Ling Duan,
Weidong Yu

Novel and Efficient Method to Reduce the Jute Fibre Prickle Problem

College of Textiles,
DongHua University,
Shanghai, 201620, P. R. China
E-mail: wdyu@dhu.edu.cn

Abstract
The physical especially bending properties of jute fibres treated with dimethyl sulphoxide (DMSO) were studied in this work. To the knowledge of these authors, the influence of DMSO treatment on the prickle properties of jute fibres has not been investigated earlier. The results of the investigation indicate that 8 hours of DMSO treatment at 80 °C leads to a decrease in the equivalent bending modulus of up to 38.3%. This decrease was due to the swelling of fibrils and the removal of non-cellulosic materials. It is significant to improve the wear behaviour of jute fabrics. Suitable chemical treatment not only slenderised the jute fibres, but also removed the wax dramatically.

Key words: *jute, dimethyl sulfoxide, prickle, SEM, FTIR, XRD.*

Introduction

Jute is the second most common natural fibre produced in the world. It is grown extensively in Bangladesh, India and China [1]. Jute fibres have so many advantages such as sustainable, biodegradability, being low density, low cost, and high specific properties [2]. The traditional uses of jute are as sack, bag, hessian cloth/burlap, gunny sack, gunny bag, yarn,woven bag/sack, carpet backing, etc [3]. The prickle of jute fibres is an important influencial factor for their emerging use as a raw material in the manufacture of industrial textiles. Morphologically jute fibre is composed of a number of ultimate cells, which are cemented by noncellulosic content such as lignin and hemicellulose [4, 5]. Jute contains much more lignin than other bast fibres [6], which increases the fibre stiffness and prickle problems [7].

Yu and Liu [8] used a buckling test to measure bending resistance. They solve differential equations for the bending mechanics and showed that the effective bending modulus can be determined from a linear plot of critical stress against (D^4/L^2), where D - fibre diameter and L - fibre length. Yu et al [9] investigated ramie fibres bending status. Liu [10] found suitable NaOH treatment not only slenderised hemp fibres but also softened them dramatically. Shen [11] investigated the compressing and bending characteristics of ramie, flax, jute, kenaf, bamboo and bamboo pulp fibres by means of self-developed Fibre Axial Compression and a Bending Analyzer (FACBA).The effective bending modulus of jute is of a high level due the special character of jute fibre.

There is no report on the evaluation and improvement of the jute prickle problem by DMSO treatment. The objective of this research was to decrease the bending modulus and prickle problems of jute fibre after DMSO treatment in different conditions. The fibres were characterised by means of chemical analysis, optical microscope, SEM, FT-IR, XRD and a bending test.

Experimental

Materials

The starting materials used in the study were *Corchorus olitorius* L. (Tiliaceae) (Wuxue Xinjin Jute Textile Co., Ltd, China). Dimethyl sulfoxide (DMSO). Other chemicals were of analytical purity and used without further purification, obtained from SCRC (China). The approximate chemical composition of jute includes cellulose (59 - 71 wt.%), hemicelluloses (12 - 13 wt.%), pectin (0.2 - 4.4 wt.%), fat/wax (0.5 wt.%) and lignin (11.8 - 12.9 wt.%).

DMSO treatment

All the samples were thoroughly combed. The chemical compositions were 0.55% wax, 12.7% Klason lignin, 15.8% hemicelluloses, 65.0% cellulose and 0.96% pectin. Jute (cut into 3 – 4 cm in length) was formed in a 500 ml flask with dimethyl sulfoxide for different times (1, 2, 4, 8 h) and temperatures (40, 60, 80 °C). The samples were then thoroughly washed with distilled water until the residues were neutralised, and further dried at 105 °C for 2 h.

Characterisation

Optical microscopy analysis

An optical microscope (Olympus CH-2) was used to measure the change in fibre diameter and cross section under different DMSO treatment conditions. The average of at least ten measurements of the diameter in different regions was taken to represent the diameter of a single fibre.

Chemical analysis

The chemical composition of samples such as wax, pectin, cellulose, hemicellulose and lignin was analysed using in accordance with GB5889-86 (China National Standard, 1986).

SEM analysis

A scanning electron microscope (JSM-5600LV, Jeol, Japan) was used to study the morphology of the fibres after the treatments. Samples were mounted on metal stubs with double-faced tape and surface coated with gold using a vacuum sputter coater. All images were taken at an accelerating voltage of 13 kV.

XRD analysis

The crystallinity of the fibre samples was measured with an X-ray diffractometer (D/Max-2550 PC, Rigaku, Japan) using Cu Kα radiation (λ = 1.5406 Å) at 40 kV and 200 mA. Scattered radiation was detected in the range of 2θ = 5º - 60 º at a scan rate of 4 º/min.

The relative degree of cellulose crystallinity was calculated using ***Equation 1*** [12, 13]

$$x = \frac{\sum I_c}{\sum I_c + \sum I_a} \qquad (1)$$

where, $\sum I_c$ is the diffraction intensity of crystal and $\sum I_a$ is that of the amorphous area.

