Characterization of raw, delignified and bleached jute fibres by study of absorption of moisture and some mechanical properties

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This paper describes the response to moisture absorption and some mechanical properties of jute fibres (Corchorus capsularis) on dewaxing, delignification, bleaching and treatment with jute batch oil-inemulsion. On dewaxing the dimension of the capillaries expand from 100 Å for raw fibres to 107 Å. On scouring with detergent, the capillaries contract slightly. On treatment with jute batch (JB) oil-in-emulsion the dimension of the capillaries contract to 92 Å and the angle of contact increases, and the combined effect of these factors is a reduction in moisture absorption. On delignification and bleaching the capillaries contract in diameter and the angles of contact increase with respect to the dewaxed fibres. Moisture absorption decreases on delignification and bleaching suggesting that moisture absorption takes place in the amorphous region of the fibres. The diffusion coefficient for raw fibre was $3.37 \times 10^{-13} \text{ m}^2 \text{ s}^$ which decreases on delignification, bleaching and washing with soap. Also Young's moduli decrease on removal of lignin and hemicellulose, showing their cementing role. Tensile strength at yield for raw and JB oil treated fibres are 318 and 418 MPa respectively. On delignification, bleaching and washing with nonionic soap tensile strength decreases considerably. The strain % at break increases from 5.0 for raw jute fibres to 37.0 for fibres treated with JB oil-in-emulsion; and the elastic energy increases from 0.05 J for raw jute fibres to 1.98 J for JB oil treated fibres. Breaking toughness for raw jute fibres is 13.4 MJ m^{-3} and this reduces to very much lower values on delignification, bleaching and washing with non-ionic soap. On treatment with JB oil-in-emulsion the breaking toughness increases to 80.0 MJ m⁻³. © 1997 Elsevier Science Ltd.

(Keywords: jute fibres; moisture absorption; diffusion coefficient)

INTRODUCTION

Walls of natural cellulosic fibres like cotton, ramie, jute, wood, etc. contain long, thin threads, named microfibrils, which are seen at an intermediate range of electron microscopy¹. These microfibrils are always embedded in a matrix of other polysaccharide, and in nonsugar compounds such as lignin in wood and jute fibre. Conceptually, the rodlike cellulose microfibrils are embedded in a mixture of chains that are also rodshaped²⁻⁴. The distribution of microfibrils, hemicellulose and lignin, and the nature of the bonding between them are, however, unknown for jute and other cellulosic fibres. The general consensus is in favour of extensive hydrogen and primary valence bonding in various degrees⁵. X-ray diffraction studies establish that the crystalline portion of jute fibres contain cellulose I, and also that the microfibrils are flats^{6.7}.

Washburn⁸ developed a theory for the dynamics of capillary flow, which gives

$$(dl/dt) = r\gamma(\cos\theta/4\eta l) \tag{1}$$

where t is time, l is the length of the capillary, r is the capillary radius, γ is the surface tension, θ is the contact angle, and η is the viscosity.

The quantity, $P = (\gamma \cos \theta / 2\eta)$, measures the penetrating power of a liquid and is called the penetrativity of the liquid. For small capillaries the volume, V, of liquid that penetrates is given by

$$V = \pi \Sigma (r^3 / r^{1/2}) (\gamma \text{Cos}\theta / 2\eta)^{1/2} \cdot t^{1/2}$$

= $\pi \Sigma (r^3 / r^{1/2}) \cdot P^{1/2} \cdot t^{1/2} = C^{1/2} \cdot t^{1/2}$ (2)

where,

$$C^{1/2} = \pi \Sigma (r^3 / r^{1/2}) \cdot P^{1/2}$$

C is a measure which depends on the penetrativity and the radii of the capillaries. Slopes and intercepts obtained from the plots of log V against log t verify the $t^{1/2}$ -law and gives $C^{1/2}$ respectively.

An equation describing sorption and desorption for moderate and large times is given by⁹

$$\frac{M_{\rm t}}{M_{\infty}} = 1 - \frac{8}{\pi_2} \sum_{\rm m=0}^{\infty} \frac{1}{(2m+1)^2} \exp\left[-\frac{D(2m+1)^2 \pi^2 t}{l^2}\right]$$
(3)

where M_t and M_∞ are the amounts of diffusant taken by a sheet at times t and ∞ respectively, D is the diffusion coefficient, and L is the thickness of the sheet. It turns out that equation (3) is followed for higher values of (M_t/M_∞) when the diffusion coefficient increases with concentration, while for D decreasing with increasing

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concentration, (M_t/M_{∞}) is proportional to $t^{\frac{1}{2}}$ over the very initial region of the sorption.

