Paper ID: 450

Influence of Alkali Treatment (With and Without Tension) on the Tensile Properties of Unidirectional (UD) Jute Polyester Composites

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Abstract

Jute is a cellulose-based fibre and cellulose has high mechanical properties. However, some other important components of jute such as hemicellulose and lignin have low mechanical properties. Hence removal of these elements may improve the properties of jute. Alkali treatment is a good way to remove hemicellulose and lignin from jute. In this work jute yarn is dipped in 1%, 3% and 5% (w/w) alkali solution with and without tension to observe the influence of these treatments on the Unidirectional (UD) jute polyester composites. Raw jute yarn and alkali-treated yarns are used to manufacture composites using resin transfer moulding. Tested results revealed that the tensile strength of composites made of alkali-treated yarn with tension shows a significantly higher magnitude with respect to composites made of alkali-treated yarn of the same concentration and without tension. At the same time composite plaque made of 5% concentrated alkali-treated yarn with tension shows significantly higher tensile strength with respect to that of composites made of raw yarn.

Keywords: Composites, alkali treatment, tensile properties.

1. Introduction

Cellulose is the chief element of jute fibre [54] which has very high mechanical properties [1]. However low tensile properties of jute fibres (with respect to cellulose) e.g. 7.2 [2] – 38.9 [3] GPa tensile modulus and 187 [2] – 800 [4] MPa tensile strength were observed by many researchers. Various natural defects [5, 6], processing faults and the chemical composition play a major role to reduce the tensile properties. For example, hemicellulose, the second major component of jute fibre [1] possesses 5-8 GPa tensile modulus and the third important component lignin has 2.5-3.7 GPa tensile modulus and 25-75 MPa tensile strength [7] which are much lower than the properties of cellulose. Hence, the removal of hemicellulose and lignin improves the tensile properties of cellulose fibres [8]. Thus, the intention of this research work is to remove lignin and hemicellulose to understand the effect of removing these components on the properties of UD jute polyester composites. Alkali treatment is a well-established technique to remove hemicellulose, lignin, pectin, wax and other oily substances of plant fibres [9, 10, 11]. Alkali treatment is performed in an aqueous medium and treatment of jute yarn in aqueous solution of alkali is performed with and without tension to observe the effect of this treatment condition on the mechanical properties of composites.

2. Materials and Methods

Tossa jute yarn of Bangladesh origin is used as reinforcement material in this experimental work. This is a single ply jute yarn having 8 lbs/spyndle (280 Tex, 1 lbs/spyndle = 35.090 Tex) linear density as well as 4.65 twist per inch and no extra finishes or coatings are used except the emulsion applied during spinning that contains jute batching oil. Unsaturated polyester, trade name POLYLITE® 420-100 by Reichhold UK Ltd. is used as the matrix material. Methyl ethyl ketone peroxide 35% in phthalate plasticizer trade name Butanox Catalyst by K & C Mouldings (England) Ltd. is used as a catalyst and cobalt (11) (2-ethylhexanoate) 1% CO in diisobutyl phthalate trade name Accelerator NL-40P by K & C Mouldings (England) Ltd. is used as an accelerator. The mould release agent is Chem lease® PMR-90 manufactured by Chem-Trend (Deutschland) GmbH. Hydroxyethyl cellulose (HEC) from Dow Europe GmbH, commercial name Cellosize HEC QP-52000H is used as binding material for the UD fibrous mat of yarn. Laboratory grade NaOH (> 97%) from Sigma-Aldrich, pellet form is used for alkali

treatment. > 99.7% acetic acid from Sigma-Aldrich is used to neutralise the alkali solution after treatment. All yarns to be treated by alkali are wrapped closely together on a glass fibre composite sheet in a single ply. Loose yarns dipped in water are described as water-treated yarn without tension. 1%, 3% and 5% NaOH solution (by weight) is made with tap water. Different yarn samples are dipped in these solutions separately at loose condition and using tension for 20 minutes at room temperature. Yarns are then neutralised by adding acetic acid, washed several times, squeezed gently (for loose yarn) and dried in an oven at 105^{O}C [12] for one hour keeping the extraction fan on. Yarns are then conditioned for 48 hours at room temperature before further processing.