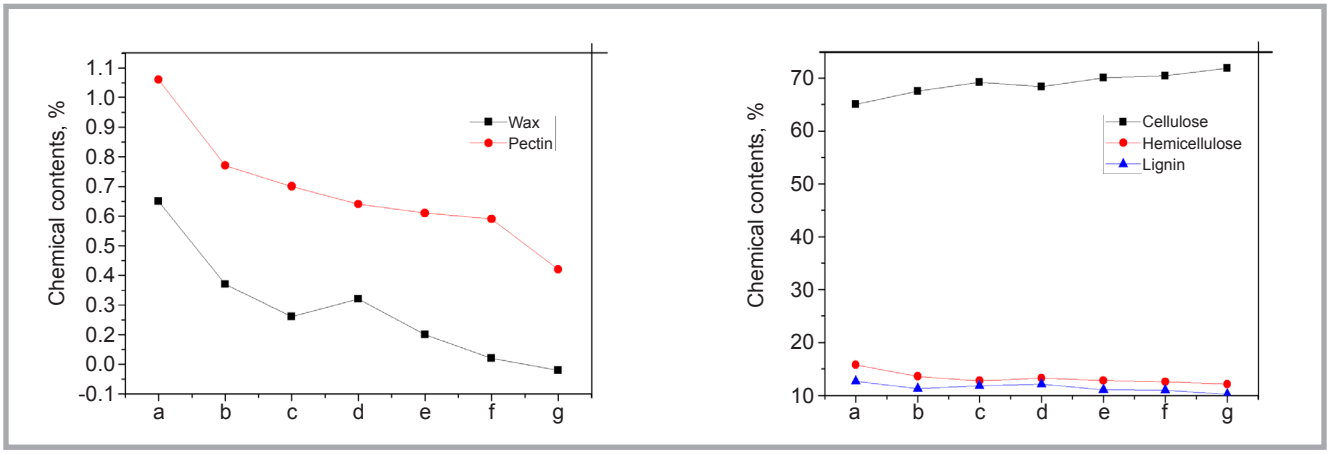

Figure 1. *Chemical content change: a) raw, b) DMSO 40 °C 8 h, c) DMSO 60 °C 8 h, d) DMSO 80 °C 1 h, e) DMSO 80 °C 2 h, f) DMSO 80 °C 4 h and g) DMSO 80 °C 8 h.*

FT-IR Analysis

Fourier transform infrared spectroscopy (Nicolet-5700, MA, Waltham, USA) was employed to analyse chemical composition changes in the structure of the fibres before and after treatment. All spectra were taken in the range of 4000 - 400 cm^{-1} at a resolution of 4 cm^{-1}.

Bending test

A single fibre axial compression bending analyser FICBA-JSW03 was used to calculate the equivalent bending modulus (The FICBA was researched and developed by the Textile Materials and Technology Lab at Donghua University [11]). Details were described by Liu [8]. The crosshead speed was 0.1 mm/s. Fibre samples were extracted with ether and ethanol for clearing, then conditioned for more than 2 days under a standard temperature of 20 ± 2 ºC and relative humidity of 65 ± 2%, and lastly the sample fibres were turned into single-fibre needles, using methods as described by Liu [8]. The length and diameter of single-fibre samples were measured by an optical microscope with a CCD camera.

Table 1. *Chemical contents of jute fibres at different treatment conditions.*

Sample	Weight loss, %	Wax, %	Pectin, %	Cellulose, %	Hemi-cellulose, %	Lignin, %
Raw	-	0.55	0.96	65.00	15.80	12.70
DMSO 40 °C 8 h	4.18	0.27	0.67	67.52	13.62	11.27
DMSO 60 °C 8 h	4.65	0.16	0.60	69.20	12.77	11.83
DMSO 80 °C 1 h	3.88	0.22	0.54	68.43	13.27	12.10
DMSO 80 °C 2 h	5.53	0.10	0.51	70.08	12.82	11.05
DMSO 80 °C 4 h	5.94	0.02	0.49	70.49	12.58	10.98
DMSO 80 °C 8 h	7.44	-0.02	0.32	71.89	12.15	9.20

