



Alkali pre-treatment of jute yarns for reinforcement in epoxy composites

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In this study, alkali treatment of jute yarns of different counts has been carried out for their subsequent application as reinforcing material in composite. The alkali treatment conditions have been optimised to improve the interaction between fibrous material and matrix. It is observed that the responses of different yarns are different. Other than the types of fibre, the structure of yarn has significant influence on the responses. The maximum tenacity is obtained at a lower alkali concentration for the lower count (4 lb/spyndle) and at a higher alkali concentration for the highest count (12 lb/spyndle). While for the medium counts of 6 lb/spyndle, 8 lb/spyndle, and 10 lb/spyndle, maximum tenacity is obtained at 10% NaOH concentration. XRD, FTIR, and SEM studies authenticate the partial delignification of the jute yarn and increase in the crystallinity after the alkali treatment.

Keywords: Alkali treatment, Epoxy composites, Jute yarn, Linear density, Tensile properties

1 Introduction

Natural fibres and natural fibre-based products are in great demand in recent years, parallel to man-made fibres like glass and carbon¹⁻³. Natural fibres are used chiefly due to low cost, light weight, easy processibility, renewable, recyclable, less fossil-fuel energy requirements, and the most importantly their high specific strength to weight ratio⁴⁻⁶. Growing demand for sustainable materials have led to shift the paradigm towards the natural fibres, resulting in the development of eco-friendly materials⁷⁻⁹.

Among all the natural fibres, jute has huge potential in textile and non-textile applications due to its porous, biodegradable, viscoelastic nature, besides its technical advantages like high tensile strength, high moisture sensitivity, low electrical insulation, and thermal properties¹⁰. Jute is majorly used in packaging¹¹, but it is also used in diversified technical textiles like jute geotextile¹²⁻¹³, jute-based polymer composites¹⁴⁻¹⁵, preparation of nanocrystals of cellulose from jute¹⁶, manufacture of activated charcoal¹⁷⁻¹⁸, etc.

The present work is to study the use of jute yarn as reinforcement material in polymer composites. However, for the fabrication of the composites, the inherent incompatibility between hydrophilic fibres and hydrophobic polymers is a major problem in achieving true reinforcement with the incorporation of

lignocellulose fibres into polymer matrixes¹⁹. To improve the quality of fibre-polymer matrix interface, surface modification of fibres is required to achieve maximum compatibility and thereby good adhesion. Many chemical treatments are used to modify the surface of the natural fibres such as alkali treatment, acetic acid treatment, silane treatment, bleaching, benzoyl peroxide treatment, potassium permanganate treatment, cellulose powder treatment, isocyanate treatment, and graft copolymerization²⁰⁻²¹ to increase the quality of the fibre-polymer matrix interface. Among all chemical treatments, alkali treatment of natural fibres is most commonly used²². This treatment removes the lignin, hemicellulose, wax, and other natural impurities, and as a result enhances the interaction between the polymer matrix and the alkali-treated fibre due to increasing reaction sites on the surface of the fibre as well as surface roughness²³⁻²⁶.

The optimization of alkali treatment of jute fibre prior to reinforcement in polymer composites at different process levels have also been reported. In many studies, optimum results are obtained at 5% NaOH concentration but at different time and temperature²⁴⁻²⁸. Sayeed and Paharia²⁹ done the alkali treatment of jute fibres at three different levels of NaOH concentration (0.5%, 4%, and 25%), and three different levels of time (24h, 30 min, and 20 min) respectively and demonstrate that the jute fibre treated with low NaOH concentration (0.5%) at more time shows the better tensile properties as compared to

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high concentrations. Bossunia *et al.*³⁰ done the optimization of alkali treatment on the jute fabric and found the best results at 50°C for 60 min treatment with low NaOH concentration (1%). Several researchers also found optimum conditions at high NaOH concentration with low time, and demonstrated that composite reinforced with alkali-treated jute fibre has good mechanical properties^{23,30-33}. Hence, it is observed that there are no definite optimum conditions for the alkali treatment of jute which can be used blindly.

Very limited study is reported on the alkali treatment of jute yarn and its use as reinforcement in composites. Studies based on textile-composites are primarily focused on the fibrous structure and interface between fibre and matrix³⁴. However, as the natural fibres have short length, hence the reinforcement material for the composite application needs to be understood in the form of long filaments or yarn³⁵. The comparative study on the mechanical properties of the jute fibre and jute yarn reinforced composites is done and it is observed that jute yarn reinforced composites show the maximum increment in tensile, flexural, and compressive properties as compared to jute fibre-reinforced composites³⁶. Therefore, it is very important to understand the influence of alkali treatment parameters (NaOH concentration, temperature, and time) on the properties of jute yarn for its successful application as reinforcement material in composites.

The focus of current work is to study the effect of alkali treatment on various counts of jute yarn at different NaOH concentrations to achieve a better reinforcement material for polymer composites.

2 Materials and Methods

2.1 Materials

Single-ply yarns of five different counts were prepared from TD₃ grade jute. All the yarns were manufactured from ICAR-NINFET, West Bengal, India. Sodium hydroxide (NaOH) was supplied by Sd-fine Chem. Ltd. for alkali treatment of jute yarn.

