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PJBS

ISSN 1028-8880

Pakistan Journal of Biological Sciences

ANSInet
Asian Network for Scientific Information
308 Lasani Town, Sargodha Road, Faisalabad - Pakistan

Pakistan Journal of Biological Sciences 9 (8): 1424-1429, 2006
ISSN 1028-8880

Studies on the Physico-mechanical Properties of the Modified Jute Fibre by Sulphonation Method

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Abstract: Modification of jute fibre was due to sulphonation which were improved the physico-chemical and physico-mechanical properties of jute fibre. The change in chemical composition, morphological, geometrical structure, tensile properties and stress-strain characteristics were determined at different degree of sulphonation at optimal reaction conditions. The dimensional structure and mechanical properties of sulphonated jute fiber was studied. In sulphonation no damage and disintegration of the fibre cells were noticed except swelling of the secondary wall only and the fibre tenacity was not impaired. The sulphonation treatment in the optimal condition were improved the fineness in terms of linear density and developed flexibility, softness and compressibility of jute fibre. These values were measured in order to assess the textile importance of sulphonated jute fibre compared to cotton and other textile fibres for fine spinning. The stress-strain properties of the sulphonated fiber were much improved. The breaking extension of jute fibre was increased up to 3% with the degree of sulphonation against the initial value of 1%. The evaluation of these properties was regarded very significant towards improvement of spinnable characters and other textile performances of the sulphonated jute fibre.

Key words: Sulphonation, modified jute fibre, physico-mechanical properties, physico-chemical properties, textile fibres

INTRODUCTION

Jute is a hard and coarse fibre. It contains cellulose (58-63%) incrusted with non-fibrous substances like lignin (12-14%), hemicelluloses (21-24%), pectin (0.2-0.5%) and waxy substances, which are encrusted with the fibrils making them coarse and non-flexible (Ali *et al*., 2000). Hence, jute as textile fibres suffers from inherent defects, which limit it spinning to course yarn for producing coarse packing cloths (Ali *et al*., 2001a). Partial removal of non-cellulosic components of jute fibre to improve its characteristics has recently been a subject of much interest (Ali *et al*., 2001b). It has been reported that some chemical treatments improve the fibre characteristics of jute, modifying its physico-mechanical and physico-chemical properties (Islam *et al*., 2000; Rahman, 1989; Jabber and Rahman, 1990; Ali and Mian, 1995). Topo-physical properties of jute fiber characterized by its diameter, length, coarseness and hardness do not favor its entry in the cotton-like textile field. Moreover its mechanical properties indicate that the fiber is so strong and rigid that its fiber to fiber cohesion or tendency to close lying of fibres during spinning is very low being not suitable for making fine and flexible yarns required for diversified textile uses (Klein, 1994; Kong and Wang, 1996; Hsieh *et al*., 1995; Sarkanen *et al*., 1984). Since the fiber is a chemical entity, the values of these physical aspects are in general dependent upon the nature and internal assembles of the different chemical component of jute fibre. Incorporation of an emulsion which was made of oil water during jute fibre processing, however soft ended the fibre the usual way without any change of the intrinsic structure and composition of the fibre. Alkaline solution of sodium hydroxide, ammonium hydroxide partially dissolves only hemicelluloses and swells up the fibre without improving softness of the fibre, while solution of the chlorite removed lignin only developing more harshness in the fibre. On the contrary when jute fibre is sulphonated in sodium sulphite solution, buffering with sodium carbonate at about pH = 7 at temperature upto 170°C above normal pressure, both lignin and hemicelluloses are partially removed and thereby occurs quantitative, constitutive and dimensional changes of these internal components of the fiber (Yorston, 1942;

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Pearson *et al*., 1962). This causes modification of physical and mechanical properties of jute fiber, depending on the of sulphonation treatment.

Jute fibre was modified by sulphonated method at different conditions and these fibre samples were preserved their characteristics as textile fibre and have got improvement in their physical and mechanical properties. The physical study of these modified fibres includes their morphological examination together with the evaluation of their stress-strain properties measured in terms of tenacity; extension, stiffness, compression and toughness were determined. This study deals with the effects of sulphonation on the structural-properties of jute fibre.