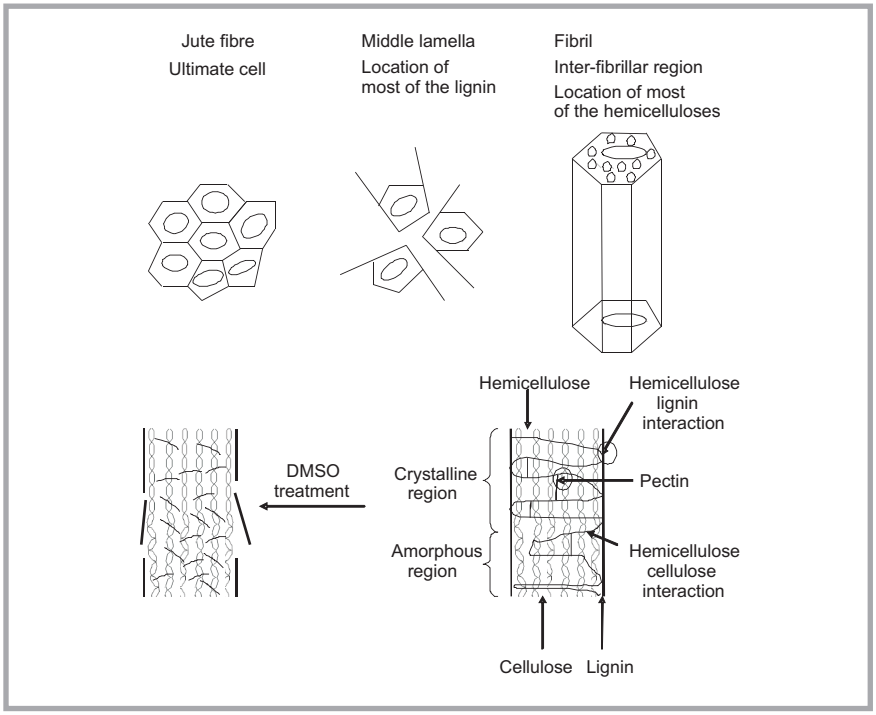

Figure 2. *Schematic representation of the DMSO treatment of jute fibres based on literature studies [14 - 16].*

The bending modulus was calculated by measuring the protruding length and diameter of fibre needles and the critical force, P_{cr}, obtained from the peak point of the force-displacement curve. This is detailed presented in the following formula (2)

$$P_{cr} = \frac{20.19E_BI}{L^2} = \frac{20.19E_BK_BI_0}{L^2} = (0.99E_BK_B)\frac{D^4}{L^2} \quad (2)$$

where, P_{cr} - critical force, E_B - equivalent bending modulus, K_B - shape factor, L - fibre protruding length, I_0 - moment of inertia of the circle.

Results and discussion

Chemical contents

The weight loss and chemical contents of jute fibres after DMSO treatment are shown in ***Table 1***. Changes in the chemical composition are given in ***Figure 1.***

