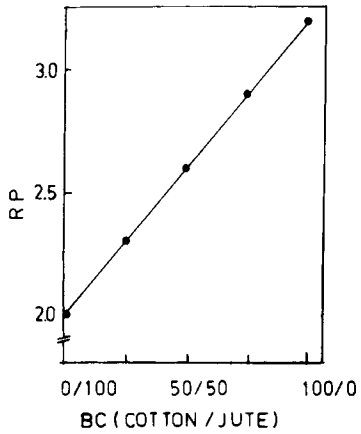

Fig. 2. Relationship between RP and blend composition obtained from synthesized XRD patterns.

and did not cause any appreciable conversion of the lattice to cellulose II. Further, these conditions of treatment gave identical RP values of 3.1 ± 0.1 for three cottons having maturity coefficient (Mc) values 0.57, 0.80, and 0.97, where $Mc = (M + 0.6H + 0.4I)/100$, M, H, and I being the percentages of mature, half-mature, and immature fibers, respectively, in the sample.

Therefore, it appears that, unlike the effect observed on lattice conversion when higher alkali concentrations are used,[2] maturity of a cotton does not significantly affect its RP under the present

conditions of treatment. As only marginal removal of the impurities in jute is involved during treatment, different varieties of jute, unless widely different in lignin and other noncellulosic contents, can be expected to give identical RP values after treatment.

From the RP values of 100% jute and 100% cotton, a calibration curve was drawn between BC and RP (Fig. 2). Blended powder samples of known composition were prepared, treated as described, and analyzed using this curve. The results are shown in Table I. It may be seen that the maximum error is only about 3%, even though only one sample was tested in all the cases. Further, when multiple tests were done on one of the samples, it was observed that the maximum deviation from the true value was of the same order for any particular test.

Attempts were also made to utilize the differences in crystallite orientation distribution in cotton and jute fibers for determining BC. The (002) orientation profiles of jute and Shyamali, a cotton with fairly high orientation, are shown in Figure 3. The intensity values on the equator have been normalized to an arbitrary value in the figure for the sake of presentation. It may be observed that the profile of jute is not very intense at angles greater than 40° from the equator, while the profile of Shyamali is fairly strong. Therefore, the intensity at these angles in a profile from a blend sample can be taken to arise mainly from cotton.

Taking the 50% x-ray angle[3] (ψ) as an index of orientation, the relationship between BC and ψ (Fig. 4) was obtained from profiles synthesised using the intensity distribution curves of jute and Shyamali fibers. Curves like this can be used directly for analysis if the control samples are available.

TABLE I
True and Estimated BC Using the RP Method

Sample no.	True BC (cotton/jute)	Measured BC (cotton/jute)
1	25/75	25/75
2	49/51	52/48
3	75/25	76/24

TABLE II
ψ/ϕ Values for Blends of Cottons with Jute

a. Group I Cottons

BC (cotton/jute)	ψ/ϕ Values						
	G. herbaceum			*G. arboreum*			
	Digvijay	Jayadhar	V797	Shyamali	AK277	G. 22	Mean
0/100	0.33	0.33	0.33	0.33	0.33	0.33	0.33
25/75	0.28	0.26	0.26	0.26	0.28	0.26	0.27
50/50	0.33	0.30	0.30	0.29	0.32	0.30	0.31
75/25	0.42	0.40	0.40	0.38	0.38	0.39	0.40
100/0	0.48	0.47	0.47	0.47	0.44	0.45	0.46

b. Group II Cottons

BC (cotton/jute)	ψ/ϕ Values						
	G. barbadense			*G. hirsutum*			
	Giza 45	Sujata	Sudan XG2 VS	BP. 52	Nimbkar 1	Deviraj	Mean
0/100	0.33	0.33	0.33	0.33	0.33	0.33	0.33
25/75	0.29	0.27	0.27	0.25	0.24	0.21	0.26
50/50	0.41	0.42	0.35	0.36	0.38	0.35	0.38
75/25	0.52	0.53	0.47	0.50	0.54	0.52	0.51
100/0	0.57	0.58	0.53	0.57	0.60	0.59	0.57

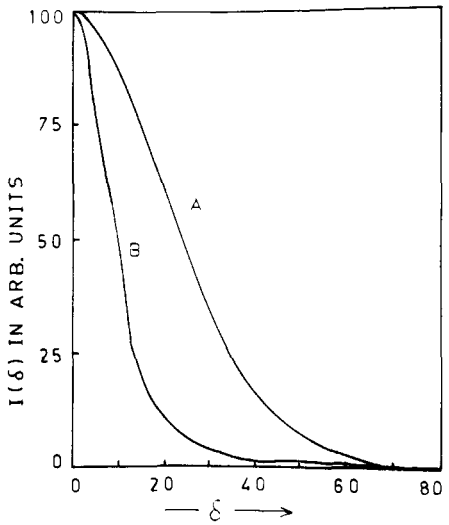

Fig. 3. Variation of diffracted intensity $I(\delta)$ from 002 planes with azimuthal angle δ measured from the equator for (A) cotton (Shyamali) and (B) jute.

