X-ray diffraction study of jute fibres treated with NaOH and liquid anhydrous ammonia

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Natural jute fibres mercerized by 20% NaOH treatment and by liquid ammonia reverted to the natural morphology on washing with water. Mercerization treatments produce more amorphous content than cellulose I to cellulose II conversion. Equatorial X-ray diffraction patterns obtained from these specimens were analysed by a function that best fits the profile giving appropriate weights to the Cauchy and Gaussian components in the peaks. Particle sizes were calculated after applying the Stokes deconvolution method to correct instrumental broadening. The difficulty in mercerizing this natural fibre is attributed to the higher lignin and hemicellulose contents that surround the cellulose I, thus preventing swelling, which is the starting point for mercerization.

(Keywords: jute fibres; mercerization; cellulose; X-ray diffraction)

INTRODUCTION

It has been reported that complete native cellulose I–hydrate cellulose II transformation is impossible in jute fibres, and X-ray photographs revealed the presence of appreciable quantities of cellulose I[1]. Mukherjee and Woods[2] observed that complete mercerization occurred for NaOH concentration greater than 10% (w/w), but cellulose I reappeared when alkali is removed by washing in water.

Recent studies on ramie (*Boehmeria nivea*) have thrown light on the mechanism of mercerization[3,4]. It was observed that mercerization proceeds through a number of crystalline alkali–cellulose complexes. The Na–cellulose structure, an intermediate product, is quite crystalline. The Na–cellulose complex transforms into cellulose II through some interesting phases. In this solid-state phase transformation the alkali enters the amorphous regions where the phase conversion starts. In the phase transformation where parallel to antiparallel chains result, the chain motions need not be extensive, for example the motions can be lateral only. In the light of this knowledge obtained from the studies on the mercerization of ramie, it was decided to study the mercerization of jute fibre treated with 20% NaOH and liquid ammonia by the recent quantitative X-ray diffraction method. The effect of anhydrous liquid ammonia on the structure and morphology of jute fibre has not been studied before.

SPECIMEN MATERIAL CHARACTERISTICS AND METHODS

Jute fibre on average contains 60% cellulose, 26% hemicellulose and 14% lignin[5]. The morphology of jute was explained on the basis of crystalline, paracrystalline and amorphous phases[6].

Fibres were taken from the middle section of Bangladesh jute (*Corchorus olitorius*). For liquid ammonia treatment the method used by the International Wool Secretariat, Valley Drive, Ilkley, Yorkshire, UK, was followed[7,8]. Jute fibres were put in a dewar flask and liquid ammonia from a pressurized cylinder obtained from a local urea plant was poured in until the fibres were fully covered with liquid ammonia at atmospheric pressure. Samples were treated with liquid ammonia (20 min) without water washing and with water washing, and with full bleaching by hydrogen peroxide (30 vol.). Another batch of samples were prepared by 20% NaOH treatment (20 min) with and without water washing.

X-ray diffraction data were collected by the system developed at the Textile Physics Laboratory, University of Leeds, UK. Equatorial diffraction profiles were collected by 15 step scanning (2θ) from a modified Hilger and Watts Y115 diffractometer mounted on a Hilger and Watts Y90 constant-output X-ray generator. Bundles of parallel fibres were examined in the symmetrical transmission mode. Fibre diagrams were also recorded.

PROFILE RESOLUTION

Diffraction data were directly fed into the University of Leeds' Amdahl mainframe computer for various geometrical corrections, normalization, resolution of peaks and correction of the resolved peaks for instrumental broadening[9]. Mean incoherent scattering ($\bar{c}$) was calculated from the best-fit third-degree polynomials of data obtained from tables for atoms in the molecular repeat. Lorentz correction for a parallel bundle of fibres, polarization correction and normalization were made by the method of Hindeleh *et al.*[9]. Peaks were resolved by fitting t separate peaks and polynomial background to the corrected and normalized intensity data. Each peak

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Table 1 Results from equatorial X-ray diffraction data for 20% NaOH and liquid ammonia treated and natural jute fibres