A schematic representation of a fibre wall segment in jute and treatment by DMSO

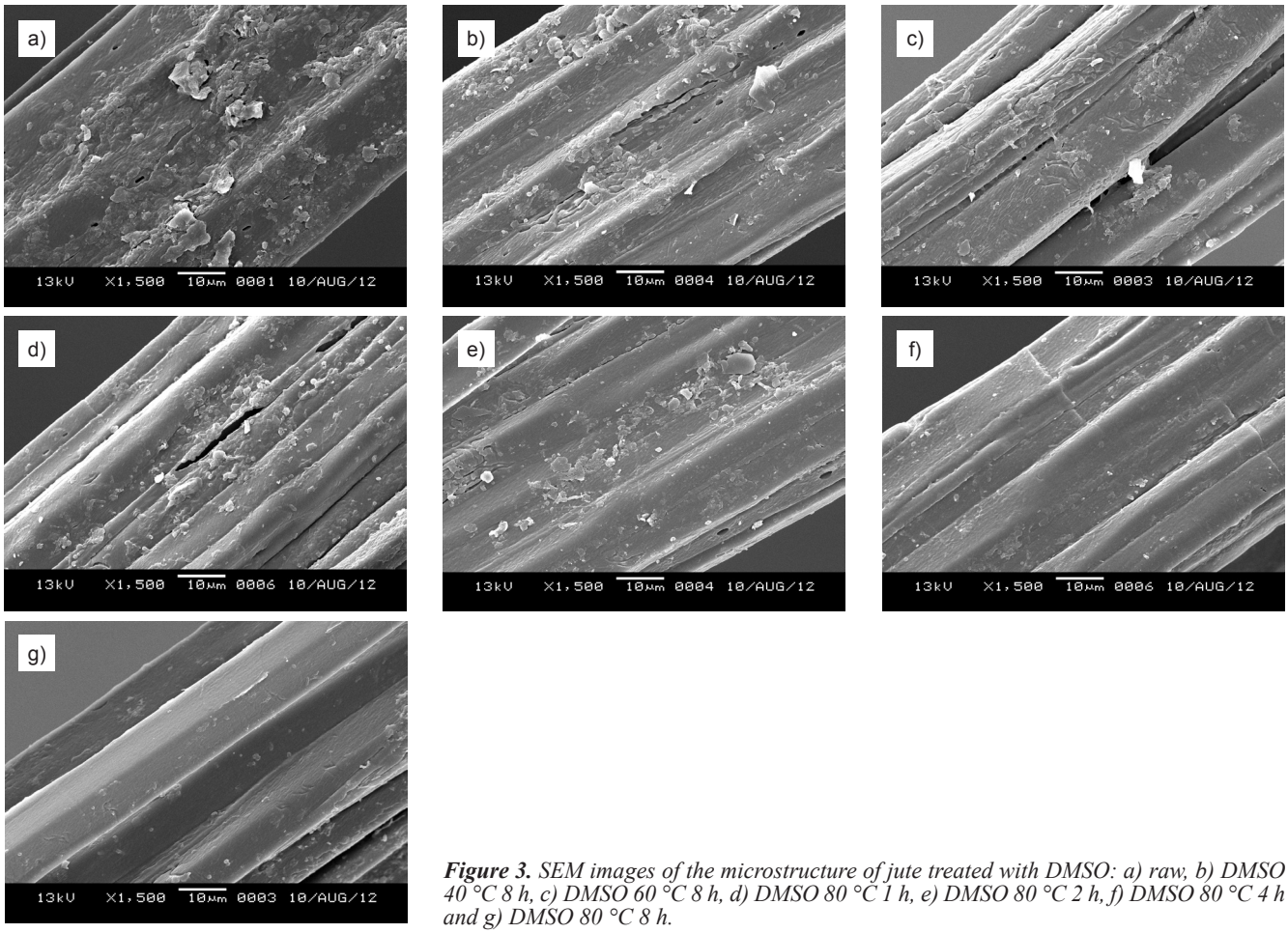

Figure 3. *SEM images of the microstructure of jute treated with DMSO: a) raw, b) DMSO 40 °C 8 h, c) DMSO 60 °C 8 h, d) DMSO 80 °C 1 h, e) DMSO 80 °C 2 h, f) DMSO 80 °C 4 h and g) DMSO 80 °C 8 h.*

based on literature studies [14 - 16] is shown in ***Figure 2***. Where cellulose is linear, hemicellulose is a two dimensional polymer and lignin - a three-dimensional macromolecule. Cellulose is the main structural component of plant cell walls, and different cellulose chains interact with each other by hydrogen bonding and van der Waals forces [16].

Lignin is believed to be located in the inter-cellular region [17], which is considered to be the cellular glue that provides plant tissues and individual fibres that yield compressive strength and stiffen the cell wall [18]. Some experimental facts have been obtained which indicate that hemicelluloses act as the cementing material for the small ultimate cells of jute, which is believed to be mainly present in inter-fibrillar regions within the ultimate cell [19], However, hemicellulose can link cellulose fibres into microfibrils and cross-link with lignin, creating a complex network of bonds that provide structural strength [20]. Pectin is composed of cross-linked polysaccharides forming a hydrated gel that "glues" the cell-wall components together [21].

As shown in ***Figure 2***, during the DMSO processes, the swelling of fibrils in the fibres would facilitate the removal of non-cellulosic materials such as pectin, hemicellulose and lignin [22]. Nakatani found that DMSO has stronger affinity for lignin than for cellulose and hemicellulose for dry wood and preswollen wood [23], which help the removal of lignin.

The pectin and lignin content of jute fibres were decreased by about 66.67% and 30%, respectively.

The fibre becomes softer due to the reduction of pectin and lignin, which causes an increase in the fibre stiffness [7]. Cuticular waxes are a mixture of several compounds, such as very-long-chain fatty acids, fatty aldehydes, primary and secondary alcohols, ketones,and esters [24]. The wax removal efficiency was found to decrease by 96% after treatment at 80 °C over 4 h, with the dewax effect of DMSO treatment being in agreement with observations on sisal fibres by Pei-Ying [25].

Fibre diameter

The change in diameter after DMSO treatment is shown in ***Table 2***. The diameter showed no obvious change under low temperature treatment. The results of this investigation indicate that the diameter of jute fibres was decreased by 17.61% under 80 °C 8 h DMSO treatment. The

Table 2. *Fibre diameter of jute fibres at different treatment conditions.*

Sample	Temperature, °C	Heating time, h	Mean diameter, µm	Standard diameter deviation, µm
Raw	-	-	56.96	12.55
DMSO 40 °C 8 h	40	8	54.30	9.90
DMSO 60 °C 8 h	60	8	51.05	11.40
DMSO 80 °C 1 h	80	1	52.89	11.51
DMSO 80 °C 2 h	80	2	47.30	5.97
DMSO 80 °C 4 h	80	4	47.89	5.82
DMSO 80 °C 8 h	80	8	46.93	7.06