After drying raw yarn and alkali-treated yarn without tension, was also wrapped on a glass fibre composite sheet to make a UD fibrous mat. All these yarns are sized with HEC to produce a UD fibrous mat. Tap water is used to make 0.6% (by weight) HEC solution. A homogenous solution is produced with the help of a mechanical stirrer. Stirring is performed for 10 minutes at 200 revolutions per minute. This solution is applied by hand to the yarns wrapped on the board using a brush. Yarns are dried in an oven at 105^{O}C [12] on the board for one hour. After drying, yarns are allowed to cool and are removed from the drum surface by cutting. These yarns are stored in ambient conditions for at least 48 hours before being used for composite manufacturing. The area density of the UD mat is 400-500 gm/m^2. The binding agent take-up is measured as 1.5-1.8% (by weight) of the yarn.

UD mat of sized yarn is cut according to the inside dimension of a picture frame (250x250 mm). Yarn weight is noted before infusion excluding the weight of HEC to calculate the fibre volume fraction. Resin infusion is performed at ambient temperature perpendicular to the yarn axis to generate uniform resin flow. Before infusion, mould release is applied (4-5 times) to the mould and the picture frame (Fig. 3.9) and allowed to dry. After infusion, the tool is kept closed overnight for solidification of resin to form composites. The composite plaques are then post-cured for 6 hours at 60^{O}C inside an oven, placed between two pieces of glass fibre composites with some weight added to avoid warping.

Composite plaques are stored at ambient conditions for at least 48 hours after post-curing prior to conducting any test. Before performing tensile tests, the volume fraction of fibre of the composites is measured. The volume of the fibres is calculated from the weight noted before infusion considering the density of jute fibre is 1.45 g/cc [13]. The volume of the composite plaque is determined by measuring the length, width and thickness. At least three readings are taken for each dimension and the mean values are considered for calculating the volume of the composite plaque. Fibre volume fraction of composites is obtained by dividing the volume of fibres by the volume of the composite plaque. As the fibre volume fraction of composites is measured for the whole plaque thus standard deviation is not available.

The tensile tests of composites are conducted with reference to BS EN ISO 527-4:1997 using an Instron 5969 fitted with a 50 kN load cell (software Bluehill®) and a video gauge extensometer. The cross-head speed used is 1mm/min. Five tests are performed for each plaque to find the mean values of tensile properties. Tests are performed under ambient conditions (15-25^{O}C and 60-90% RH).

3. Statistical hypothesis test

A statistical interference test is performed to draw the conclusion of random variation between two sets of data and the result is known as statistical significance. Each test of significance starts with a null hypothesis H_O which describes that there is no variation between data sets. The alternative hypothesis, H_1 is configured to establish the null hypothesis. The result of the test is always concluded in terms of the null hypothesis and denoted by 'P', either H_O is rejected in favour of H_1 or it is accepted. Rejecting the null hypothesis means that variation exists between data sets.

For a given hypothesis test the significance level α is a value considered for which a P-value is less than or equal to α that is to be as statistically significant. Typically, the values of α are 0.1, 0.05 and 0.01, corresponding to the probability of observation for an extreme value by chance [14]. For example, if a P-value of the test is 0.042, the probability of finding this value by chance is less than 0.05 and the result is significant at the level of 0.05 [15] and denoted by $P<0.05$. For accepting or rejecting the null hypothesis two different types of tests can be performed, a one-tailed test (i.e. one direction of interest) and a two-tailed test (i.e. the level of significance is divided into two halves). In this work, a two-tailed test is performed i.e. testing the possibility of a relation in both directions and the data were considered as two-samples of unequal variance.

