OPEN ACCESS
Saudi Journal of Engineering and Technology
Abbreviated Key Title: Saudi J Eng Technol
ISSN 2415-6272 (Print) |ISSN 2415-6264 (Online)
Scholars Middle East Publishers, Dubai, United Arab Emirates
Journal homepage: https://saudijournals.com

Original Research Article

Modification of Jute Fibre by Etherification Method for Diverse Textile Uses

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DOI: 10.36348/sjet.2022.v07i02.007 | **Received:** 17.01.2022 | **Accepted:** 24.02.2022 | **Published:** 28.02.2022

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Abstract

Etherification of jute fibre with butyl chloride was carried out at a temperature of 30^0C for a period of 2 hours in different solvents. Sodium hydroxide was selected as swelling agent for cheap and availability. Petroleum ether was used as a solvent in this research. The degree of substitution, the percent elongation, and bundle strength and moisture regain of the butyl substituted jute fibre was studied. It is observed that the bundle strength of the butyl substituted jute fibre decreases and percentage of moisture regain of the butyl substituted jute fibre decreases with the increases of substitution. The extensibility of the butyl substituted jute fibre increases up to about 10 times of its original value of 1% extension at break with the increase of substitution.

Keywords: Etherification, Modified jute fibre, Physico-mechanical properties, Physico-chemical properties, Textile fibres.

INTRODUCTION

Jute is called the golden fibre of Bangladesh. Jute plant produces fibre which are woody and course in nature having been suitable for making cordage, ropes and bags etc. from the very down of the human civilization (Ali *et al*., 2000). Partial removal of non-cellulosic components of jute fibre to improve its characteristics has recently been a subject of much interest. During microbial or chemical retting (Ali *et al*., 2000), only waxy and pectic substances are mostly removed, resulting in the ordinary fibre filament for commercial use. Lignin and other phenolic components are progressively degraded and released from their incrustation when the fibre is treated with chlorite or hypochlorite solution (Chattopadhyaya and Sarker, 1994; Salam et al., 1978) without appreciable improvement of textile properties except whiteness, which accelerates loss of strength (Klein. W, 1994; Jabber and Rahman, 1990). The non-fibrous carbohydrate materials are hydrolyzed by alkaline treatment, which impairs both the dry and wet strength of the fibre with immediate effect to such an extent that it may deteriorate its textile qualities (Hsieh, Y. L. *et al*., 1995; Kong, L. X.1996; Mukherjee, A. C, 1981). To obtain both lignin and hemicelluloses removed partially or wholly from the fibre one needs to have alternately two-stage treatments with chlorite and alkaline charges, which eventually accelerate degradation of structure and properties of the fibre (Ali *et al*., 2001). These components are not usually removed together from the fibre with a single-stage chemical charge even at the boiling temperature under normal atmospheric conditions (Ali *et al*., 1995). The changes in the chemical properties and the fine structure of jute fibres mercerized with liquid ammonia were studied (Islam *et al*., 2000). It is reported that the mercerization of jute fibre with liquid ammonia increased the extensibility of fibre without loss of strength and is therefore, more desirable swelling agent than sodium hydroxide. Sodium hydroxide also produces improvement in extensibility but causes loss of strength due to removable of part of hemicellulose (Islam *et al*., 2006). Esterification of jute fibre with a long chain fatty acid is reported by some workers (Islam *et al*., 2000). Lignified cellulose does not respond to organic base catalysed esterification reaction (Islam *et al*., 2006). However, partial esterification may be effected if the jute fibre is pretreated with alkali at concentrations between 5 and 15 percent and then treated with a fatty acid chloride (Pearson *et al*., 1962). The degree of acylation depends on the concentration as well as the total amount of alkali, the amount of moisture, the ratio of the acid chloride to the total alkali and the reaction time (Jame and Sugihara, 1953). Maximum acylation results when jute fibre is pretreated with 10 percent alkali and the

alkali cellulose containing 100 percent moisture is reacted for 3 hours with acid chloride at a molar ratio of about 0.5 of the total alkali present (Eliot and D. S, 1960; Roy and M. K, 1952). This paper contains the modification of jute fibre by etherification for textile uses of jute.

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.