2.2 Experimental Plan

As the linear density of the yarn increases, its diameter also increases. For same weight percentage of reinforcement, lower count yarns are supposed to provide more number of yarns and hence more area for the interaction with the resin. Keeping this in mind, lower counts of yarns are primarily targeted for application as reinforcing elements. Also, the tenacity

of 6 lb/spyndle and 10 lb/spyndle is somewhat same while 4 lb/spyndle jute yarn has the minimum tenacity. So, 6 lb/spyndle jute yarn was selected for optimisation treatment. Moreover, as the fibre used in all count is same, similar trends in change of properties is expected for other counts (4, 8, 10 and 12 lb/spyndle) as well.

Based on the literature, the treatment parameters were selected and alkali treatment was performed using 3 variables and 3 levels Box and Behnken experimental design for 6 lb/spyndle yarn. The three independent variables; viz concentration, temperature, and time were used in three different levels to study the effect of parameters on tensile properties and weight loss percentage. Variables and their levels are shown in Table 1.

2.3 Alkali Treatment

The alkali treatment was carried out on jute yarn in lea form of 120 yds having 1:20 ML ratio. Before the alkali treatment, the samples were oven-dried at 100°C for 2h. After the treatment, the samples were washed with water until residual NaOH was removed from the samples to ensure the complete neutralization of the samples. The samples were finally dried at room temperature (25°C) for 24h followed by oven drying at 100°C for 2h. After drying, the samples were weighed, and weight loss (W) percentage was calculated using the following formula:

$$W \% = \frac{W_1 - W_2}{W_1} \times 100 \quad \dots (1)$$

where W_1 is the initial oven-dry weight of the jute yarn; and W_2 , the weight of jute yarn after the alkali treatment.

2.4 Characterization Techniques

2.4.1 Tensile Testing

The tensile testing of treated and untreated jute yarns was conducted according to the ASTM D2256 using a Universal tensile testing machine (Aimil, Qmat 5.01). Breaking force, tenacity, elongation percentage, and initial modulus of jute yarn were calculated. Fifty tests of each sample were carried out.

Table 1 — Variables and levels

Variables	Levels		
	Low (-1)	Medium (0)	High (+1)
NaOH concentration, %	2	4	6
Temperature, °C	20	40	60
Time, h	2	4	6

2.4.2 XRD, FTIR and SEM Studies

The X-ray diffraction (XRD) analysis of the treated and untreated jute yarns was performed in an EMPYREAN diffractometer, with Cu radiation ($\lambda = 1.544 \text{ \AA}$) at room temperature (25°C). The scattered radiation was detected in a 2θ range of 5°- 40°. The crystallinity index (CI) was estimated using the following equation:

$$\text{Crystallinity \%} = \frac{A_c}{A_c + A_a} \times 100 \quad \dots (2)$$

where A_c and A_a are the area under the crystalline peak; and the area under the amorphous region in the XRD curve respectively^{37,38}.

FTIR spectra of the treated and untreated jute yarn were obtained using Alpha II (Bruker) FTIR in the 400-40000 cm^{-1} wavenumber range.

SEM analysis is carried out by JEOL 7800 (USA) to analyze the surface characteristics of the alkali and non-treated jute yarn at a magnification of $\times 500$.

3 Results and Discussion

3.1 Properties of Untreated Jute Yarns

The properties of the different counts of jute yarn are shown in Table 2.

It is clearly seen that with the increase in the count, the elongation% and breaking force increase, but tenacity and initial modulus show a different trend. Breaking force mainly depends on the number of fibres in the cross-section of the yarn and cohesion between the fibres. As the count increases, the number of fibres in the cross-section of the yarn increases and load acting on the individual fibre decreases, which has a positive effect on the breaking force. With the increase in count, TPI decreases, leading to decrease in the cohesion between the fibres

in the yarn. This has a negative effect on the breaking force. It seems that for the types of yarn used in the study, the first effect (increase in number of fibres in the cross-section) dominates over the second effect (reduction in cohesion between the fibres). Resultant effect is an increase in the breaking force with increase in count. Elongation of yarn is generally affected by the inter fibre movement and cohesion between the fibres in the yarn. As the twist per inch decreases, the cohesion between the fibres decreases and inter fibre movement in the yarn increases, where fibres tend to slip rather than break. As a result, elongation% increases with increase in the count³⁹.

Maximum tenacity is observed for the 8 lb/spyndle and minimum for the 4 lb/spyndle. While for the other counts the tenacity is more or less same. Tenacity is the ratio of breaking force and linear density of the yarn⁴⁰. With change in the count, breaking force changes. The TPI of the yarns for the different counts are also different. For 4 lb/spyndle yarn the minimum breaking force may be due to less number of fibres in the cross-section although the more TPI signifying more cohesion between the fibres in the yarn. With increase in TPI, obliquity effect may also come into play and a resultant effect of all these factors may lead to minimum tenacity value for the 4 lb/spyndle yarn.