4. Fibre and matrix volume fraction in composite

Mechanical properties of composites depend on the relative volume of reinforced fibres and matrix and the fibre volume fraction (V_f) in composites is defined according to equation 1.

$$V_f = \text{Volume of fibre/Volume of composite} \quad (1)$$

The volume fraction of the matrix (V_m) is defined according to equation 2.

$$V_m = \text{Volume of matrix/ Volume of composite} \quad (2)$$

As the volume of the composite is the sum of the fibre volume and the matrix volume, thus

$$V_f + V_m = 1 \quad (3)$$

Longitudinal modulus of composites in fibre direction is calculated by the formula called the rule of mixtures (ROM). Important assumption to calculate modulus using ROM is that the strain in the matrix and the fibre is same i.e. bond between fibre and matrix is perfect [271]. It means during stretching of composite in the fibre direction equal elongation would occur for fibre and matrix.

According to definition,

$$\text{strain } \varepsilon_1 = \Delta L/L \quad (4)$$

where L = original length and ΔL = elongation

As both fibre and matrix are elastic thus stress on fibre (σ_f) and stress on the matrix (σ_m) can be defined according to equation 6 and equation 7

$$\sigma_1 = E_1\varepsilon_1 \quad (5)$$

$$\sigma_f = E_f\varepsilon_1 \quad (6)$$

$$\sigma_m = E_m\varepsilon_1 \quad (7)$$

The load carried by lamina (P), fibre (P_f) and matrix (P_m) can be defined as-

$$P = \sigma_1 A \quad (8)$$

$$P_f = \sigma_f A_f \quad (9)$$

$$P_m = \sigma_m A_m \quad (10)$$

Where A, A_f and A_m are the areas of a cross-section of composite, fibre and matrix respectively.

The total load carried by the composite is the sum of the load carried by the fibre and the matrix.

$$P = P_f + P_m = \sigma_f A_f + \sigma_m A_m = \sigma_1 A \quad (11)$$

Then

$$E_1\varepsilon_1 A = E_f\varepsilon_1 A_f + E_m\varepsilon_1 A_m \quad (12)$$

Divide through by $\varepsilon_1 A$

$$E_1 = E_f(A_f/A) + E_m(A_m/A) \quad (13).$$

Thus

$$E_1 = E_f V_f + E_m V_m \quad (14)$$

Where V_f (fibre volume fraction) = A_f/A and V_m (composite volume fraction) = A_m/A

The rule of mixtures formula comes to

$$E_1 = E_f V_f + E_m(1-V_f) \quad (15)$$

Equation 15 is used to determine the back-calculated tensile modulus of fibre.

5. Results and Discussion

The influence of alkali treatment with and without tension on the tensile properties of jute composites is presented in Table 1. The composite plaques possess large variations in fibre volume fraction (minimum fibre volume fraction, V_f=35% and maximum V_f=46%). Thus, back-calculated tensile modulus and tensile strength of fibres are determined to compare the results. The calculation method is explained in Section 4 and the data are presented in Table 2.

Table 1. Fibre volume fraction and tensile properties of composites

Name of the composite plaque	Fibre volume fraction (%)	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation	Strain at failure (%)	Standard deviation
Raw yarn	35	18.3	0.6	187	4	1.34	0.07
1% NaOH no tension	36	18.1	0.8	160	13	1.30	0.06
1% NaOH with tension	36	19.9	1.1	196	8	1.34	0.03
3% NaOH no tension	37	19.0	0.8	167	5	1.29	0.08
3% NaOH with tension	38	20.3	1.0	207	10	1.33	0.06
5% NaOH no tension	46	22.7	1.3	214	7	1.44	0.08
5% NaOH with tension	43	23.1	1.6	242	11	1.36	0.07