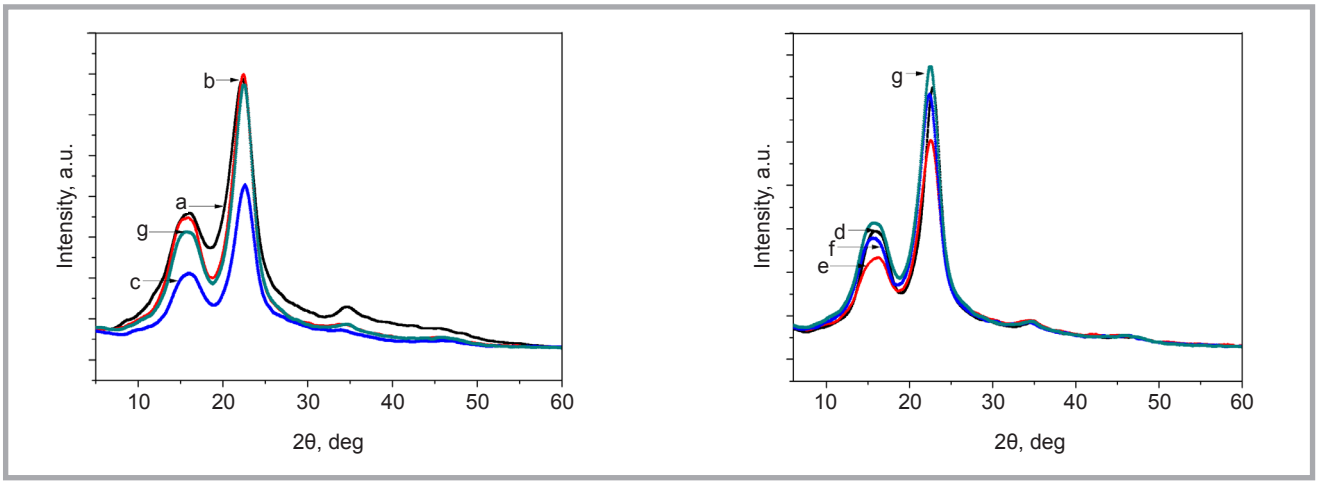

Figure 4. *X-ray diffraction profiles of jute: a) raw, b) DMSO 40 °C 8 h, c) DMSO 60 °C 8 h, d) DMSO 80 °C 1 h, e) DMSO 80 °C 2 h, f) DMSO 80 °C 4 h and g) DMSO 80 °C 8 h.*

removal of non cellulosic constituents in DMSO created displacement between fibres and their swelling was observed to have occurred. The loss in weight caused the fibre diameter to fall sharply.

The current hypothesis for fabric-evoked prickle is that in some fabrics with fibres protruding from the surface, which are able support loads above the threshold before buckling (usually coarser or shorter fibre ends because the fibre buckling load is proportional to ***d4/12,*** where *l* is the fibre length and *d* the fibre diameter), this can trigger nerve endings, leading to the sensation of prickle [26]. The swelling of jute fibres by DMSO treatment, in particular, leads to the destruction of the mesh structure and splitting of fibres into finer filaments . The splitting of cemented fibres causes a reduction in the fibre diameter, which improves the prickle problem.

SEM analysis

It can be seen that raw jute fibres are covered with some impurities (***Figure 3.a,*** see page 30). Samples treated at different conditions (temperature: 40, 60 and 80 °C and time: 1, 2, 4 & 8 h are presented in ***Figure 3.b - 3.g)***.

The surface of raw jute fibre was embedded in a matrix of hemicellulose, lignin, pectin, waxy substances and so on [18]. For chemical analysis, the removal efficiency of impurities was very remarkable at high temperature, with the treatment time increasing at temp. 80 °C. The fibre bundle structure becomes loose and more of the ultimate cell could be seen from picture. After 4 hours of treatment, a clean surface was observed, with only a little debris adhering to the fibre surface. ***Figure 3.g*** shows that jute fibre after treatment had a clearer surface and polygon shape. A crisscross network of fibrils could be observed at the primary wall, just as in Mukherjee's observation [27]. As the DMSO effect deepened, the structure of the primary wall broke , and we could see more fibrils parallel to the fibre axis as well as the helical orientation of fibrils in the secondary layer (***Figure 3.g***).

Significant changes in surface morphology were observed after DMSO treatment. Fibres were swollen in the DMSO solution [28], which caused relative displacement between them. The removal of surface impurities, hemicellulose, lignin and waxes was found to result in smooth surfaces and better fibre separation after DMSO treatments (***Figure 3.g***), indicating that DMSO treatment is effective for impurity removal, especially for wax and pectin.

Table 3. *Crystallinity of jute fibres at different treatment conditions.*

Sample	Temperature, °C	Heating time, h	Crystallinity, %
Raw	-	-	55.37
DMSO 40 °C 8 h	40	8	66.00
DMSO 60 °C 8 h	60	8	65.96
DMSO 80 °C 1 h	80	1	66.27
DMSO 80 °C 2 h	80	2	72.06
DMSO 80 °C 4 h	80	4	73.64
DMSO 80 °C 8 h	80	8	75.27

XRD analysis

XRD studies of the treated and untreated jute fibres were done to investigate changes occurring in crystallinity. Sharp peaks occurred around 2θ - 16° and 22.6°, which were believed to represent a typical cellulose I form, indicating that the crystal structure of cellulose was not changed during the chemical treatment. The crystallinity of each sample was also calculated and is listed in ***Figure 4*** and ***Table 3***. The crystallinity of jute raw fibres was estimated as 55.37%, while jute fibres showed an estimated crystallinity of 66% after treatment at 40 °C for 8 h, and a crystallinity of 75.27% at 80 °C for 8 h. The increase in crystallinity was undoubtedly due to the dissolution of amorphous zones and a part removal of hemicelluloses, lignin and impurities, leading to the realignment of cellulose molecules. An increase in crystallinity after chemical treatments has been reported by several authors [29].