With increase in count till 8 lb/spyndle, there is consequent increase in the breaking force of the yarn. When going from 4 lb/spyndle to 6 lb/spyndle and from 6 lb/spyndle to 8 lb/spindle, the rate of change in breaking force is higher as compared to rate of change in linear density. This leads to increase in the tenacity of the yarn when going from 4 lb/spyndle to 8 lb/spyndle. With further increase in the count change in the linear density outweighs the improvement in

Table 2 — Properties of untreated jute yarn

Count lb/spyndle	Count tex	TPI	Twist factor $\text{tex}^{1/2} \times \text{turns/cm}$	Diameter mm	Quality ratio	Moisture regain, %	Initial modulus cN/tex	Breaking force, cN	Tenacity cN/tex	Elongation %
12	413.4 (6.51)	3.75	30.02	0.7791	91.001	13.049	449.78 (11.05)	4857.50 (17.35)	11.75 (17.35)	2.553 (14.62)
10	344.5 (6.74)	4.00	29.23	0.7101	94.717	13.228	581.52 (10.00)	4213.24 (14.94)	12.23 (14.94)	2.298 (13.45)
8	275.6 (6.89)	4.28	27.97	0.6837	109.587	13.073	708.41 (10.17)	3899.74 (13.95)	14.15 (13.95)	2.025 (12.92)
6	206.7 (6.98)	5.50	31.13	0.5489	94.485	13.183	614.147 (8.07)	2521.74 (19.32)	12.20 (19.32)	1.810 (15.14)
4	137.8 (7.01)	6.00	27.73	0.4896	62.035	13.476	531.442 (10.07)	1103.78 (21.15)	08.01 (21.15)	1.620 (15.47)

Values in parentheses represent CV% of corresponding values.

the breaking force. As a result, the tenacity again starts showing downward trend. Similar to tenacity, initial modulus also increases up to 8 lb/spynidle and then it starts decreasing. The reason may be, interaction between the number of fibres, cohesion between the fibres, i.e. TPI and arrangement of fibres in the yarn cross-section. With increase in the count, number of fibres in the cross-section increases. Hence, more stress is required to produce an equivalent level of extension which gives rise in the initial modulus. Also, with increase in the count, TPI decreases and then arrangement of fibres in the cross-section of the yarn is more parallel to the yarn axis. Hence, the cohesion between the fibres reduces, which has negative effect on the initial modulus.

3.2 Alkali Treatment of 6 lb/spynidle Jute Yarn

Responses for all 17 runs of the box-behnken design are shown in Table 3.

The best-fit regression equation for all responses are developed using the backward elimination method on statistical software using a box-behnken design (Table 4). The accuracy of a model is determined by the value of R² which is very close to 1, and hence

can be used to study the effect of different parameters on all the responses^{41,42}.

3.2.1 Effect of Alkali Treatment on Weight Loss%

Figure 1 shows the effect of NaOH concentration and temperature on the weight loss percentage. With increase in NaOH concentration and temperature, the weight loss percentage also increases. The alkali

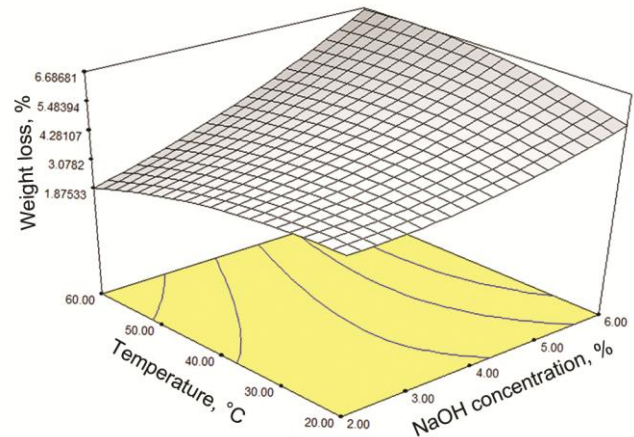


Fig. 1 — Effect of NaOH concentration and temperature on weight loss %

Table 3 — Process parameters and responses

Run	Variables			Responses			
	NaOH concentration %	Temperature °C	Time h	Weight loss %	Tenacity cN/tex	Initial modulus cN/tex	Elongation %
1	-1	0	-1	3.35	11.92	464.90	2.466
2	0	-1	-1	3.99	11.87	480.80	2.450
3	0	0	0	4.62	10.52	430.70	2.495
4	+1	+1	0	6.64	13.75	494.80	2.795
5	0	0	+1	2.95	11.40	471.00	2.376
6	+1	0	+1	6.89	13.66	502.00	2.750
7	0	+1	+1	3.28	12.01	410.70	2.780
8	0	-1	+1	4.12	12.53	526.00	2.320
9	-1	+1	0	2.01	11.58	481.50	2.260
10	-1	-1	0	3.99	12.23	500.20	2.345
11	0	0	0	4.42	10.81	446.10	2.501
12	0	0	0	4.69	11.01	459.80	2.499
13	0	0	0	4.72	10.99	438.70	2.501
14	0	0	0	4.52	11.11	411.50	2.498
15	+1	-1	0	5.50	12.16	542.56	2.290
16	+1	0	-1	6.00	12.30	481.90	2.537
17	0	+1	-1	4.25	11.14	430.80	2.467