Table 2. Back calculated values of tensile properties of fibres

Name of the composite plaque	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation
Raw yarn	45.3	1.6	405	10
1% NaOH no tension	43.5	2.3	319	36
1% NaOH with tension	48.1	3.0	420	24
3% NaOH no tension	45.5	2.2	331	14
3% NaOH with tension	47.3	2.6	429	26
5% NaOH no tension	44.8	2.8	383	15
5% NaOH with tension	48.5	3.7	469	28

No significant differences ($P<0.05$) of back-calculated fibre tensile strength are found for composites manufactured with 1% NaOH treated yarn with tension and composites manufactured with 3% NaOH treated yarn with tension and tensile strengths of these two composite plaques are very similar to those of composites manufactured with raw yarn. Possibly lower concentrated (1% and 3%) alkali solutions are not able to influence

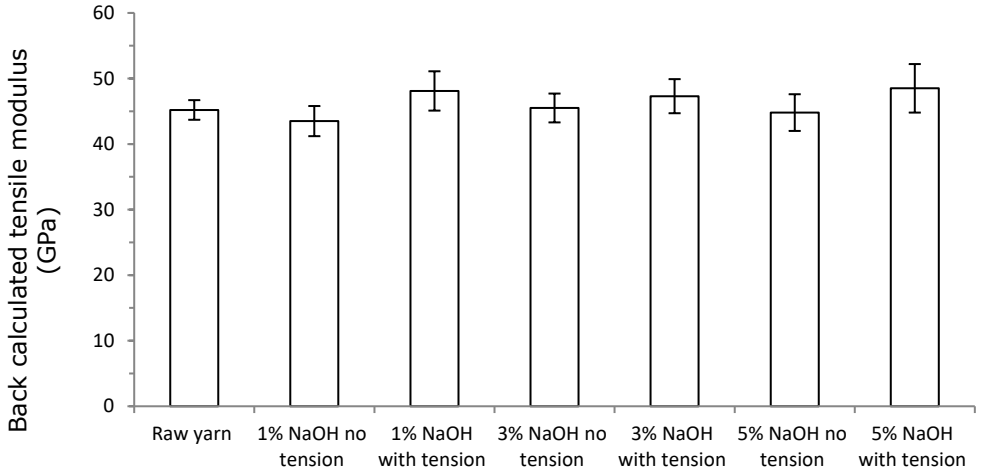


Fig. 1. Back calculated tensile modulus of fibres (error bars indicate standard deviation)

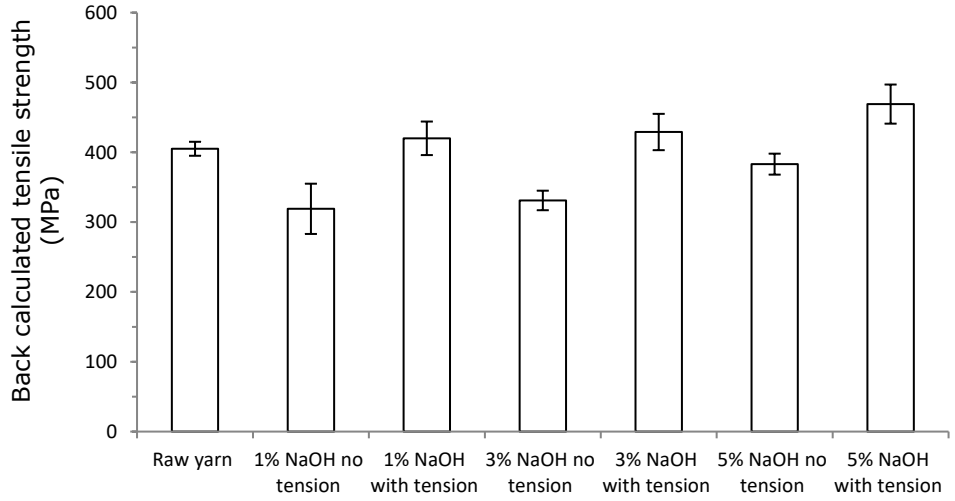


Fig. 2. Back-calculated tensile strength of fibres (error bars indicate standard deviation)