FT-IR analysis

FTIR spectroscopy is a powerful tool in the analysis of the chemical change in the samples treated. FT-IR spectra of the untreated and treated samples are shown in ***Figure 5***. An absorbance peak at approximately 3369 cm^{-1}, representing O-H stretching of the hydrogen bond network, could be observed in the spectra [30], which becomes less intense upon DMSO treatment. Such a decrease is due to the breaking of the hydrogen bond between O–H groups of cellulose. The 2900 cm^{-1} band is assigned to C-H stretching in me-

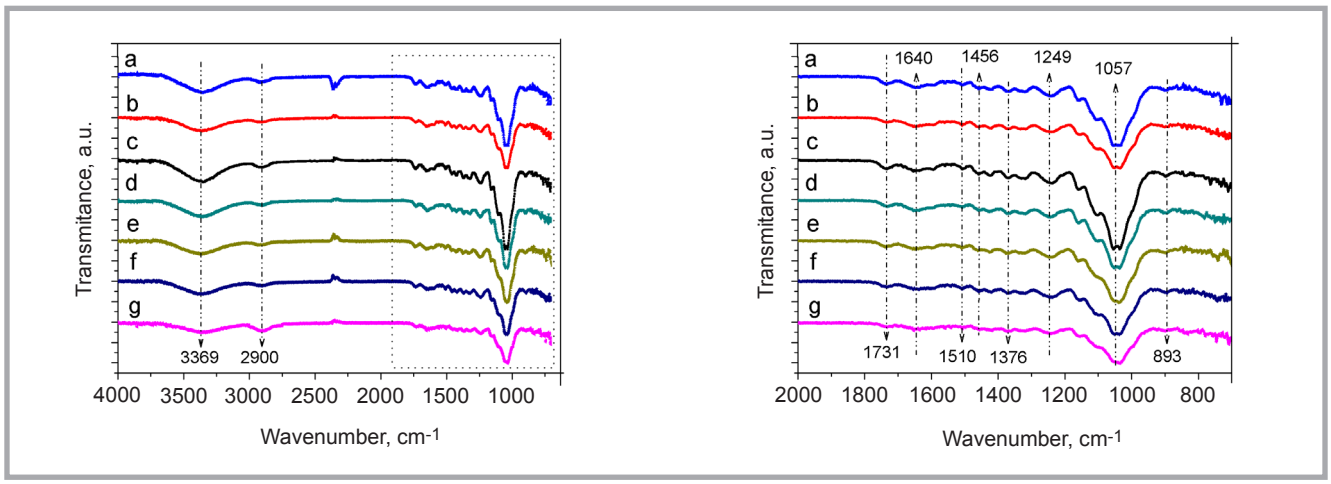

Figure 5. *FTIR spectra of a) raw, b) DMSO 40 °C 8 h, c) DMSO 60 °C 8 h, d) DMSO 80 °C 1 h, e) DMSO 80 °C 2 h, f) DMSO 80 °C 4 h and g) DMSO 80 °C 8 h.*

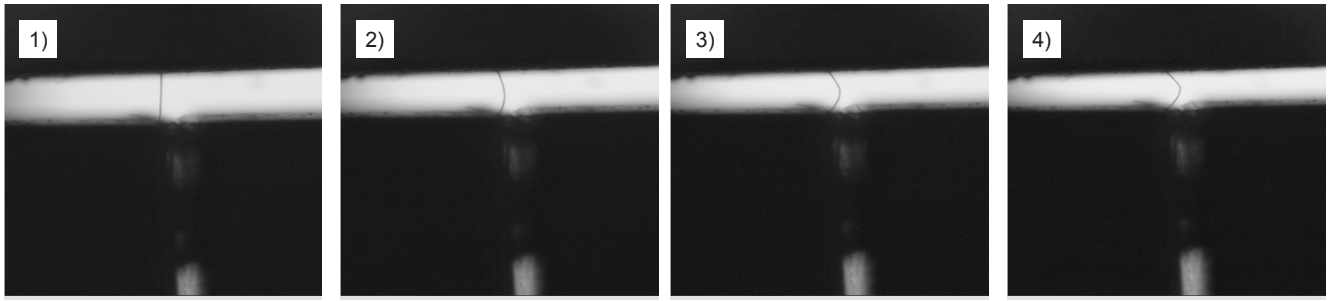

Figure 6. *Single fibre bending process: 1) axial compression bending, 2) concentration of stress, 3) beginning of axial compression bending fracture, 4) continuous compression after break.*

thyl and methylene groups in cellulose and hemicellulose [31]. Absorption peaks at 1057 and 893 cm^{-1} are associated with C-O stretching and β-glycosidic linkages of the glucose ring of cellulose [32]. These absorptions are consistent with those of a typical cellulose backbone [19]. Almost the same absorption peaks as shown in the spectra were observed for the untreated and treated samples. This indicated that the structure of cellulose had not been damaged after the DMSO treatments.