Table 4 — Regression equation for various responses

Properties	Regression equations	R ²
Weight loss, %	4.51+1.59A-0.18B+0.40A ² -0.48B ² +0.78AB	0.952
Tenacity, cN/tex	10.89+0.59A-0.039B+0.30C+0.99A ² +0.55B ² +0.4C ² +0.56AB+0.47AC	0.931
Initial modulus, cN/tex	437.34+12.96A-28.97B+42.64A ² +24.76B ²	0.819
Elongation, %	2.50+0.11A+0.11B+0.038C-0.029A ² -0.057B ² +0.062C ² +0.14AB+0.076AC+0.11BC	0.996

A– Concentration in %, B– Temperature in °C and C– Time in h.

treatment process involves partial removal of lignin and hemicellulose content which reduces the weight^{25,43}. As shown in Table 3, maximum weight loss of 6.89% is achieved when treated with 6% NaOH concentration for 6h at 40°C treatment condition, and minimum weight loss of 2.01% is achieved when treated with 2% NaOH concentration for 2h at 60°C.

It is mentioned earlier that with an increase in temperature and time, fibre swells and provides more surface area, thus increasing the rate of the delignification process⁴⁴. Similarly, as the NaOH concentration increases, weight loss% increases due to the increase in the rate of a hydrolysis reaction.

3.2.2 Effect of Alkali Treatment on Tensile Properties

The effect of NaOH concentration and temperature on the tensile properties of the jute yarn when treated for 4h is shown in Fig. 2. It is observed that with an increase in NaOH concentration, there is increase in the tenacity and elongation%, while initial modulus shows a different trend. After the alkali treatment, there is defibrillation of the yarn as well as partial removal of the lignin and hemicellulose, which act as a cementing material¹⁹, and hence breaking load decreases. Also, alkali treatment reduces the weight and diameter of the yarn, consequently decreases linear density. With the increase in NaOH concentration, there is marked reduction in the linear density which outweighs the effect of breaking force that decreases marginally. As a result, with increase in NaOH concentration, tenacity of the jute yarn increases. Elongation % also increases after the alkali treatment, due to the shrinkage of fibre as well as yarn, which causes waviness in the yarn. Also, with the leaching of hemicellulose and lignin material, which act as binding material, disorients the structural arrangement of cellulose⁴⁵. Hence, this increases the inter fibres movement in the yarn, as a result elongation% increases.

The alkali treatment is done in a slack condition which causes the shrinkage in yarn, hence disorientation of molecules in the fibre and yarn takes place, resulting in reduction in the initial modulus. But there are slight changes in the initial modulus with change in the NaOH concentration. This may be due to the variation in inherent properties of material.

3.3 ANOVA Analysis

The alkali treatment is promoting weight loss and change in the tensile properties of the jute yarn. It is clearly seen from the surface plots of tenacity, initial modulus and elongation % (Fig. 2) with respect to

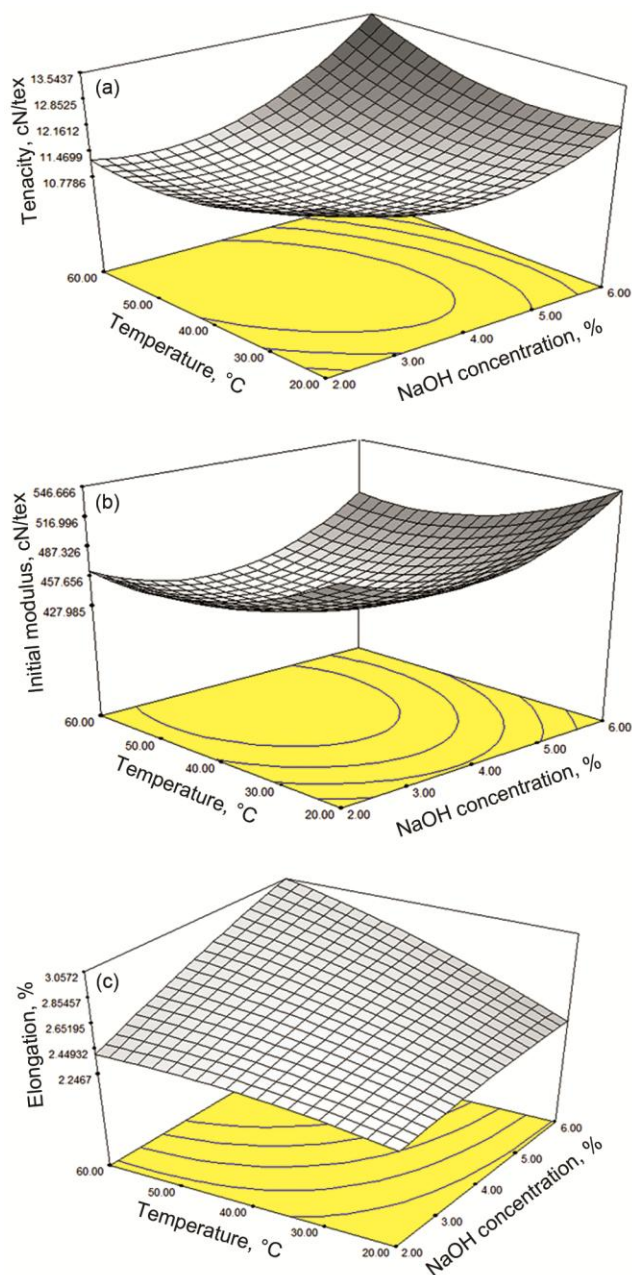


Fig. 2 — Effect of NaOH concentration and temperature on (a) tenacity, (b) initial modulus, and (c) elongation % at 4h