From equation (3), the value of (t/L^2) for which $(M_t/M_\infty) = \frac{1}{2}$, usually written as $(t/L^2)_{\frac{1}{2}}$ is given by

$$\left(\frac{t}{l^2}\right)_{\frac{1}{2}} = -\frac{1}{\pi^2 D} \ln\left[\frac{\pi^2}{16} - \frac{1}{9}\left(\frac{\pi^2}{16}\right)^9\right] l \tag{4}$$

Thus, approximately it is found that

$$D = \frac{0.04919}{(t/l^2)_{\frac{1}{2}}} \tag{5}$$

and so, if the half-time for the sorption or desorption process is observed experimentally, the value of the diffusion coefficient assuming to be constant can be determined.

The modulus of elasticity is simply calculated from the ratio of stress and strain. The work done in pulling a sample to break is determined by measuring the area under the load-extension curve.

Jute fibres contain on average 60% cellulose, 26% hemicellulose and 14% lignin¹⁰. The hydrophilic properties of cellulose fibres are attributed to the hydrogen bonding in the spaces between the cellulose fibrils and microfibrils¹¹. Pores arise from imperfections in the lateral packing of micro-structural elements. Swelling opens up networks of capillaries which do not exist to any great extent in the dry fibres¹². This microporous structure influences the penetration of dyes, cellulose-reactive compounds, and retention of moisture. Thus understanding of the microporous structure of cellulose fibres is of great importance.

Removal of the lignin and hemicellulose by alkali and bleaching treatments are expected to adversely affect the tensile characteristics of jute fibre. The objective of this study is to investigate the microporous structure of scoured, dewaxed, delignified and bleached jute fibres from absorption of moisture, and also to study some of the mechanical properties in relation to oil treatment, dewaxing, delignification and bleaching.

MATERIAL CHARACTERISTICS

Raw fibres were always lightly combed, to free the fibres from impurities on the surface. Fibres were dewaxed by Soxhletting with 1/2 v/v alcohol-benzene mixture at a reflux rate of 8 cycles h^{-1} for 21 h. These fibres were washed with ethanol, followed by washing with distilled water, and then dried.

Combed raw jute fibres were treated with jute batch (JB) oil-in-water emulsion for 24 h; these were kept in a cellophane bag for 2 days and then dried in air.

Raw and dewaxed jute fibres were washed by treating with 10% non-ionic detergent solution (Sunperonic N) at 60°C for 2 h in an agitated bath. The scoured fibres were then washed thoroughly with hot distilled water and then dried in air, followed by drying in a vacuum. Raw and dewaxed jute fibres were bleached by soaking in 10% and 20% NaOCl solutions for 4 h at ambient temperature, then the fibres were thoroughly washed with distilled water and dried in air. Similarly raw and dewaxed jute fibres were delignified with 17.5% NaOH and 17.5% KOH solutions for 1 h at ambient temperature; then the samples were washed with distilled water until neutrality was achieved, followed by drying in air at room temperature. All the specimens, except the JB oil-in-emulsion treated specimen, were prepared by Dr E. Kronfli and Mr Ferdous Khan at the Royal Military College of Sciences, University of Cranfield, Shrivenham, England.

EXPERIMENTAL

A computer-controlled multifunctional analytical balance was set up to measure the mass of moisture absorbed with time, where data can be sampled at the maximum of 1 s^{-1}

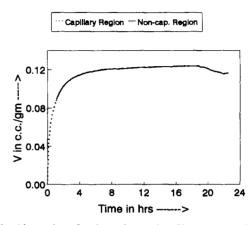


Figure 1 Absorption of moisture by raw jute fibres (*C. capsularis*) with time at 51% relative humidity

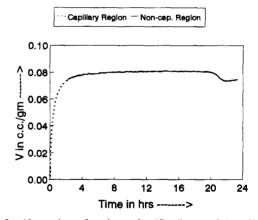


Figure 2 Absorption of moisture by JB oil treated jute fibres (C. capsularis) with time at 51% relative humidity