MATERIALS AND METHODS

Materials: Jute plant of variety C-145 (Corchorus Capsularies) was collected from the experimental plot of Bangladesh Jute Research Institute and retted under fresh water for 18 days. The fibre was separated and sun dried. The resulting fibre was used as the fibrous raw materials. The extracted fibre was ten feet long. The cuttings were discarded and the rest of the fibre reed was cut into three parts, (i) bottom part 10%, (ii) middle part 70% and (iii) top part 20%, which were used for this investigation.

Sulphonation: Jute fibre samples 15 g was taken in a glass tube and sulphite liquor 75 mL was poured into the tube in such a way that no air bubble remained stuck inside the fibre mass. The sulphite liquor was prepared by dissolving the measured amount of sodium sulphite or bisulphite with or without sodium carbonate or hydroxide. The glass tube with the contents was sealed at the open end and was then placed inside an autoclave, which was rotated twice in a minute. The temperature was raised upto 165°C and held constant for a definite period of time. The chemicals were used as received. The sulphonation process was described in study (Ali *et al*., 2000).

Fibre diameter and density: The fibre diameter was determined at relative humidity 65% and temperature at 25°C by a standard method. The average diameter of the filaments was calculated statistically from the measurements of 100 random samples. The values for the density of the fibre in carbontetrachloride and xylene were determined by volume displacement method (Roy and Sen, 1952; Mukhopadhyay and Mukherjee, 1977). The amount of fibre was introduced into the previously weighed bottle and dried in it until a constant weight was reached. The liquid was then introduced and air bubbles removed by mechanical pressing of the fibre mass. The bottle was then put into a thermostat at 25°C until equilibrium was reached. The cap was then placed over the stopper, the bottle was taken out and dried and the whole reweighed. The sequence of operation was repeated until a constant value for the final weight was obtained.

Measurement of moisture regain: The moisture regains for different treated and untreated samples were calculated from the relation:

$$\text{Moisture regain} = \frac{\text{conditoned wt} - \text{dry wt.}}{\text{dry weight (wt)}} \times 100$$

The conditioned weight is the weight of the sample after conditioning for 24 h at 63% relative humidity and 20°C. The dry weight is the weight after dry in the sample in an oven at 105°C until a constant weight was obtained.

Measurement of cell-wall thickness: The cell wall thickness of the treated jute fibre sample was calculated by the following equation:

$$t = \tfrac{1}{2}(B - L)$$

Where, B and L are the cell breadth and lumen width of the fibres (Neelkantan and Patel, 1967), respectively.

Measurement of Luster: The luster or brightness of the different jute samples was obtained from their diffused reflectance values measured with the help of Electro Colorimeter, using blue filter against a comparative standard of magnesium oxide block of 100% reflectance.

Measurement of Bundle strength: The specimen of fibre filaments was placed as a flat bundle of about 1/4th of an inch in width between the jaws of the instrument to hold the bundle. Tension by loading weight was applied to break the fibre bundle as instructed in the procedure and the strength of the bundle at zero extension was calculated.

$$\text{Bundle strength (Ib mg}^{-1}) = \frac{\text{Breaking load in pound}}{\text{Wt. of fibre bundle in mg}}$$

RESULTS AND DISCUSSION

Physico-chemical properties: Jute fibres were changed their intrinsic structure to a very limited extent during treatment of the sulphite solutions. Measurement of moisture regain, density and geometrical properties of the fibre indicated the changes in some structural features of