NaOH concentration and temperature that the tenacity is showing an increasing trend. The conditions for the alkali treatment as selected according to the literature review²⁴⁻³³ do not produce maxima and it is evident that the maxima lies beyond the range of alkali concentrations used. ANOVA also confirms that the NaOH concentration has the highest contribution as compared to the temperature and time and has a significant effect on all responses as shown in the

Table 5. Therefore, subsequent experiment is planned by further enhancing the alkali concentration to identify the concentration producing maximum value of tenacity. Accordingly, three different alkali concentration, i.e. 8%, 10% and 12%, are used to see the effect on tensile properties, keeping the temperature and time constant (60°C and 6 h).

3.4 Effect of NaOH Concentration on Different Counts of Jute Yarn

The effects of alkali concentration on the tensile properties of five different jute yarns are shown in Table 6.

It is observed that 6 lb/spyndle, 8 lb/spyndle and 10 lb/spyndle jute yarns show the same trend, where the maximum tensile properties are achieved at 10% NaOH concentration. On the other hand, for the lower count (4 lb/spyndle), the maximum tensile properties are achieved at 8% NaOH concentration. Hence, lower concentration of alkali (6%) is also tested to study the maximum tensile properties for 4 lb/spyndle jute yarn. For the higher count (2 lb/spyndle), further enhancement of alkali concentration is also found necessary to achieve the same and the maximum improvement in tensile properties. For 12 lb/spyndle jute yarn, maximum improvement is achieved at 14% NaOH concentration. It is also observed that as the concentration of alkali increases from 8% to 10% for 4 lb/spyndle yarn, there is marked change in the weight loss percentage and shrinkage, consequently breaking force of the yarn decreases. As a result, tenacity of the yarn decreases at 10% NaOH concentration. Similarly, for the medium counts (6, 8 and 10 lb/spyndle), after the 10% NaOH concentration there is noticeable change in the weight loss percentage, consequently breaking force decreases and tenacity shows the down trend. For the highest count (12 lb/spyndle) yarn, after the 12% NaOH concentration there is significant change in weight loss and shrinkage percentage, consequently breaking

force reduces. Hence, at 12% NaOH concentration, maximum tenacity is achieved for 12 lb/spyndle yarn. Also, the moisture regain% of jute yarn increases after the alkali treatment which may be due to the removal of lignin and hemicellulose, rendering the cellulose more accessible to moisture between the yarn⁴⁶.

3.5 Characterization of Treated and Untreated Jute Yarn

Figure 3 shows the diffraction patterns of alkali-treated and non-treated jute yarn. Two well-defined reflection peaks for cellulose I at around 20° and 23.5° are observed in both the curves. The highest peak at 2θ between 22° and 24° corresponds to the I₂₀₀ plane lattice, which represents the crystalline material, and the 2θ at about 17°-18° corresponds to the I_{non-cr} plane, which represents the diffraction of non-crystalline material⁴⁷. The crystallinity index is calculated to be about 58.06% and 59.93% for the untreated and alkali-treated respectively. The increase in the crystallinity index of treated yarn as compared

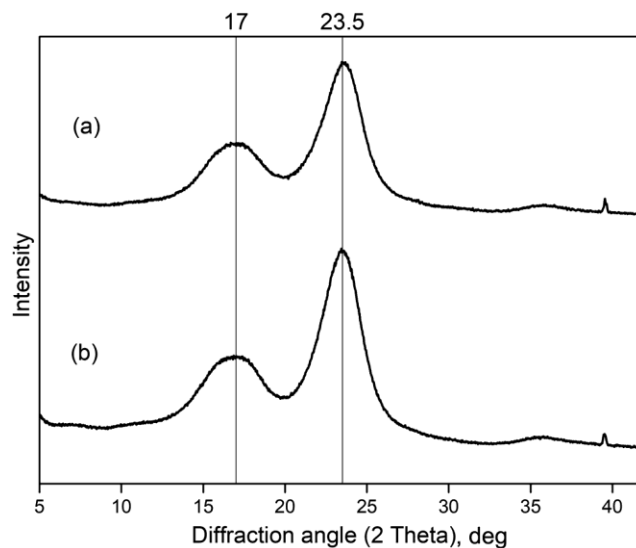


Fig. 3 — XRD pattern for (a) alkali-treated and (b) untreated jute yarns

Table 5 — ANOVA analysis of processing parameters

Factors	Weight loss, %			Tenacity, cN/tex			Initial modulus, cN/tex			Elongation %		
	Sum of squares	F value	Contribution %	Sum of squares	F value	Contribution %	Sum of squares	F value	Contribution %	Sum of squares	F value	Contribution %
NaOH concentration, %	20.26	180.10*	82.59	2.81	24.35*	22.46	1343.17	3.86*	6.85	0.099	435.90*	26.05
Temperature, °C	0.25	0.24	1.03	0.012	0.10	5.59	6714.09	19.28*	34.23	0.093	408.87*	24.47
Time, h	-	-	-	0.70	6.09	0.70	-	-	-	0.012	51.64	3.16
Error	0.06	-	0.24	0.22	-	1.86	1294.83	-	6.60	0.005	-	0.13

*Significant level.