METHODS

Preparation of n-butyl chloride

Concentrated Hydrochloric acid (200ml) was taken in a distilling flask (1litre) and andydrous zinc chlorides (340 gm) were added to it. A reflux condenser was fitted into the mouth of the distilling flask and a tube connected to an inverted funnel was attached to the top of the condenser .The funnel was dipped just below the surface of water (250ml) in a breaker. n-butyl alcohol (115 ml) was introduced into the distilling flask and refluxed gently on a wire gauge for 2 hours. The flask was kept inclined during the refluxing period (2 hours). After cooling it was distilled and the distillate was collected up to the temperature of 115^0C. The upper layer of the distillate was separated and mixed it with an equal volume of concentrated sulfuric acid and the mixture was refluxed gently for 30 minutes. The n-butyl chloride was distilled from the acid which passed over at 76^0C-79^0C. The distillate was washed successively with distilled water (100ml), 5 percent sodium hydroxide solution (50ml) and distilled water (100ml). It was then dried over anhydrous calcium chloride, filtered and distilled. The n-butyl chloride was collected at 75^0C-78^0C.The yield was 85%

Determination of alkoxyl group and degree of substitution:

Procedure

Moisture free sample (50gm) was introduced into the reaction acetic anhydride (3-4 drops), distilled phenol (5-6 drops) and a small bit of red phosphorus were added into the flask .After that freshly distilled hydrocloric acid (10 ml; d.1.7) was added into the reaction flask and attached immediately to the condenser with water supply. Carbon dioxide was passed through the reaction mixture at the rate of 2-3 bubbles per second and the flask was heated under micro burner, care was taken to ensure that the reaction mixture oiled only very gently during the heating period. The water was run out of the condenser during the last 45 minutes so that the alkoxyl group might pass over easily to the absorber. The absorption vessel was removed and the contents of the vessel were quantitatively transferred into the iodine flask. The vessel was washed well with distilled water and then with aqueous sodium acetate solution. The washings were collected into the iodine flask. The excess of bromine was destroyed by adding drop by drop formic acid until it was free from bromine (tested with methyl indicator, color persisted when bromine was completely destroyed). The sides of the flask were washed with distilled water, potassium iodide (1gm) was added into the flask followed by sulfuric acid (10ml, 10% w/v). The flask was stopped, shaked well, and allowed to stand for 5 minutes, and the liberated iodine was titrated with standard sodium thiosulphate solution (0.05N) using starch solution (1%) as indicator. The process was carried out identically for the control.

Calculation of Results

The percent alkoxyl was calculated from the following formula:

$$\% \text{ alkoxyl} = \frac{(V-b)\times N\times f}{W}\times 100$$

Where,

V = Volume (ml) of standard $Na_2S_2O_3$ used for treated sample

b = Volume (ml) of standard $Na_2S_2O_3$ used for untreated sample

N = Normality of standard $Na_2S_2O_3$ solution.

W =Weight (mg) of the sample.

F =factor.

$$\text{Degree of substitution (D.S)} = \frac{1.62\times \%\text{ alkoxy}}{M-0.14\%\text{alkoxy}}$$

Where, M =Molecular weight of the alkoxy group

Determination of moisture regain

The jute fibre was dried at 105^0C to constant weight and exposed to a standard atmosphere (62±2%) R.H.,Temperature (20 ± 2^0C) for 48 hours and again weighted carefully in the conditioned atmosphere. The percent moisture regain was calculated from the following relation.

$$R = \frac{b-a}{a}\times 100$$

Where,

R =Moisture regain percent.

a = Dry weight of the sample.

b = Moisture weight of the sample.

Treatment of jute fibre with sodium hydroxide solution at different times

Jute fibre was immersed in sodium hydroxide solution (10% strength, w/v) at 30^0C for different times. It was then washed successively with cold and hot water till alkali free. It was dried in air and stored in stopper bottle. Similar experiments were carried out with 20% alkali. The bundle strength, the percent

elongation and percent moisture regain of the treated fibre were determined.

Treatment of jute fibre with sodium hydroxide solution at different strengths

Jute fibre was treated with sodium hydroxide solution of (%w/v) at 30^0C for 2 hours. After washing and drying percent elongation, bundle strength and percent moisture regain of each treated sample were determined.

Treatment of jute fibre with n-butyl chloride alone at 30^0C at different times

Jute fibre was treated with n-butyl chloride solution of (the ratio of fibre to n-butyl chloride, 1:1 w/v) at 30^0C for different hours. It was then dried in air. The percent elongation, bundle strength, and percent moisture regain of each treated sample were determined.