Table 1: Density, moisture regain and fibre dimensions of the sulphonated fibre

Sample of jute fibre	Density (g cm^{-1})	Linear density (mg m^{-1})	Moisture % at 65% RH	Fibre diameter (μm)	Fibre length (cm)	Swelling (%)	Cell wall thickness (μm)
Untreated	1.46	2.566	10.0	41	7.0	22	5.0
Treated at 110°C, 1 h	1.46	2.48	10.1	40	7.1	22	5.6
Treated at 145°C, 1 h	1.45	2.21	10.5	39	6.9	25	6.9
Treated at 145°C, 3 h	1.47	1.93	10.9	40	7.0	31	7.8
Treated at 165°C, 1 h	1.46	1.94	10.9	38	7.5	30	8.0
Treated at 165°C, 3 h	1.48	1.85	11.2	37	6.7	35	8.5

Table 2: Brightness of the sulphonated jute fibre

	Brightness (%)		
Treatment	Unexposed	Exposed to the artificial sun light	Exposed to UV radiation
Untreated (control)	42	38	37
Sulphonated at 110°C	44	40	40
Sulphonated at 150°C	58	52	49
Sulphonated at 170°C	60	55	52
Bleached sample	70	50	48

Table 3: Rigidity of sulphonated yarn and fabric

Sample	Flexural rigidity of yarn (dyne cm^{-1})	Blending length fabric (cm)
Untreated	668	5.0
Sulphonated at 110°C	500	4.8
Sulphonated at 150°C	423	4.0
Sulphonated at 165°C	297	3.1

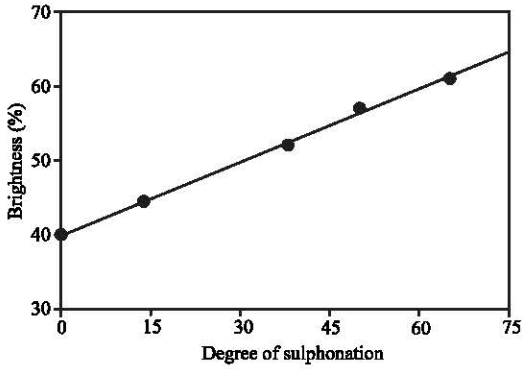

Fig. 1: Lustre of the sulphonated jute fibre

the sulphonated jute fibre. Moisture absorption of sulphonated jute fibres increases slightly at all humidities, which may be due to increase of accessible regions in the fibre (Table 1). On sulphonation the fibre does not change in length, diameter and density suggesting that the treatment does not affect the geometrical pattern of the bundle forming the fibre filament. Table 1 shows some significant decrease in linear density, increase of moisture regain and swelling in water indicating that there is some change in the order of packing of the intrinsic fibrous elements constituting the fibre bundle. It may be seen from the Table 1 that the dimension of the cell wall thickness of the sulphonated fibre increases depending on the condition of the treatment. Similar effects of cell wall thickening and lumen closing on liquid ammonia and caustic mercerization of jute fibre have been reported (Das and Roy, 1983) but they attain their normal shape in water washed samples. The change was irreversible of sulphonated fibres. It is probably due to the fact that the partial removal of lignin and hemicellulose from secondary wall induces freedom for a change in molecular configuration and rearrangement of molecular packing, which might have occupied the space available from closure of lumen and removal of interfibrillar matrics. The Table 2 shows that the effect of sunlight and ultra violet radiation. It will be seen from the Table 2 that the neat luster of sulphonated jute fibre increases on sulphonation and is much more stable than the bleached jute fibre with chlorite and peroxide. Cotton linters when treated with sodium hydroxide (Mukherjee *et al*., 1981; Roussel *et al*., 1996) and liquid ammonia (Pandey and Nair, 1975; Grover and Hamby, 1960) show the similar effect of increasing luster due to the peripheral change of the cross section to circular or oval shape and to the bulging of the cell wall. The Fig. 1 shows that the cottonisation effect is related to the degree of sulphonation, which improves luster of the jute fibre. The flexibility of jute fibre is very poor. If the cellulose molecules are taken as tiny rods, they are embedded in the matrices of lignin, pectin and hemicellulose in the ratio 3:2. Rigidity of the jute fibre depends on the geometric structure of the cellulose micro fibrils and the nature of the incrusted and adcrusted materials around them. On sulphonation the jute fibre decreases rigidity substantially and hence improves the flexibility and the results have been demonstrated in Table 3 from the values of flexural rigidity of the yarns and the bending length of fabrics obtained from the sulphonated fibre. Table 3 shows that flexural rigidity of yarns and bending length of fabric decrease with the extent of sulphonation and these properties are developed in the fibre because of partial removal of lignin and celluloses. Jute fibre is not soft; rather it gives pinching sensation on soft skin of the body. It may be obtained from the results (Table 4) that the reaction or resistance of the treated fibre to compression was comparatively lower than the untreated fibre, confirming the effect that softer the fibre, easier to compress it. The development of this property in jute fibre was due to the resulting effect of the partial removal of some incrusted materials from middle