Specimen	Specification	Reflection	Corrected peak position, 2θ (deg)	Corrected d-spacing (Å)	Corrected size (Å)	Peak area (%)
Raw jute	Lignin not being	(1 0 $\bar{1}$)	15.60	5.68	33.18	3.42
(*Figure 1a*)	considered	(1 0 1)	17.34	5.11	35.47	3.18
		(0 0 2)	23.06	3.86	32.30	32.39
						61.00 (background)
Raw jute	Lignin being considered	(1 0 $\bar{1}$)	16.30	5.44	33.50	3.61
(*Figure 1b*)		(1 0 1)	18.01	4.93	41.12	1.93
		(0 0 2)	23.58	3.77	34.90	24.73
		lignin	22.95	3.90	–	11.78
						58.00 (background)
Jute 3	20% NaOH treated	(1 0 $\bar{1}$)	15.67	5.66	36.18	3.98
(*Figure 2*)	(20 min) and	(1 0 1)	17.47	5.08	39.26	4.37
	neutralized jute fibre	(0 0 2)	23.48	3.79	36.02	34.40
		lignin	22.07	4.03	–	7.49
						49.80 (background)
Juteam 3	Liquid ammonia treated	(1 0 $\bar{1}$)	15.14	5.85	25.45	0.99
(*Figure 3*)	(20 min), fully bleached	(1 0 1)	17.42	5.09	–	1.66
	by hydrogen peroxide (30 vol.) jute fibre	(0 0 2)	23.45	3.79	–	6.21
						91.10 (background)
J4am	Liquid ammonia treated	(1 0 $\bar{1}$)	15.07	5.88	40.20	3.03
(*Figure 4*)	and water washed	(1 0 1)	16.86	5.26	42.95	2.60
	jute fibre	(0 0 2)	22.96	3.87	34.23	31.67
		lignin	19.12	4.64	–	1.81
						60.90 (background)
Juteam 14	Liquid ammonia treated,	(1 0 $\bar{1}$)	15.88	5.58	33.25	1.55
(*Figure 5*)	fully bleached and	(1 0 1)	18.10	4.90	26.63	2.28
	carded jute fibre	–	22.17	4.01	47.95	5.58
		(0 0 2)	23.62	3.77	44.39	8.60
						82.00 (background)

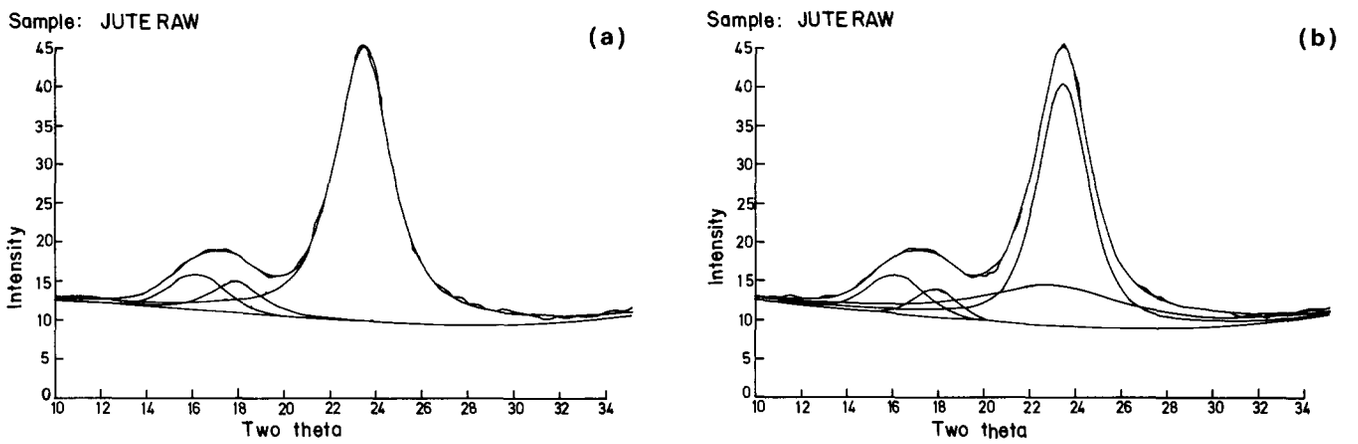

Figure 1 (a) Resolved equatorial X-ray diffraction pattern for raw jute fibre. (b) Resolved equatorial X-ray diffraction pattern for raw jute fibre considering lignin at 23° (2θ)

profile is considered to have the form:

$$f_t G_t + (1 - f_t C_t) \quad (1)$$

where G_t is the Gaussian function:

$$A_t \exp\{-\ln 2[2(X - P_t)/W_t]^2\} \quad (2)$$

and C_t is the Cauchy function:

$$A_t/\{1 + [2(X - P_t)/W_t]^2\} \quad (3)$$

Here, A_t is the peak height, W_t the width of the peak at half-height and P_t the peak position; f_t is the profile function parameter, which may vary between -0.5 and 1.0 for sensible profiles.

RESULTS AND DISCUSSION

Peaks were resolved by a function that weights the Cauchy and Gaussian components in order to give the

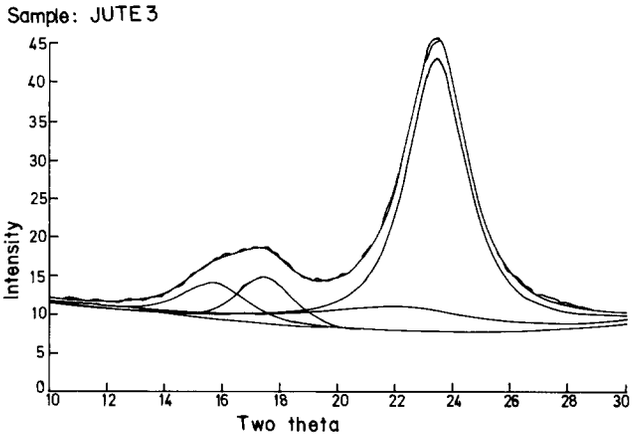

Figure 2 Resolved equatorial X-ray diffraction pattern for 20% NaOH treated (20 min), water washed and carded jute fibre (jute 3)