Treatment of jute fibre with n-butyl chloride using different n-butyl chloride and fibre ratios

Jute fibre was pretreated with sodium hydroxide solution of 20% strength and then etherified with n-butyl chloride using different n-butyl chloride and fibre ratios. The time of etherification for each sample was 2 hours and the reaction temperature was 30^0C. The degree of substitution, percent elongation, bundle strength and percent moisture regain were determined.

Etherification of jute fibre with n-butyl chloride different solvents

Jute fibre was pretreated with sodium hydroxide solution of 20% strength and then etherified with n-butyl chloride (the ratio of fibre to butyl chloride, 1:1 w/v) at 30^0C for 2 hours in different solvents. The solvents were petrol (b.p. 80-120^0 C), ethyl alcohol (95%) and water. The reaction temperature was fixed at 100^0C when water was taken as a solvent.

Etherification of jute fibre with n-butyl chloride in petrol medium

To a steel vessel containing alkali fibre and petrol (volume ratio n-butyl chloride/petrol = 1:5), n-butyl chloride was added. The mouth of the vessel was kept air tight with a lid. The etherification was carried out at 30^0C for 2 hours. The contents of the vessel were stirred occasionally. After two hours the fibres were taken out of the vessel. It was then washed successively with cold and hot water till alkali free and dried in the air. The degree of substitution, percent moisture regain, percent elongation and bundle strength of the treated fibre were determined.

Etherification of jute fibre with n-butyl chloride in alcohol medium

Alkali fibre was taken in a steel vessel containing ethyl alcohol (volume ratio n-butyl chloride/ ethyl alcohol =1:5,), and butyl chloride. The reaction was performed at 30^0C for 2 hours. The mouth of the vessel was kept air tight with a lid. The whole mass of the steel vessel was agitated from time to time. The resulting jute fibre was washed first with cold water and then with hot water till alkali free. It was then dried in the air. The degree of substitution, percent moisture regain, percent elongation and bundle strength of the treated fibre were determined.

Etherification of jute fibre with n-butyl chloride in water medium

N-butyl chloride was treated with alkali fibre and 3% aqueous sodium hydroxide [volume ratio n-butyl chloride and aqueous sodium hydroxide (3%) =1:5] in a digester at a temperature of 100^0C and pressure 5kg/cm^2 for 2 hours. The digester rotated upside and downside two times a minutes. The jute fibre was then taken out of the digester. It was washed with cold water and then hot water till alkali free and dried in the air. The degree of substitution, percent moisture regain, percent elongation and bundle strength of the treated fibre were determined.

Measurement of Moisture Regain

The moisture regains for different treated and untreated samples were calculated from the relation

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

The conditioned weight is the weight of the sample after conditioning for 24 h at 63% relative humidity and 20^0C. The dry weight is the weight after dry in the sample in an oven at 105^0C until a constant weight was obtained.

Measurement of Bundle strength

The specimen of fibre filaments was placed as a flat bundle of about 1/4^{th} 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.

$$\textbf{Bundle strength (lb/mg)} = \frac{Breaking load\ in\ pound}{Wt.of\ \ fibre bundle in\ mg}$$

RESULTS AND DISCUSSION

Effect of time on swelling properties of jute fibre

Jute fibres were changed their intrinsic structure to a very limited extent during treatment of the n-butyl chloride solutions. Measurement of moisture regain, density and geometrical properties of the fibre indicated the changes in some structural features of the etherification. Jute fibre was treating with sodium hydroxide (10% and 20% in w/v) separately at different times so as to find out the effect of time on swelling of the fibres. The results of the elongation, bundle strength and moisture regain after etherification of jute fibre are shown in Table 1. From Table 1, it is seen that with

10% alkali treatment there is slightly increase in percentage elongation of the fibre while the bundle strength decreases with the increased time of swelling. This is compatible in that as the alkali action is increasing the compactness or crystalline is decreasing, hence the bundle strength is falling. The behavior of the 20% alkali treated jute fibre is following the same pattern as found with 10% alkali treated fibres. The percent elongation is increased from the first hour and continues to increase as the time of alkali treatment increases. This is due possibly to the increase in fibre diameter and cell wall thickness which sustain to increase the percent elongation.

Treatment of jute fibre with sodium hydroxide solution of different strength

The treatment of jute fibre with sodium hydroxide solution of different strength shows (Table-1) that with the increase of alkali concentration the percent elongation increases while the bundle strength decrease. This is quite obvious that with the increased concentration of alkali the intermolecular/inter chain hydrogen bonds are loosen at the expense of bundle strength and consequent increase in percent elongation. The moisture regains increases with the increase of alkali concentration which is due to increased amorphous character of the fiber.