Table 6 — Tensile properties of Jute yarn of different counts at different alkali concentration

Count lb/spynde	Alkali conc, %	Weight loss, %	Shrinkage %	Count decrease, %	Moisture regain, %	Breaking force, cN	Tenacity cN/tex	Initial modulus cN/tex	Elongation %	Strength: weight ratio
4	6	3.22	2.74	0.96	23.838	1142 (26.24)	8.20 (26.24)	350.7 (16.62)	1.713 (16.94)	2.546
	8	3.89	3.04	1.19	23.252	1022 (29.68)	9.05 (29.68)	371.7 (16.82)	1.771 (17.19)	2.326
	10	5.96	3.77	2.27	20.587	966 (34.85)	7.17 (34.85)	346.4 (16.89)	1.537 (19.32)	1.203
	12	8.89	3.77	5.33	18.437	964 (29.22)	7.05 (29.22)	310.3 (16.40)	1.513 (13.93)	0.793
6	8	7.20	3.62	2.89	23.759	2511 (19.48)	15.29 (19.48)	433.6 (14.17)	2.524 (12.03)	2.124
	10	6.97	3.77	3.32	22.887	2406 (23.60)	15.86 (23.60)	459.8 (15.41)	2.615 (14.17)	2.275
	12	7.11	4.78	3.45	22.711	2264 (19.85)	13.21 (19.85)	411.8 (19.85)	2.281 (12.12)	1.858
8	8	7.20	5.36	1.94	23.232	3891 (15.65)	14.76 (15.65)	455.0 (12.00)	2.533 (12.38)	2.050
	10	7.63	5.51	2.25	22.942	3784 (13.75)	15.45 (13.75)	464.3 (13.44)	2.553 (10.13)	2.024
	12	8.11	6.09	2.55	20.669	3452 (16.95)	15.05 (16.95)	457.4 (14.08)	2.484 (12.24)	1.856
10	8	7.60	5.94	1.77	23.491	4209 (13.33)	14.18 (13.33)	375.4 (16.13)	2.725 (11.24)	1.866
	10	7.16	6.23	1.99	22.979	4185 (13.25)	15.77 (13.25)	396.0 (16.07)	3.015 (13.65)	2.203
	12	8.47	6.52	2.08	21.086	3840 (17.29)	12.22 (17.29)	336.0 (18.43)	2.736 (15.88)	1.443
12	8	4.32	5.65	1.41	22.998	4753 (11.38)	15.11 (11.38)	389.8 (12.87)	2.951 (10.61)	3.498
	10	4.52	5.22	0.73	22.193	4651 (14.50)	15.69 (14.50)	377.7 (15.71)	3.031 (10.64)	3.471
	12	5.29	5.94	1.37	22.114	4049 (13.33)	15.82 (13.33)	398.1 (12.32)	2.978 (11.61)	2.991
	14	7.07	6.23	2.06	19.445	3724 (12.58)	17.05 (12.58)	427.4 (12.28)	3.498 (11.95)	2.412
	16	8.65	6.52	2.646	18.988	3471 (9.45)	16.06 (9.45)	333.4 (11.72)	3.283 (7.73)	1.857

Values in parenthesis represent CV% of corresponding values.

to the untreated yarn can be explained by the removal of amorphous non-cellulosic compounds induced by the alkali treatment¹⁶.

To investigate the structural changes in fibres during the chemical treatment, FTIR spectroscopy is used (Fig. 4). In both untreated and alkali-treated spectra, three main bands are observed that are related to cellulose such as C-O-C band at 1027 cm⁻¹, C-H band at 2910 cm⁻¹ and -OH band at 3310 cm⁻¹ (ref. 48). The acetyl or uronic ester groups of hemicellulose normally appear in the region of 1700-1740 cm⁻¹ (ref. 16) and it is observed that the band at 1728 cm⁻¹ is higher in the case of untreated jute yarn as compared to that in the alkali-treated jute yarn which represents

