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Crystallinity in Jute Fiber as Revealed by Multipeak Resolution

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Synopsis

A method of multipeak resolution and computation of background scatter in x-ray diffraction from fiber has been applied to jute fibers. While crystallinity as determined by this method in raw and delignified jute fibers remains unchanged, perfection of the crystalline regions improves on delignification.

INTRODUCTION

According to the two-phase theory of fine structure, fibers and polymers consist of crystalline and noncrystalline (amorphous) regions. The crystalline regions scatter x-rays to give sharp reflections, and the noncrystalline regions form diffuse background in the x-ray diffraction pattern. Crystallinity is generally determined after separating the crystalline from the noncrystalline component, and this separation is done arbitrarily. Because this method of arbitrary separation of crystalline phase from an arbitrary amorphous phase neglects the overlap of adjacent diffraction maxima, the amorphous phase is generally overestimated.[1]

Thus, the method of determination of crystallinity should consist in resolving various peaks and drawing the background line unequivocally. For this purpose, a computational method of separating resolved diffraction maxima and background scatter was developed in this laboratory.[2]

This paper is a report on the results of application of this method to jute fiber the crystallinity of which has been measured earlier by various workers[1,3,4,5] based on arbitrary drawing of the background line.

EXPERIMENTAL

Material

The material used was defatted and dewaxed jute fiber, and the same fiber was delignified by chlorine dioxide. Thus, one raw jute and one delignified jute were investigated.

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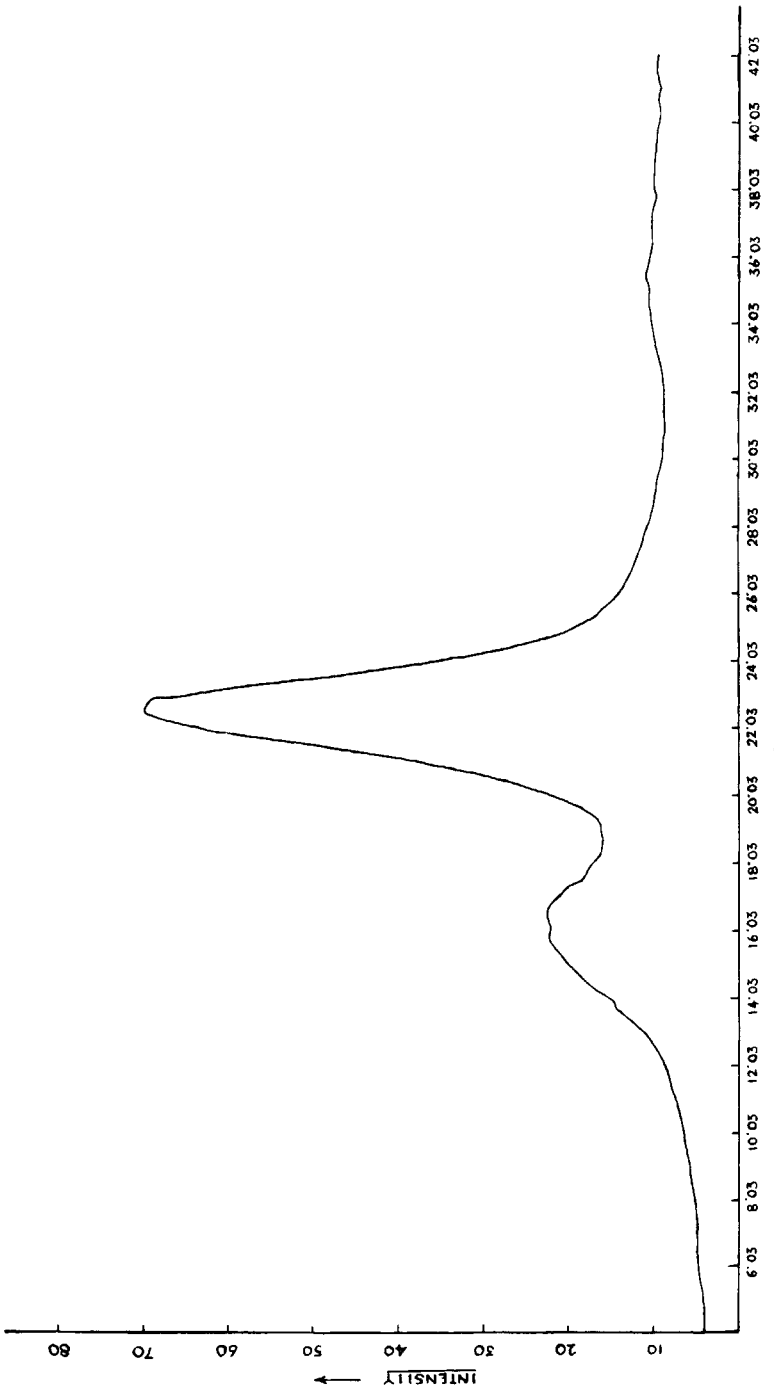

Fig. 1. Corrected and normalized x-ray diffraction trace of raw jute.