Treatment of jute fibre with n-butyl chloride at 30^0 C in different times

The jute fibre was treated with n-butyl chloride at 30^0 C in different times. From Table–2 it is seen that time of treatment has practically no effect on elongation and bundle strength although there is a considerable improvement of moisture regain properties. This indicates that loss in moisture regain property is probably due to impregnation or add-on effect of butyl chloride on the fibre.

Effect of Jute fibre and n - butyl chloride ratios

The jute fibre was pretreated with 20% sodium hydroxide. Etherification reactions were then carried out with different proportions of fibre and butyl chloride in petrol medium at 30^0c. It is observed (Table-3) that the Degree of Substitution (DS) is maximum in the jute-butyl chloride 1:1 and 2:1 in two hours reaction time. It is seen that as the proportion of fibre to etherifying agent increases, the degree of substitution decreases. The bundle strength is remaining more or less unaffected. The moisture regains decreases (control has 8.9 moisture) with the increase of substitution. This is due to the increasing hydrophobic character of the substituents on the jute fibre.

Etherification of Jute fibre with n - butyl chloride in different solvents:

Alkali Jute fibre (20%) was etherified with butyl chloride in different solvents. The degree of substitution, percent moisture regains percent elongation and bundle strength was determined in each case. It is seen from the table 4 that the solvent like petrol, ethyl alcohol and water have marked effect on etherification and consequently on bundle strength, percent moisture regains and percent elongation. In the case of petrol as a solvent a high value of D.S, percent elongation and bundle strength in comparison with that of alcohol and water were obtained. By product formation took place in water and alcohol medium. For this reason a low D.S value was obtained in these media. In this study, three solvents were used to find out suitable one for industrial purpose. In the petrol medium encouraging result was obtained. So petrol is a better solvent than any other tried for. Hence subsequent investigation will base on petrol medium. From the findings, it may be concluded that modification of jute fibre was due to etherification which were improved the physico-chemical and physico-mechanical properties of jute fibre which are suitable for blends with others fibre and diverse use of jute in textile sectors.

Table 1: Effect of alkali concentration on jute fibre

Strength of NaOH (%w/v)	Elongation (%)	Bundle strength (g/Tex)	Moisture regain (%)
0	1	17.73	8.7
2	1	17.51	9.0
5	1.4	17.0	9.3
10	1.9	16.62	9.8
15	2.5	16.10	10.1
20	4.1	14.89	10.3
30	4.6	13.37	11.5

Table 2: Effect of the treatment of butyl chloride alone on elongation, bundle strength and moisture regain of jute fibre

Time of butyl chloride treatment (hour)	Elongation (%)	Bundle strength (g/tex)	Moisture regain (%)
1	1	17.70	6.7
2	1	17.1	6.4
4	1.5	16.8	6.1
8	1.6	16.3	5.7

Table 3: Etherification of jute fibre with butyl chloride using different fibre: butyl chloride ratios, the reactions time being 2 hours at 30^0C

Jute fibre -butyl chloride ratio	OC_4H_9 (%)	D.S	Elongation (%)	Bundle strength (g/tex)	Moisture regain (%)
1:1	0.98	0.021	9.7	13.21	5.6
2:1	0.97	0.021	9.5	13.10	5.8
3:1	0.90	0.020	9.2	12.92	5.9
10:1	0.79	0.017	8.6	12.70	6.3
15:1	0.71	0.015	8.4	12.51	6.5
20:1	0.60	0.013	8.2	12.43	6.7

Table 4: Effect of solvents on the degree of substitution, percent moisture regain, percent elongation and bundle strength of the butyl substituted jute fibre

Solvent	Reaction temp.0^0c	C_4H_9O (%)	D.S	Elongation (%)	Bundle strength(g/Tex)	Moisture regain (%)
Control	-	-	-	1	17.70	8.9
Petrol (b.p.80-120^0c)	30	0.98	0.021	9.75	13.27	5.1
Ethyl alcohol (95%)	30	0.21	0.004	7.20	9.73	7.5
Water	100	0.10	0.003	5.30	8.62	8.3

CONCLUSION

The etherification 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 etherified jute fibre compared to cotton and other textile fibres for fine spinning. The evaluation of these properties was regarded very significant towards improvement of spin able characters and other textile performances of the etherified jute fibre.

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