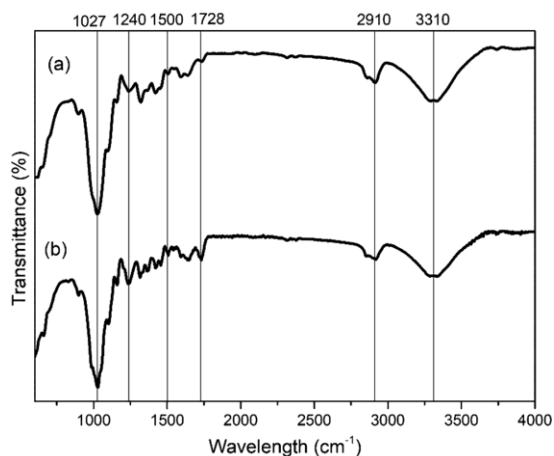


Fig. 4 — FTIR spectra of (a) alkali-treated and (b) untreated jute yarns

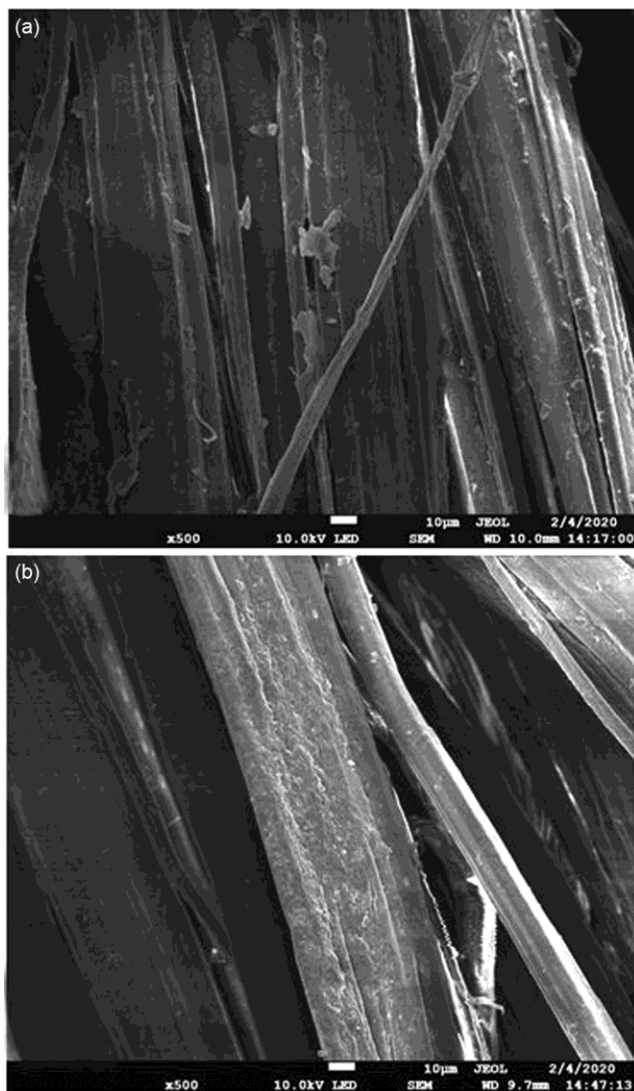


Fig. 5 — SEM images of (A) non-treated and (B) alkali-treated jute yarns

the partial removal of hemicellulose after the treatment. Also, peaks at 1240 cm^{-1} (C-O of guaiacyl ring) and $1500\text{-}1645\text{ cm}^{-1}$ (aromatic skeletal vibration) represent the presence of lignin in the fibres⁴⁹. Figure 4 also shows that in the case of alkali-treated jute yarn there is a decrease in the intensity of the lignin band, which results in the partial delignification of jute yarn after the alkali treatment.

The SEM images of treated and untreated jute yarn are shown in Fig. 5. It is observed that the surface of the untreated jute yarn shows the presence of wax and impurities on the surface of the fibre bundles, and bundles of individual fibres are bound together by lignin. After the alkali treatment, the surface of the yarn seems to be clean.

4 Conclusion

Alkali treatment of jute yarn leads to weight loss as well as changes in the tensile properties of the yarn. For subsequent application of the yarn as reinforcement in composite, the target yarn should have high strength: weight ratio. To achieve this, the alkali treatment condition is optimised. During the process of optimization, it is observed that the responses of the yarns towards alkali treatment are different and there is no fixed optimum condition which can be used blindly for all the yarns.

Final property of the yarn depends primarily on the weight loss% and reduction in breaking force along with other change, like shrinkage, change in diameter leading to a change in twist, cohesion between the fibres in the yarn, etc.

From the experimental observations, it is found that for the type of yarns and the conditions for treatment used, in case of finer yarns best result is achieved at lower alkali concentration and in case of coarser yarns higher concentration is necessary.

Results of the XRD, FTIR spectra and SEM images of the alkali treated and un-treated jute yarns are in line with the previously reported literature, confirming an increase in the crystallinity as well as partial delignification of the alkali-treated jute, leading to modification of the fibre surface.

References

- 1 Mahir F I, Keya K N, Sarker B, Nahiun K M & Khan R A, *Mater Eng Res*, 1 (2019) 88.
- 2 Ryszard K M, Maria M T, Malgorzata M & Jorge B B, *Mol Cryst Liq Cryst*, 556 (2012) 200.
- 3 Begum K & Islam M A, *Res J Eng Sci*, 2 (2013) 46.
- 4 Buitrago B, Jaramillo F & Gómez M, *J Nat Fibers*, 12 (2015) 357
- 5 Malik D S, Jain C K & Yadav AK, *Appl Water Sci*, 7 (2017) 2113
- 6 Salman S D, *J Ind Text*, 51 (2019)1.
- 7 Bajwa D S & Bhattacharjee S, *J Nat Fibers*, 13 (2016) 660
- 8 Thyavihalli Girijappa Y G, Mavinkere Rangappa S, Parameswaranpillai J & Siengchin S, *Front Mater*, 6 (2019) 1.
- 9 Madhu P, Sanjay M R, Senthamaraiannan P, Pradeep S, Saravanakumar S S & Yogesha B, *J Nat Fibers*, 16 (2019)1132
- 10 Samanta A K, Mukhopadhyay A & Ghosh S K, *Handbook of Natural Fibres* (Elsevier Ltd),2020, 49
- 11 Banerjee P K. *Environmental Textiles from Jute and Coir* (Elsevier Ltd), 2020, 621.
- 12 Datta U, *J Nat Fibers*, 4 (2007) 67.
- 13 Shukla S, Tiwari R P, Rajbhar V & Mittal A. *Geotechnics for Transportation Infrastructure* (Springer Singapore), 2019, 295
- 14 Singh H, Singh J I P, Singh S, Dhawan V & Tiwari S K, *Mater Today Proc*, 5 (2018) 28427.