Procedure

The specimen, in the form of a bundle of parallel filaments, was held between the jaws of a special frame. Ni-filtered CuK_α radiation was used for x-ray diffraction. Equatorial x-ray diffraction traces were recorded with a modified Hilger and Watts YII5 diffractometer and a Y90 constant-output generator. Corrections to the diffraction traces were made for air scatter, polarization, Lorentz factor, and Compton scattering; they were then normalized to a convenient standard area and analyzed in the range of 3° to 50° (2θ) by a resolution program which resolves multipeak data into individual peaks and a background. The program incorporates an iterative procedure which ensures efficient convergence of the function S, where

$$S = \sum_{i=1}^{n} [Y_{(\text{obs})i} - Y_{(\text{calc})i}]^2$$

for n values of $Y_{(\text{obs})}$ obtained from the corrected diffraction traces, and for n values of $Y_{(\text{calc})}$ evaluated from the expression

$$Y_{(\text{calc})} = \sum_{t=1}^{B} Q_t + R$$

where B is the number of peaks and $Q_t = fG_t + (I - f)C_t$. Here, f is called the profile function parameter of the resolved peak; its value is 1 when the profile is Gaussian and 0 when the profile is Cauchy. The Gaussian function, G_t, is

$$G_t = A_t \exp\left[-\ln 2 \left(2\frac{X - P_t}{W_t}\right)^2\right]$$

The Cauchy function, C_t, is

$$C_t = (A_t/I) + [2(X - P_t)/W_t{}^2]$$

where X represents the angle 2θ. Each peak is defined in terms of the peak height A_t, the peak position P_t, and the peak width at half-height, W_t. The background scatter R is defined in terms of the parameters a, b, c, and d in a polynomial expression of the form $R = a + bx + cx^2 + dx^3$.

TABLE I
Resolved Peak Parameters for Raw and Delignified Jute Fibers

	Raw jute			Delignified jute		
	Peak 1	Peak 2	Peak 3	Peak 1	Peak 2	Peak 3
Profile factor	1.0	0.0	0.3	1.0	0.0	0.3
Height	4.9	10.8	60.4	5.8	10.9	65.2
Width	3.46	3.7	2.8	3.2	3.4	2.5
Position	15.13	16.48	22.7	15.12	16.50	22.7
% Area	4.3	14.0	53.25	4.7	12.8	53.4
% Area under peaks = 71.5					70.9	
% Area under background = 28.5					29.1	
% Error in best fit = 3.67					3.3	

[a] Positions and widths are given in 20°.