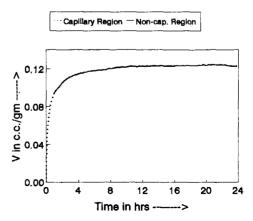


Figure 3 Absorption of moisture by dewaxed jute fibres (*C. capsularis*) with time at 51% relative humidity

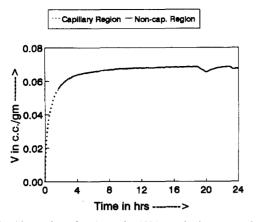


Figure 4 Absorption of moisture by 10% non-ionic soap washed jute fibres (*C. capsularis*) with time at 51% relative humidity

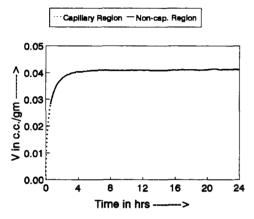


Figure 5 Absorption of moisture by dewaxed and delignified (by 17.5% KOH) jute fibres (*C. capsularis*) with time at 51% relative humidity

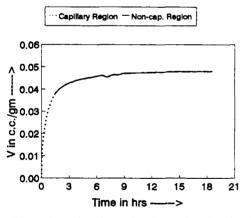


Figure 6 Absorption of moisture by dewaxed and delignified (by 17.5% NaOH) jute fibres (*C. capsularis*) with time at 51% relative humidity

to any slower rate. The samples were dried by infra-red (i.r.) radiation heat using a Philips Infraphil (150W) i.r. lamp for about 2h. These were placed on the balance which was sealed from outside. Humidity was controlled at $51.0 \pm 0.5\%$ by saturated salt solution of Mg(NO₃)₂.6H₂O kept inside the air-tight enclosure of the balance¹³.

Tensile properties were measured by a computer controlled constant rate of extension type (speed accuracy better than 0.2%) LRX Materials Testing

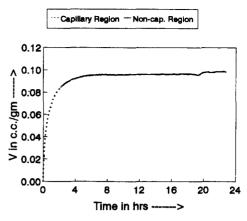


Figure 7 Absorption of moisture by dewaxed and bleached (by 10% NaOC1, 2h) jute fibres (*C. capsularis*) with time at 51% relative humidity

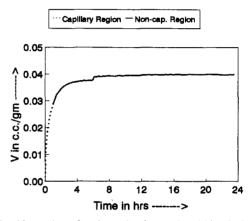


Figure 8 Absorption of moisture by dewaxed and bleached (by 20% NaOCL, 4h) jute fibres (*C. capsularis*) with time at 51% relative humidity

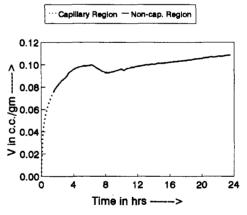


Figure 9 Absorption of moisture by benzene soaked (72h) and delignified (20% NaOCl, 1h) jute fibres (*C. capsularis*) with time at 51% relative humidity

Machine (Lloyd Instruments Ltd) which meets the ASTM E4 accuracy (0.5%) in the measurement of force and less than $5 \,\mu$ m resolution in the measurement of extension.

For optimum degree of comparability, the tensile properties of specimens having under 8% elongation at break were measured at the same rate of elongation, which was 10% of the initial length of specimen per minute. For the JB oil treated and delignified by 17.5%

NaOH jute fibres the rate of elongation was 60% of the initial length of the specimen (ASTM D2101).

Data were transmitted to the computer at 5 ms rate for analysis by software (DAPMAT). The specimens were preconditioned by keeping them in a saturated salt solution at $7 \pm 1\%$ relative humidity and 25°C temperature, following ASTM D 1776, prior to conditioning in a controlled atmosphere for testing. All specimens were tested in an air conditioned room where relative humidity and temperature were maintained at about 65% and 25°C respectively.

For tensilometry data were collected and analysed in accordance with the ASTM D 2101-94 specifications. The DAPMAT programme calculates the best straight line by the least squares method from the selected linear portion of the load–extension curve. Initial modulii were calculated from this line (ASTM D 2101). This programme also calculates the area under the load– extension curve, giving the total work done on the fibres by stretching.

RESULTS AND DISCUSSIONS

Moisture absorbtion

Figures 1-9 are the graphs of wt% increase in mass with time for raw, JB oil treated, dewaxed, delignified and bleached jute fibres. The results due to moisture absorption are summarized in *Table 1*.