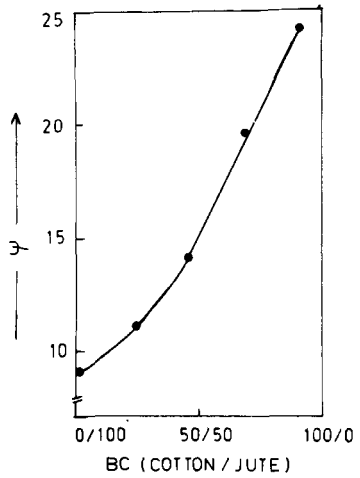

Fig. 4. Variation of ψ with BC for the jute–Shyamali system.

However, if one of the components, say, cotton, in a blend differs widely in orientation from Shyamali, the curve in Figure 4 will not be of use. Therefore, a parameter which varies with the orientation of cotton used and which is measurable from the profile of a blended sample is required to normalize differences in orientation between cottons.

Such a parameter was obtained in the 5% x-ray angle (ϕ) after trying out various other measures. For any cotton used with the variety of jute, ψ/ϕ was found to approach the same value for any particular BC, though the differences between extreme cases was still substantial (Table II). However, when the samples were classified according to botanical species, it was found that cottons belonging to *G. arboreum* and *G. herbaceum* could be classed into one group (group I) and those from *G. hirsutum* and *G. barbadense* into another (group II). The ϕ values measured from the synthesized profiles were found to be higher than 40° for all the cottons studied (covering a range ψ = 22.5°–38.5°), thus justifying the choice of ϕ for normalizing orientation differences between cottons.

The mean ψ/ϕ value for each group is plotted against BC in Figure 5. Using the curves shown, the BC value for each jute–cotton system studied was estimated from the synthesized profiles and

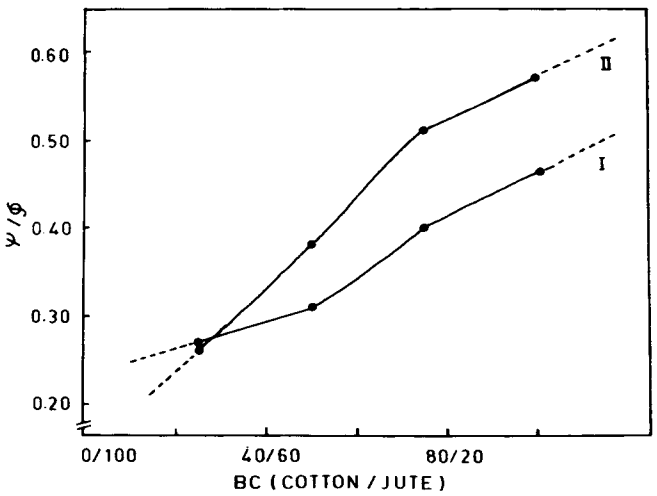

Fig. 5. Variation of ψ/ϕ with BC for blends of jute with (I) cottons from *G. arboreum* and *G. herbaceum* and (II) cottons from *G. hirsutum* and *G. barbadense*.

analyzed. It was found that the estimated BC values deviated from the actual values only by 6% in 75% of the blends considered and the *maximum* deviation was about 8% from the true values for systems in which cottons from group I were used. The corresponding values for group II were 7% and 10%, respectively. It may be mentioned here that the cottons that are likely to be used for blending with jute fibers are likely to belong to group I.

Regarding the applicability of this method if the jute used in a blend belongs to a variety different from the one used in the present study, it is not expected to be a serious handicap since it has been reported[4] that orientation differences are only marginal between different varieties of jute. Further, they are likely to be less than the differences in orientation between cottons belonging to either group.

Considering the utility of the RP and orientation methods in practice, the orientation method has certain drawbacks. First, it was found that the method is applicable only if the proportion of cotton in a blend is higher than 10%. It may be observed from Table II that while the ψ/ϕ values increase with the proportion of cotton for BC values higher than 25/75, the values for jute alone (BC = 0/100) is higher than that for a blend of 25/75. This discrepancy probably arises because the profiles from samples with cotton content higher than 25% have more resemblance with the profiles of cottons themselves while the nature of the orientation profile of jute is considerably different. Secondly, the error in the estimate obtained by this method is slightly higher. Thirdly, unless the effect of yarn structure on the orientation profiles is known, the relationships obtained from profiles of fiber bundles are of little use. However, the present study has shown that orientation methods might be useful, though with limitations, for blend analysis. Further, the time involved in measurement would be considerably less if the orientation technique can be used. Studies on the effect of yarn structure on orientation profiles are currently being made to determine whether yarns can be analyzed by this method. Until information in this regard is available, the method using RP would be better suited for assessing the BC of cotton–jute systems.

The authors are grateful to the Director, Jute Technological Research Laboratory (ICAR), Calcutta, for the supply of jute caddies. They are also thankful to Mr. C. R. S. Moni for experimental help.

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Received March 9, 1976