- 15 Balcioglu H E, *Res Eng Struct Mater*, 5(2019) 213.
- 16 Thomas M G, Abraham E, Jyotishkumar P, Maria H J, Pothan L A & Thomas S, *Int J Biol Macromol*, 81 (2015) 768.
- 17 Ghosh R K, Ray D P & Chakraborty S, *Int J Environ Anal Chem*, (2020), 1.
- 18 Chen W, Zhang S, He F, Lu W & Xv H, *J Mater Cycles Waste Manag*, 21 (2019) 315.
- 19 Saha P, Chowdhury S, Roy D, Adhikari B, Kim J K & Thomas S, *Polym Bull*, 73 (2016) 587.
- 20 Ferreira D P, Cruz J & Fangueiro R, *Green Composites for Automotive Applications* (Elsevier), 2019,3.
- 21 M.R. S, Siengchin S, Parameswaranpillai J, Jawaid M, Pruncu C I & Khan A, *Carbohydr Polym*, 207 (2019) 108.
- 22 Sahu P & Gupta M K, *J Mater Des Appl*, 0 (2019) 1.
- 23 Kapatel P M, *J Nat Fibers*, (2019) 1.
- 24 Manikandan N, Morshed M N & Karthik R, *Am J Curr Org Chem*, 3 (2017) 9.
- 25 Sudha S & Thilagavathi G, *J Text Inst*, 107 (2016) 691.
- 26 Mukherjee A, Ganguly P K, Sur & D D, *J Text Inst*, 84 (1993) 348.
- 27 Ray D, Sarkar B K, Rana A K & Bose N R, *Compos Part A*, 32 (2001) 119.
- 28 Kumar P, Tiwari M & Elizabeth M, *Trans Indian Inst Met*, 73 (2020) 1573.
- 29 Sayeed M M A & Paharia A, *J Text Inst*, 110 (2019) 1588.
- 30 Bossunia M T I, Poddar P & Hasan M M, *J Mater Sci Eng*, 5 (2016).
- 31 Gassan J & Bledzki A K, *Compos Sci Technol*, 59 (1999) 1303.
- 32 Owen M, *Int J Fiber Text Res*, 4 (2014) 32.
- 33 Lakshmanan A, Ghosh R K, Dasgupta S, Chakraborty S & Ganguly P K, *J Ind Text*, 47 (2018) 640.
- 34 Messiry M E I, *Alexandria Eng J*, 52 (2013) 301.
- 35 Shah D U, Schubel P J & Clifford M J, *J Compos Mater*, 47 (2012) 425
- 36 Zakaria M, Ahmed M, Hoque M & Shaid A, *J Nat Fibers*, (2018) 1.
- 37 Ioelovich M Y & Veveris G P, *J Wood Chem*, 80 (1987) 72
- 38 Correia C A, Oliveira L M de & Valera T S, *Mater Res*, 20 (2017) 466
- 39 Shao X, Qiu Y & Wang Y, *J Text Inst*, 96 (2005) 61
- 40 Goswami B C, Martindale J G & Scardino F L, *Textile Yarns; Technology, Structure, and Applications* (John Wiley and Sons), 1995.
- 41 Emmanuel E, Yong L L, Asadi A, & Anggraini V, *J Nat Fibers*, 00 (2020) 1.
- 42 Salih A A, Zulkifli R & Azhari C H, *J Nat Fibers*, April (2020) 1.
- 43 Shaha S K, Dyuti S, Ahsan Q & Hasan M, *Adv Mater Res*, 264 (2011) 1922.
- 44 Mishra L, Basu G & Samanta A K, *Fibers Polym*, 18 (2017) 357.
- 45 Goda K, Sreekala M S, Gomes A, Kaji T & Ohgi J, *Compos Part A Appl Sci Manuf*, 37 (2006) 2213.
- 46 Majumder A, Samajpati S, Ganguly P K, Sardar D & Gupta P C D, *Text Res J*, 50 (1980) 575.
- 47 Jabbar A, Militký J, Wiener J, Kale B M, Ali U & Rwawiire S, *Compos Struct*, 161 (2017) 340.
- 48 Delgado-Aguilar M, Oliver-Ortega H, Alberto Méndez J, Camps J, Espinach FX & Mutjé P, *Int J Biol Macromol*, 116 (2018) 299.
- 49 Singh H & Chatterjee A, *Cellulose*, 27(2020) 2555.