The room temperature was maintained at about $28 \pm 2^{\circ}$ C, ensuring a relative humidity of $51.0 \pm 0.5\%$ for the salt used. Some of the absorption curves show fluctuations in the saturated portions due to temperature variations.

The $t^{1/2}$ law was found to be universal at the initial stage of moisture penetration by capillary action followed by a non-linear stage of moisture absorption. The slopes found from the plots of log V against log t of the linear portions of the moisture absorption (*Figures* 1-9) gave values of 0.50 having 0.91-0.99 values of R^2

(coefficient of determination). It is suggested that the non-linear portion for moisture absorption arises from the later filling of the micropores and the slower filling of enlarged pores or pockets. For the absorption of moisture by charcoal similar non-linear behaviour of the absorption of moisture with time was observed⁸.

In general, a high wt% moisture absorbed is followed by a high value for the C-factor, which depends on the penetrativity $(P = (\gamma \cos \theta / 2\eta))$ and radii of the capillaries.

For the dewaxed fibre wt% moisture absorbed by capillary action (10.0) and the C-factor (3.56×10^{-6}) are maximum (Table 1). Assuming the same length of capillaries for the dewaxed and raw jute fibres, the ratio of radii as calculated from the wt% moisture absorbed was $(r_{raw}/r_{dewaxed}) = 0.938$, suggesting expansion of the capillary diameter on dewaxing. Assuming the same penetrativity for dewaxed and raw jute fibres, this ratio was 0.94. For recording the surface topology, the raw and dewaxed jute fibres were coated with a thin film (10 Å) of gold in a sputtering coater and then these were examined under a scanning electron microscope (SEM). The SEMs at a magnification of $\times 200$ (Figures 10a and 10b) show that dewaxing does not adequately remove the wax, and as such the assumption of the same penetrativity for the raw and dewaxed jute fibres was reasonable. The dimension of the intermicellar region and

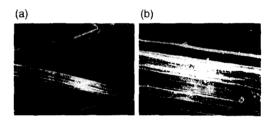


Figure 10 SEMs of (a) virgin jute fibres, and (b) dewaxed jute fibres, at a magnification of $\times 200$

Table 1 Summary of results for the absorption of moisture by raw (C. Capsularis), jute batch (JB) oil treated, dewaxed, non-ionic soap washed, delignified and bleached jute fibres at 28° C, relative humidity of 51.0% and sampling of 1/30 s

Sample: variously treated jute fibres (C. Capsularis)	Wt% moisture absorbed due to capillary action	Total wt% moisture absorbed	R^2 coefficient of determination and degrees of freedom (d.f.)	$\begin{array}{c} C \text{-factor} \\ (\times 10^{-6}) \end{array}$	Diffusion coefficient $(\times 10^{-13} \text{ m}^2 \text{ s}^{-1})$
Raw jute fibre	8.8	11.7	0.96 d.f. = 130	2.74	3.38
JB oil treated fibre	7.4	8.1	0.91 d.f. = 242	0.70	3.74
Dewaxed jute fibre	10.0	12.4	0.96 d.f. = 76	3.56	3.44
10% non-ionic soap washed at 60°C	8.5	10.6	0.95 d.f. = 194	0.75	2.34
Fibre dewaxed and delignified (17.5% KOH solution 1 h)	5.7	8.3	0.99 d.f. = 74	0.43	1.82
Fibre dewaxed and delignified (17.5% NaOH solution 1 h)	7.9	9.2	0.96 d.f. = 266	0.33	0.76
Fibre dewaxed and bleached (10% NaOCl, 2h)	8.5	9.8	0.95 d.f. = 274	1.34	2.20
Fibre dewaxed and bleached (20% NaOCl, 4h)	5.9	8.0	0.98 d.f. = 124	0.28	2.05
Dewaxed (benzene 72 h) and bleached by NaOCl, 1 h	7.7	10.9	0.93 d.f. = 194	1.42	1.30

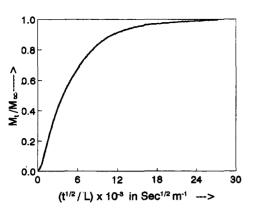


Figure 11 Reduced absorption curve for raw jute fibres (C. capsularis) at relative humidity 51%. Fibre diameter = 0.0952 mm