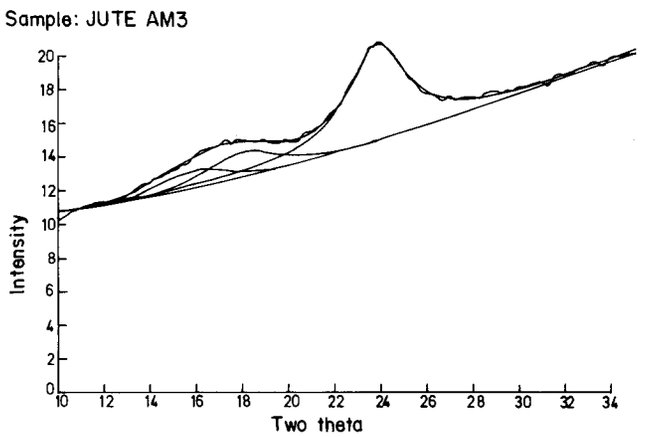

Figure 3 Resolved equatorial X-ray diffraction pattern for liquid ammonia (20 min) treated and fully bleached by hydrogen peroxide (30 vol.) jute fibre (juteam 3)

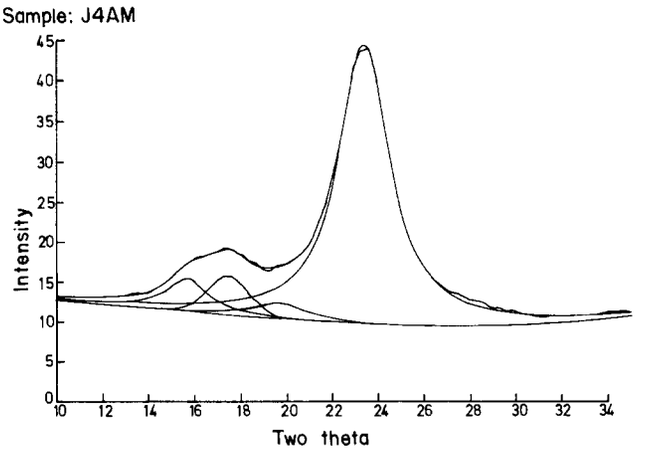

Figure 4 Resolved equatorial X-ray diffraction pattern for liquid ammonia treated and water washed jute fibre (J4am)

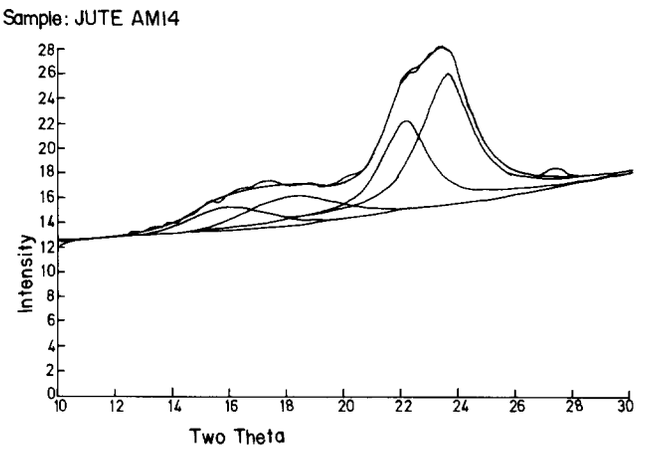

Figure 5 Resolved equatorial X-ray diffraction pattern for liquid ammonia treated, fully bleached by hydrogen peroxide (30 vol.) and carded jute fibre (juteam 14)

best fit. Particle sizes were evaluated by means of a peak broadening formula after correcting the profiles for instrumental broadening by the Stokes deconvolution method[9]. Thus analysed results from the equatorial X-ray diffraction data and resolved peaks for 20% NaOH and liquid ammonia treated and natural jute fibres are given in *Table 1* and *Figures 1–5* respectively. When 20% NaOH (20 min) treated jute fibres were examined by X-ray diffraction, a practically amorphous halo appeared along the equatorial trace; the fibre diagram showed a similar result. On washing the fibre, a diagram similar to that of raw jute appeared (*Table 1*, *Figures 1* and *2*). Liquid ammonia treated fibres also reverted to original morphology on water washing (*Figure 4*). Treatment with liquid ammonia (20 min) and fully bleaching by hydrogen peroxide (30 vol.) produce considerable amounts of amorphous content (*Figures 3* and *5*, *Table 1*), and also there is evidence of mercerization. This study suggests that chemical treatments cannot swell alpha-cellulose in jute fibre to the extent needed for mercerization, possibly due to the higher content of lignin and hemicellulose that surround the crystalline content.

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