Table 4: Hardness of the modified fibres

Specimen	Thickness (inch) of fibre mass at pressure		Compressibility (lb^{-1} in^2)	Hardness (Ibf in^{-3})	Thickness recovery (inch)	Compression recovery (%)
Untreated jute	0.884	0.650	0.769	0.0195	60.6	61.3
Sulphonated at 145°C, 1 h	0.850	0.625	0.761	0.0225	52.2	60.4
Sulphonated at 145°C, 3 h	0.920	0.591	0.758	0.0304	35.7	50.7
Sulphonated at 165°C, 1 h	0.879	0.602	0.760	0.0268	42.2	57.0
Sulphonated at 165°C, 3 h	0.925	0.579	0.750	0.0318	33.9	49.4

Table 5: Results of the physico-mechanical properties of the jute fibre filament at different stages of sulphonation

Sample of jute fibre	Linear density (g km^{-1})	Bundle strength (lb mg^{-1})	Breaking load (gf)	Tenacity (gf/tex)	Breaking extension	Textile modulus	Toughness (mg tex^{-1})	Delignification sulphonation
Untreated	2.56	12.8 (138)	70	27.3	1.0	2734	138	0
Treated at 110°C, 3 h	2.48	12.7 (136)	68	27.4	1.2	2285	165	7.5
Treated at 130°C, 3 h	2.35	12.0 (130)	66	28.1	1.3	2161	183	10
Treated at 145°C, 1 h	2.20	11.2 (122)	64	28.8	1.8	1600	259	9.8
Treated at 145°C, 3 h	1.93	9.80 (105)	58	29.8	2.6	1192	373	21
Treated at 165°C, 1 h	2.10	10.3 (110)	62	29.5	2.4	1340	325	10
Treated at 165°C, 3 h	1.85	9.40 (9.8)	52	28.8	3.0	1028	403	37

Figure in parenthesis denote the corresponding tensile strength in 100 lb $inch^{-2}$

lamella and secondary wall of the fibre cell depending on the rate of sulphonation. Fineness of the textile fibre may be determined from its width and linar density. From Table 1, it will be seen that the average fibre length remained unchanged while fibre diameter decreases very slowly indicating that the fibre-to-fibre adhesion weakens and that the splitting becomes easier after sulphonation. Decreasing of linear density with the extent of sulphonation is quite obvious due to partial removal of non-fibrous materials and has bearing effect on physico-mechanical properties of the sulphonated fibre.

Physico-mechanical properties: The results of physico-mechanical properties of sulphonated jute fibre treated at 110, 145 and 165°C for 1 and 3 h separately are shown in Table 5 alone with those of untreated raw jute fibre. It will be seen that bundle strength and tenacity of the sulphonated fibre are not significantly changed from the results obtained with the untreated raw fibre, but breaking load decreases from 70 to 52 gf while breaking extension increases from 1 to 3% as temperature and time of reaction are increased. Accordingly, textile modules which is the ratio of tenacity and breaking extension goes down from 2734 to 1028 g/tex while toughness of the fibre expressing area under load-extension curve increases from 138 to 403 mg tex^{-1} with the increase of the extent of sulphonation. From Table 5 it is found that the fibre strength slowly decreases with the sulphonated treatment. It will be seen from Table 5 that the intrinsic strength of the fibre is not very much impaired at different conditions of the treatment since the difference of about 1 lb mg^{-1} in bundle strength from its original value of 12.8 lb mg^{-1} is statistically significant. From the results it seems that the dry strength of the fibre is not significantly different under the present experimental condition of sulphonation of jute lignin. The similar effect has been