the tensile strength of composites. However, a significant improvement ($P<0.05$) of back-calculated fibre tensile strength is found for composites manufactured with 5% alkali-treated yarn without tension with respect to composites manufactured with 1% alkali-treated yarn without tension (20%) and with tension (12%). However, the differences between composites made with raw yarn and composites made with 5% alkali-treated yarn without tension is not statistically significant ($P<0.05$). Composites made with 5% alkali treated yarn with tension show 16% higher back calculated fibre tensile strength with respect to that property of composites manufactured with raw yarn which is statistically significant ($P<0.05$).

After comparing the back calculated tensile strength of fibres, composites made with 5% alkali treated with tension show a statistically significant increase in property with respect to that of composites manufactured with raw yarn. Thus, it can be said that lower concentration of alkali (1% and 3%) treatment has no effect on the back calculated tensile strength of fibres however 5% concentrated alkali solution showed significant improvement of the tensile strength. Possibly higher concentration of alkali with tension could able to remove hemicellulose and lignin that increases the back calculated tensile strength of fibres.

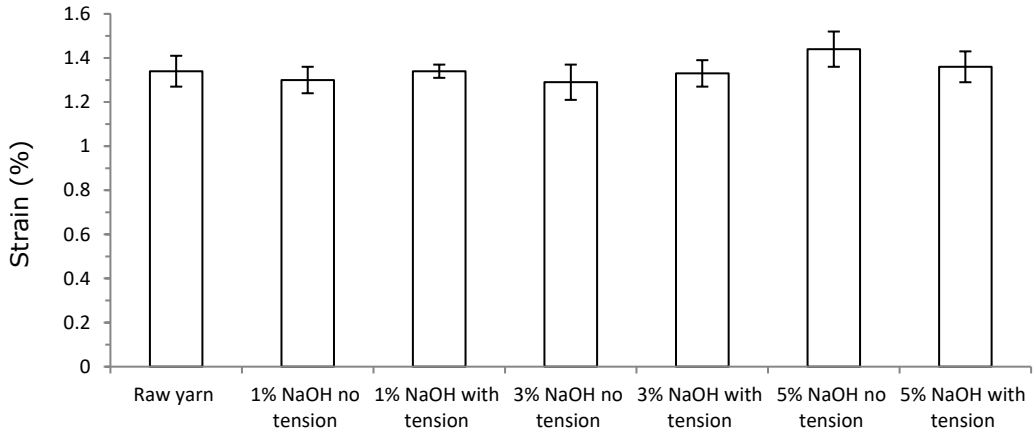


Fig. 3. Strain at failure of composites (error bars indicate standard deviation)

Composites manufactured with raw yarn as well as alkali treated yarns with or without tension do not show a marked change in strain at failure (Table 1. and Fig. 3.) revealing that the treatments performed in this chapter do not affect the strain at failure of composites.

6. Conclusions

Composites manufactured with yarns treated in alkali solution of different concentration with and without tension do not influence the back calculated fibre tensile modulus. However, tension during alkali treatment is found to be influential on back calculated fibres tensile strength. Back calculated fibre tensile strength increases for composites made with alkali treated yarn with tension with respect to that of composites made with a similar type of alkali treated yarn without tension for each case and this effect is possibly due to preventing shrinkage as a result of tension may helped to orient fibres toward the yarn axis. Composites manufactured with 5% alkali treated yarn with tension show significantly higher back calculated fibre tensile strength with respect to composites manufactured with raw yarn. No other composites manufactured with treated yarn show significant improvement of back calculated tensile strength of fibre with respect to composites manufactured with raw yarn. No significant variation is observed for strain at failure of composites due to alkali treatment.

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