The intensity decreased in the treated samples at 1731 cm^{-1}, which is attributed to either the acetyl and uronic ester groups of the hemicelluloses or the ester linkage of carboxylic groups of the ferulic and p-coumeric acids of lignin and/or hemicellulose [33]. This peak decreased due to the partial removal of hemicelluloses and more cleavage of lignin chains after DMSO treatment at 80 °C. A shoulder at 1640 cm^{-1} in the spectra is associated with the vibration of water molecules adsorbed by the noncrystalline regions in cellulose [31].

Consistent with the literature, the peaks observed at 1510 and 1456 cm^{-1} reflect the aromatic ring vibration and CH_3 asymmetric deformation of lignin, and a reduction or shift in this position is attributed to condensation reactions and/or the splitting of lignin aliphatic side chains [34]. The decrease in the intensity of these peaks could be a result of lignin loss under high temperature treatment.

DMSO treatments of jute fibres at 80 °C in 4 hours reduced the intensity of the 1376 cm^{-1} and 1249 cm^{-1} bands ,which was attributed to the cleavage and/or alterations of lignin C-H symmetric deformations and C-O stretching in the hemicelluloses acetyl groups [7, 33].

The DMSO process was carried out by soaking raw materials in solution for several hours, with noticeable changes being observed at 1726 cm^{-1} and bands relating to lignin aromatic ring vibrations at 1510 and 1456 cm^{-1}. The disappearance of these bands revealed that lignin was more largely removed in comparison to polysaccharides during the DMSO treatment. Thus we can conclude that jute fibres under high temperature treatment is rather an effective way to remove non-cellulosic materials, which coincides with the result of chemical & SEM analysis.

Single fibre bending test

Neurophysiological studies [35] showed that these nerve cells can be triggered by

Table 4. *Results of the compressing and bending characteristics test.*

Sample	Mean diameter, μm	Slenderness range	Mean slenderness	Equivalent bending modulus E, GPa
Raw	56.96	26.0-35.5	32.6	11.43
DMSO 40 °C 8 h	54.30	27.1-38.7	34.0	11.83
DMSO 60 °C 8 h	51.05	29.0-35.1	30.5	9.60
DMSO 80 °C 1 h	52.89	29.0-43.2	35.5	9.42
DMSO 80 °C 2 h	47.30	23.7-37.3	36.2	8.20
DMSO 80 °C 4 h	47.89	26.7-35.4	31.0	8.19
DMSO 80 °C 8 h	46.93	21.6-39.5	31.2	7.05