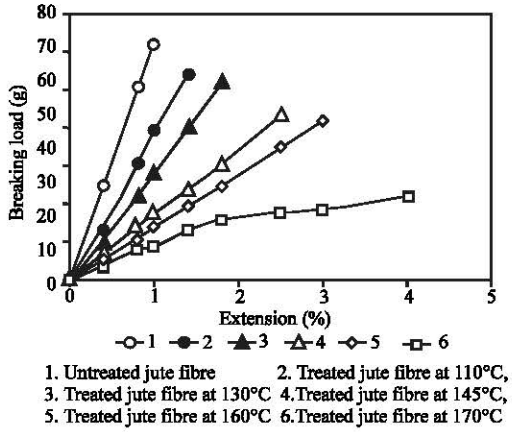

Fig. 2: Effect of load extension curve for sulphonated jute fibre

found in the case of delignification with sodium chlorite. Formation of new hydrogen bond among cellulosic units or their closer packing may be a possible explanation to it, because this may have compensated the loss of strength due to removal of lignin. In sulphonation treatment, lignin is not completely removed or degraded compared with the cholorite treatment, rather the sulphonated lignin from insoluble condensation products. So the strength properties of the sulphonated jute fibre are influenced by a combined effect from new hydrogen bonding and modified plastic behavior of residual lignin. The strength properties of the sulphonated jute fibre in terms of fibre were shown in Table 5 indicated that the sulphonation treatment increases the tenacity of the fibre very slowly in consistent with the fineness of the treated fibre. It is seen from Table 5 that jute fibre extensively treated at 165°C for 3 h breaks at an average load of 52 gf which is 25% less than that of an untreated fibre but its strength in terms of tenacity remains statistically the same.

It can be seen from Fig. 2 that the extensibility of the fibre filament gradually increases by about four folds of the original value of 1% extension at break depending on the reaction conditions of the treatment. The loss of filament strength was linearly related to delignification of the sulphonated jute fibre. The tenacity and elongation of the sulphonated jute fibre are very much comparable to those of low-grade cotton linters. Sulphonation treatment improves the effect of stiffness in the jute fibre. Results of the modulii for the sulphonated fibre at different reaction conditions have been shown in Table 5. It has been observed that the modulii decrease almost linearly with the degree of sulphonation at 150-160°C. Since textile modulus is a measure of resistance or strength of a fibre to make a unit deformation. It suggests that the sulphonation of fibre show a very significant improvement of the textile performance of jute fibre and the results show comparatively better yield in all sorts of deformation caused by mechanical stresses. The toughness results of the treated jute fibre have been shown in Table 5. It will be seen that toughness of the jute fibre increase as the fibre is sulphonated and it goes on increasing from 138 to 403 mg tex^{-1} as the sulphonation of the fibre increases from zero level to about 75% and it indicates jute fibre shows improvement of its ability to absorb strain due to any stress.

From the findings, it may be concluded that modification of jute fibre was due to sulphonation which were improved the physico-chemical and physico-mechanical properties of jute fibre. The change in chemical composition, morphological, geometrical structure, tensile properties and stress-strain characteristics were determined at different degree of sulphonation at optimal reaction conditions. Jute fibre appeared to be brighter and softer and fibre strands were more easily individualized on fine gill pins, after having sulfonated at optimal conditions. The sulphonation treatment in the optimal condition were improved the fineness in terms of linear density and developed flexibility, softness and compressibility of jute fibre. These values were measured in order to assess the textile importance of sulphonated jute fibre compared to cotton and other textile fibres for fine spinning. The evaluation of these properties was regarded very significant towards improvement of spinnable characters and other textile performances of the sulphonated jute fibre.

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