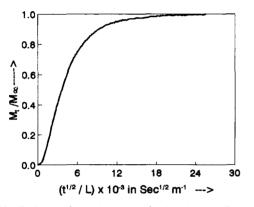


Figure 12 Reduced absorption curve for jute batch oil treated jute fibres (C. capsularis) at relative humidity 51%. Fibre diameter = 0.08808 mm

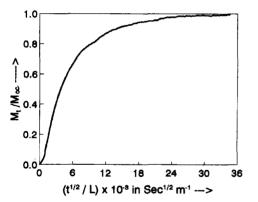


Figure 13 Reduced absorption curve for dewaxed jute fibres (C. capsularis) at relative humidity 51%. Fibre diameter = 0.07663 mm

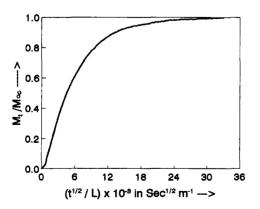


Figure 14 Reduced absorption curve for 10% non-ionic soap washed jute fibres (*C. capsularis*) at relative humidity 51%. Fibre diameter = 0.07919 mm

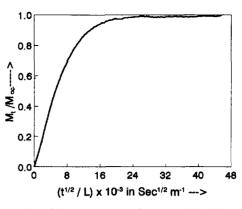


Figure 15 Reduced absorption curve for dewaxed and delignified (by 17.5% KOH) jute fibres (*C. capsularis*) at 51% relative humidity. Fibre diameter = 0.05959 mm

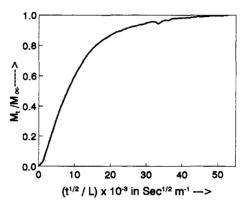


Figure 16 Reduced absorption curve for dewaxed and delignified (by 17.5% NaOH) jute fibres (C. capsularis) at 51% relative humidity. Fibre diameter = 0.04800 mm

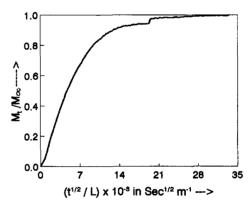


Figure 17 Reduced absorption curve for dewaxed and bleached (by 10% NaOCl, 2h) jute fibres (*C. capsularis*) at 51% relative humidity. Fibre diameter = 0.07499 mm

capillaries in raw jute fibres was estimated as about 100 Å from the sizes of the colloidal gold and silver particles deposited in the fibre¹⁴. On dewaxing, the dimension of the capillaries becomes about 107 Å explaining the higher wt% moisture absorbed.

Jute fibres treated with JB oil-in-water emulsion have the lowest moisture absorption (7.4 wt%) and a very low C-factor (0.70). These low values are due to the increase of the angle of contact and shrinkage of radii. The ratio of the radii $(r_{oil}/r_{raw}) = 0.917$, as calculated from the wt% moisture absorbed, suggests contraction of the capillaries to 92 Å on treatment with JB oil. Oils were not found to produce swelling, and ground nut oil contracted jute fibres slightly¹⁵. From the corresponding C-factors

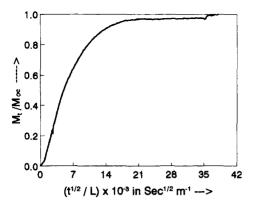


Figure 18 Reduced absorption curve for dewaxed and bleached (by 20% NaOCl, 4h) jute fibres (*C. capsularis*) at 51% relative humidity. Fibre diameter = 0.07583 mm

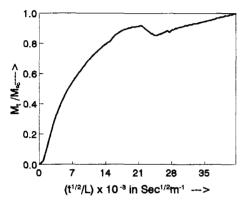


Figure 19 Reduced absorption curve for benzene soaked (72 h) and delignified (20% NaOCl, 1 h) jute fibres (*C. capsularis*) at 51% relative humidity. Fibre diameter = 0.07 mm

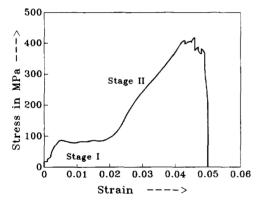


Figure 20 Graph of stress vs strain for raw jute fibres (C. capsularis) at about 65% relative humidity and 23° C

the contact angle for JB oil treated fibre θ_{oil} was found to be greater than the contact angle for raw jute fibre θ_{raw} .