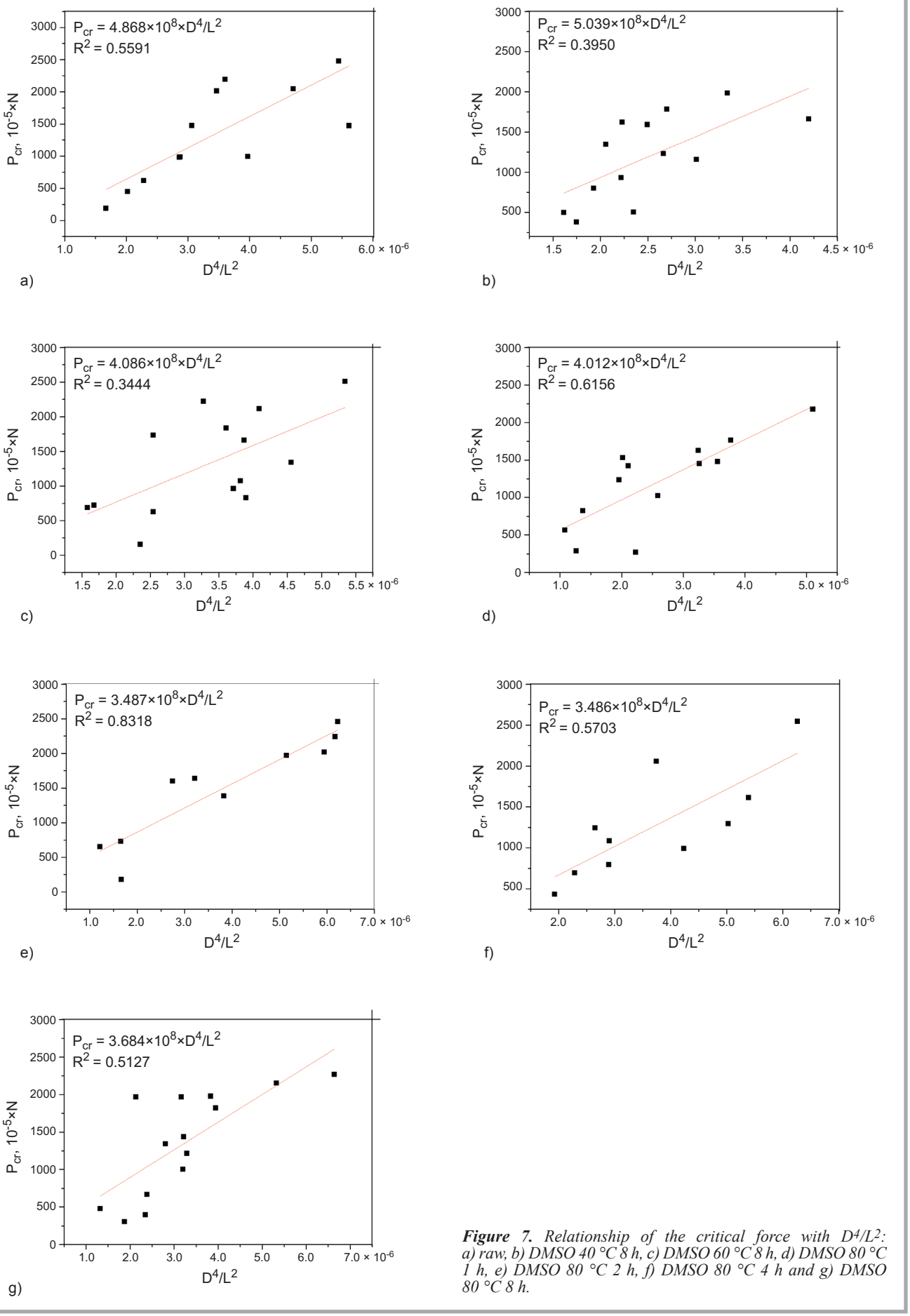

Figure 7. *Relationship of the critical force with D^4/L^2: a) raw, b) DMSO 40 °C 8 h, c) DMSO 60 °C 8 h, d) DMSO 80 °C 1 h, e) DMSO 80 °C 2 h, f) DMSO 80 °C 4 h and g) DMSO 80 °C 8 h.*

forces as low as 0.75 mN at the skin surface, although this threshold varies considerably from person to person. Coarser fibres in this range sometimes fulfil the criterion above (i.e., buckling load > 0.75 mN), leading to the prickle sensation. Test results show that the prickle force of jute fibres is greater than this value. The prickle problem always concerns jute fibre because of its stiff and high fibril orientation. The main evaluation parameter is the equivalent bending modulus, representing the softness and stiffness of fibres [36]. Bending is a very important mechanical property for jute fibres. Using FICBA, the bending properties of single fibres are quantified by calculating the equivalent bending modulus by measuring the protruding length and diameter of fibre needles as well as the critical force P_{cr} from Force-displement curves (***Figure 6,*** see page 30 and ***Figure 7***).

The critical force Pcr is directly proportional to D^4/L^2. According to formula 2, the equivalent bending modulus is

$$E_B = 1.01 \times 10^{-8} \frac{s}{K_B}.$$

Results of the equivalent bending modulus calculation are shown in ***Table 4***. The data measured showed that the equivalent bending modulus of jute fibre is softer after DMSO treatment than for raw, and that the quivalent bending modulus of the fibres decreased by 38.3%.

Jute is a multicellular fibre with a polygon shape of its cross section. Each unit cell of jute fibre is composed of small particles of cellulose surrounded and cemented together with lignin and hemicellulose. DMSO treatment tends to swell jute fibres [37], leading, in particular, to the swelling of the cross section, which makes the ratio of b/a greater, with the fibre automatically tending to bend in the easiest way. At the same time, there is the destruction of the mesh structure and splitting of fibres into finer filaments. The swelling of the cross section and removal of non-cellulosic cause the equivalent bending modulus to decrease dramatically, and the bending stiffness is decided by the equivalent diameter of the fibre and the bending modulus. It is significant that the wear behaviour of jute fabrics is enhanced when the bending stiffness is improved.

Conclusions

In the present study, the prickle properties of jute fibres after DMSO treatment at different conditions were examined. The DMSO process is an effective way for the removal of non cellulosic content such as wax, pectin and hemicellulose, based on chemical analysis. The eqivalent diameter was decreased under high temperature treatment. The ultimate cell of polygon shape was observed by SEM after 80 °C 8 h treatment. FTIR measurements of the fibres revealed that there was partial removal of hemicellulose and lignin. XRD showed that jute fibres at high temperature were much more crystalline than at low temperature. The relative crystallinity of the jute cellulose fibres reached approximately 73.64%. The equivalent bending modulus of the fibres decreased by 38.3% , which is indicative of the fibre becoming softer. DMSOwas used as the ideal solvent for solving the prickle problem under this investigation. It also has good effect on non-cellulosic component removal and increasing crystallinity, especially on the removal of wax. At the same time, the improvement of bending properties will also have potential for industrial application.

Acknowledgments

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