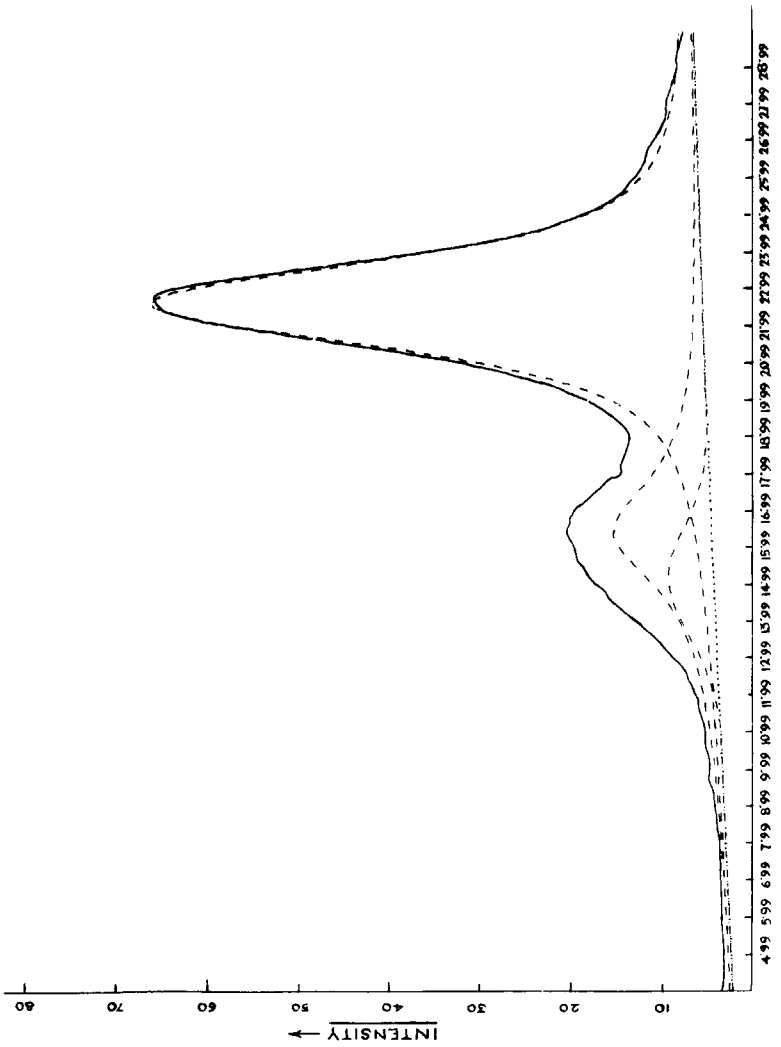

Fig. 2. Peak resolution for raw jute: (——) experimental diffraction trace; (- - -) resolved peak profiles; (· · ·) computed background.

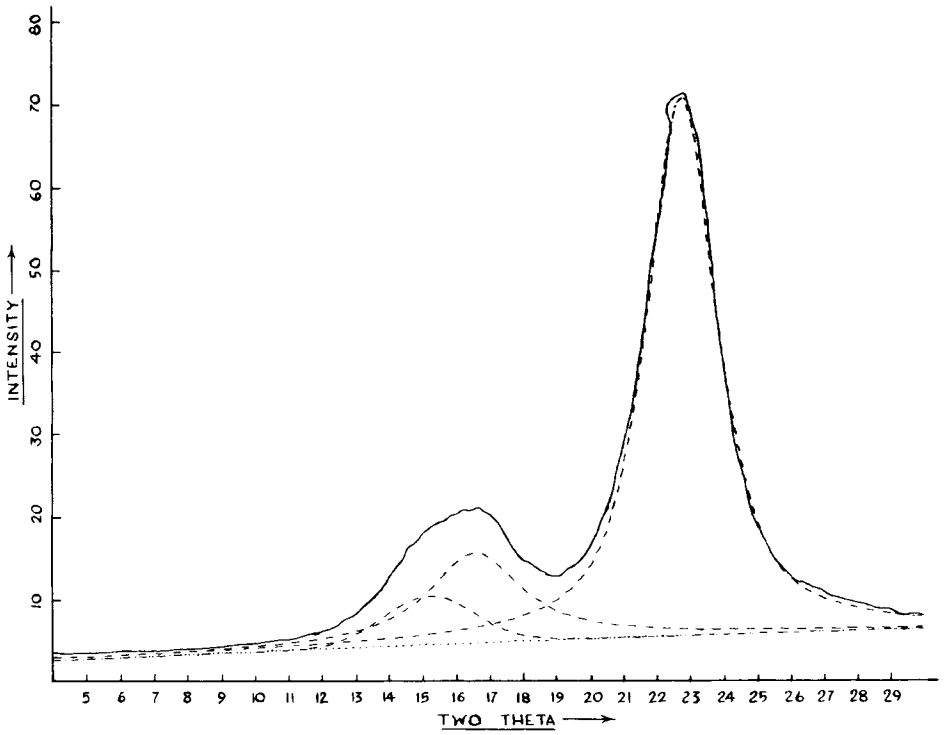

Fig. 3. Peak resolution for delignified jute: (——) experimental diffraction trace; (- - - -) resolved peak profiles; (· · · ·) computed background.

The profiles were resolved for f varying between 0 and I, in each case the program outputs the best estimate of the peak and background parameters, the limiting sum of squares S between observed and calculated Y values, and the areas under the peaks and background. The calculated area under the peak expressed as percentage of total area is the crystallinity.

RESULTS AND DISCUSSION

The corrected and normalized x-ray diffraction trace of raw jute is shown in Figure 1, and the peak profiles computed from the data in Table I, together with the background trace and the experimental envelope, are given in Figures 2 and 3.

It is seen that the difference between the crystallinity values of the two samples is negligible. It was observed that due to the breadths of the 101 and $(10\bar{1})$ peaks, the (101), $(10\bar{1})$ doublet was very difficult to resolve and, therefore, the accuracy of the position parameters was very low. The difference between the percentage areas under the peaks in the two samples is within experimental error. Crystallinity of raw and delignified jute obtained in this method is found to be higher than those reported by earlier works.[1,3,4] Thus, it is evident that the arbitrary separation of the crystalline phase from the amorphous phase involves a fraction of the crystalline scatter being counted as amorphous.

The crystallinity values of raw and delignified jute are found to be the same within experimental error. The half-width values of the peaks of delignified jute fiber are lower than those of the raw jute as found in Table I. Thus, the effect of delignification manifests itself in the improvement of the perfection of the crystalline regions rather than any change in overall crystallinity.

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