From the low value of the *C*-factor (0.75) for the detergent treated jute fibres, it is suggested that scouring by detergent produces fibres with contracted radii. From the *C*-factor using the ratio of radii $(r_{\text{soap}}/r_{\text{raw}}) = 0.98$, as calculated from the wt% moisture absorbed, it was found that $\theta_{\text{soap}} > \theta_{\text{oil}} > \theta_{\text{raw}}$. The result of this study suggests that on scouring the capillaries collapse and fat is trapped in the capillaries, thus blocking the capillary absorption.

Delignification and bleaching produce very low Cfactors, due to collapsed capillary radii and the increased angles of contact, the effect of the former being the most prominent. The ratios of radii for the specimens delignified by 17.5% KOH and 17.5%

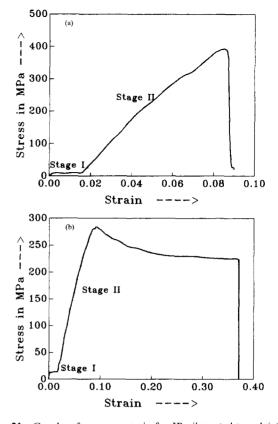


Figure 21 Graphs of stress vs. strain for JB oil treated type 1 (a) and type 2 (b) jute fibres (*C. capsularis*) at about 65% relative humidity and 23° C

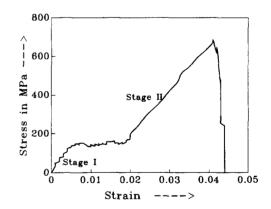


Figure 22 Graph of stress vs strain for dewaxed jute fibres (C. capsularis) at about 65% relative humidity and 23° C

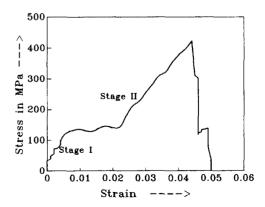


Figure 23 Graph of stress vs strain for 10% non-ionic soap washed jute fibres (*C. capsularis*) at about 65% relative humidity and 23° C

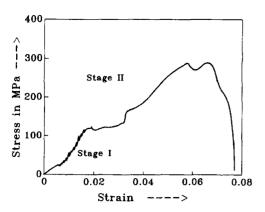


Figure 24 Graph of stress vs strain for dewaxed and delignified (17.5% KOH) jute fibres (*C. capsularis*) at about 65% relative humidity and 23°C

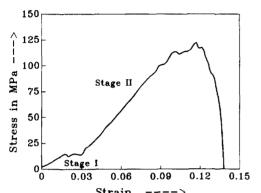


Figure 25 Graph of stress vs strain for dewaxed and delignified (by 17.5% NaOH) jute fibres (*C. capsularis*) at about 65% relative humidity and 23°C

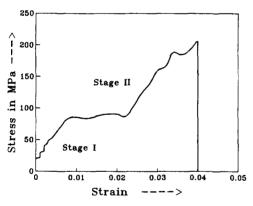


Figure 26 Graph of stress vs strain for dewaxed and bleached (by 10% NaOCl, 2h) jute fibres (*C. capsularis*) at about 65% relative humidity and 23° C

NaOH with respect to the dewaxed fibres are 0.74 and 0.86 respectively. Also, $\theta_{NaOH} > \theta_{KOH} > \theta_{dewaxed}$ explaining the lowest C-factor (0.33) for 17.5% NaOH treated jute fibres. On bleaching, a similar contraction of radii and a decrease in C-factor with respect to the dewaxed fibres are observed ($\theta_{blchd20\%} > \theta_{blchd10\%} > \theta_{dewaxed}$). Delignification and bleaching remove lignin and some amount of hemicellulose. This study of moisture absorption establishes decreased capillary absorption on removal of lignin and hemicellulose suggesting that capillary absorption in jute fibres takes place in the amorphous region.

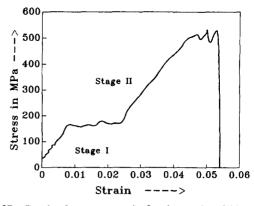


Figure 27 Graph of stress vs strain for dewaxed and bleached (by 20% NaOCl, 4h) jute fibres (*C. capsularis*) at about 65% relative humidity and 23° C

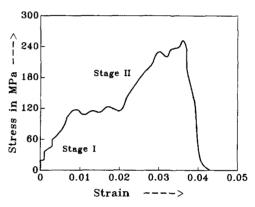


Figure 28 Graph of stress vs strain for jute fibres (*C. capsularis*) soaked in benzene for 72 h and bleached by 20% NaOCl (1 h) at about 65% relative humidity and 23° C

Diffusion coefficient

Figures 11–19 are the graphs of M_t/M_{∞} against $(t^{1/2}/L)$, known as the reduced absorption curves, for the raw, JB oil treated, dewaxed, scoured, delignified and bleached jute fibres. Fibre diameters may be approximately taken as the sheet thickness $(L)^{16}$. For each sample an average diameter from about 20–30 fibres measured at a magnification of 120 was considered. The values of diffusion coefficient (D) thus calculated are given in *Table 1*. In general the diffusion coefficients decrease on delignification, bleaching and washing with soap suggesting that moisture is absorbed in the amorphous region of the fibres.

Tensilometry

Figures 20-28 are the graphs of load vs extension for the raw, JB oil treated, dewaxed, scoured, delignified and bleached jute fibres.

In general these graphs, excepting those for JB oil treated fibres (*Figures 21a* and *21b*), show elastic elongations in two stages. Stage 1 is rather short and is followed by plastic deformation. About 50% of the delignified and bleached specimens had very low load at break, and had no stage two of elongation. Some of the delignified and bleached specimens showing stage 2 are included in the results. It is suggested that elongation first takes place in the amorphous phase of the fibres, followed by plastic flow during which entanglements at the tie ends of the molecules in the crystalline phase produce a second

Table 2 Tensile properties of raw (C. Capsularies), jute batch (JB) oil treated, dewaxed, non-ionic soap washed, delignified and bleached jute fibres at about 23°C and 65% relative humidity

Samples: variously treated jute fibres (C. Capsularies)	Young's modulus Stage 1 (GPa)	Young's modulus Stage 2 (GPa)	Strain % at break	Tensile strength at yield (MPa)	Total work due to stretching (J)	Breaking toughness (MJ m ⁻³)
Raw fibre	14.54	13.70	5.0	418	0.051	13.4
JB oil treated (1)	Absent	6.77	9.0	392	0.348	16.5
JB oil treated (2)	Absent	4.82	37.0	318	1.984	80.0
Dewaxed	24.61	23.45	4.4	680	0.050	13.4
10% non-ionic soap washed, 2 h (60°C)	16.10	6.12	4.9	422	0.040	10.1
Dewaxed and delignified (17.5% KOH, 1 h)	8.94	2.96	7.7	294	0.067	12.7
Dewaxed and delignified (17.5% NaOH, 1h)	0.70	1.40	13.7	123	0.354	8.7
Dewaxed and bleached (10% NaOCl, 2 h)	15.33	14.41	5.4	390	0.054	15.6
Dewaxed and bleached (20% NaOCl, 4h)	8.35	6.29	4.0	207	0.027	4.5
Dewaxed (benzene, 72 h) and bleached (NaOCl, 1 h)	10.57	3.17	3.5	206	0.020	4.3

stage of elastic elongation. Delignification and bleaching reduce Young's modulii and tensile strength at yield for both the amorphous and crystalline phases (Table 2), establishing the cementing role played by lignin and hemicellulose. For raw jute fibres of the variety Corchorus capsularis, the tensile strength at yield is 418 MPa (Table 2), and this is comparable to the tensile strength of 455 MPa for jute fibres of some unspecified variety¹⁷.

The specimens treated with JB oil in emulsion (Figures 21a and 21b) need special attention. Treatment with JB oil removes the elongation corresponding to stage 1 suggesting softening effect of the amorphous region on oil treatment and this produces decrease in the tensile strength at yield which are 392 MPa for type 1 and 318 MPa for type 2, for JB oil treated fibres compared to that of 418 MPa for raw jute fibres, but due to the increase in strain % at break, the total energy due to stretching increases to 0.35 J for type 1 and 1.98 J for type 2 for the JB oil treated fibres, compared to that of 0.05 J for raw jute fibres. Breaking toughness, which is an indication of the durability of articles produced from the fibre, decreases from $13.4 \,\mathrm{MJm^{-3}}$ for raw fibres to lower values on delignification, bleaching and washing with non-ionic soap; whereas on treatment with JB oil breaking toughness increases to 80.0 MJ m⁻ for type 2.

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