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Tensile, surface and thermal characterization of jute fibres after novel treatments

Abdul Jabbar[a], Jiří Militký, Jakub Wiener, Muhammad Usman Javaid & Samson Rwawiire

Department of Material Engineering, Technical University of Liberec, Studentská 2461 17 Liberec, Czech Republic

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Jute fibres have been treated with enzymes, CO_2 pulsed infra red laser, ozone and plasma to study the effect of these treatments on their tensile, surface and thermal properties. Surface characteristics of jute fibres have been examined by scanning electron microscopy and Fourier transform infrared spectroscopy while thermal characteristics using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). No significant reduction in the tensile strength of treated jute fibres is observed. Changes in the surface chemical composition and increase in surface roughness are observed after the treatments. DSC data show a decrease in decomposition temperature of hemicellulose of all treated jute fibres. DSC and TGA analyses show no marginal difference in the cellulose decomposition temperature of untreated and treated jute fibres. The study shows a change in the surface chemistry and morphology after treatments without any substantial change in the thermal stability and tensile properties of jute fibres. The study also reveals the potential use of infrared laser and ozone treatments for the modification of natural fibres besides the conventional treatments using enzymes and plasma.

Keywords: Jute fibre, Surface characterization, Thermal properties, Tensile properties

1 Introduction

The demand and consumption of natural fibres have increased in the recent years due to the environmental concerns such as global warming, energy consumption and the desire to obtain products from renewable sources for total or partial substitution of petroleum-based synthetic fibres which are neither biodegradable nor renewable[1]. Plant fibres, such as jute, hemp, flax, ramie, sisal and coconut, have some interesting characteristics such as cost effectiveness, renewable, availability in huge quantities, low fossil-fuel energy requirements, reasonably good mechanical properties and low cost compared to synthetic fibres[2]. In addition, they have high specific properties such as stiffness, impact resistance[3], flexibility[4], and modulus[5]. Due to these characteristics, plant based natural fibres have found their applications in engineering, building materials and structural parts for the automotive application where light weight is required. However, the lack of compatibility of natural fibres with most of the matrices is a major concern for their application as reinforcement for composites. The poor moisture resistance of natural fibres leads to incompatibility and poor wettability with hydrophobic polymers, and this causes weakening the fibre/matrix adhesion[6].

Physical and chemical treatments can be used for the modification of natural fibres and to optimize the fibre/matrix interface[7]. Different surface modification techniques such as alkali[8-10], enzyme[11], plasma[1, 12, 13], ultrasound[14], ultraviolet[15] and Nd-YAG laser[16] treatments have been reported in literature to overcome the incompatible surface properties of natural fibre and polymer matrix. However, the use of some techniques especially CO_2 pulsed infrared laser and ozone in such modification processes of plant fibres are less common.

Jute is an abundant natural biodegradable plant fibre used as a reinforcement in natural fibre composites[17]and occupies the second place in terms of world production levels of cellulosic fibres[18]. Among all the other lignocellulosic fibres, jute contains a reasonably high proportion of stiff natural cellulose. The objective of this work is to study the effect of some novel fibre modification techniques such as infrared laser and ozone on the tensile, surface and thermal properties of jute fibres and to compare the findings with that of enzyme and plasma modified fibres. To the best of our knowledge, no work has been reported yet that fulfills the above-mentioned objective.

2 Materials and Methods

2.1 Materials

Jute yarn of English count 1.529 Ne, produced from tossa jute (*C. olitorius*) fibres was procured from a jute mill and used to produce a woven fabric having areal density of 600 gm^{-2} with 5-end satin weave design on a shuttle loom. Warp and weft densities of the fabric were kept 16 threads per inch and 20 threads

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[a]Corresponding author.
E-mail: abduljabbarntu@gmail.com

per inch respectively. Jute fabric was first washed with 2 wt% non-ionic detergent solution at 70 °C for 30 min. prior to treatments to remove any dirt and impurities and then dried at room temperature for 48 h.

2.2 Treatment Methods

2.2.1 Enzyme Treatment

Untreated jute fabric was subjected to enzyme treatment. A solution, having 1%owf Texazym DLG new, 3%owf Texzym BFE and 0.2g/L of Texawet DAF anti-foaming agent (all supplied by INOTEX, Czech Republic) in distilled water, was prepared and jute fabric was dipped into this solution at 50°C for 2 h, maintaining a liquor-to-material ratio of 10:1. After the treatment, the fabric was rinsed with fresh water several times and dried at room temperature for 48 h.

2.2.2 Ozone Treatment

Ozone treatment was carried out by putting the jute fabric for 1 h in a closed container filled with ozone gas. The container was connected to ozone generator 'TRIOTECH GO 5LAB-K' (TRIOTECH s.r.o. Czech Republic), which was continuously generating ozone gas at the rate of 5.0 g/h. Oxygen for the production of ozone gas was generated by 'Kröber O2' (Kröber Medizintechnik GmbH, Germany).

2.2.3 Laser Treatment

Laser irradiation was performed on the surface of jute fabric with a commercial carbon dioxide pulse infrared (IR) laser "Marcatex 150 Flexi Easy-Laser" (Garment Finish Kay, S.L. Spain), generating laser beam with a wavelength of 10.6 μm. Parameters that determine marking intensity of laser are marking speed (bits/ms), duty cycle (%) and frequency (kHz). In this study, the marking speed was set to 200 bits/ms, the duty cycle (DC) to 50 % and frequency to 5 kHz. The used laser power was 100 W. Laser beams interact with fibres, causing local evaporation of material, thermal decomposition or changing the surface roughness[19].

2.2.4 Plasma Treatment

Jute fabric was treated for 60 s with dielectric barrier discharge (DBD) plasma with discharge power of 190Wat atmospheric pressure using a laboratory device (Universal Plasma Reactor, model FB-460, Czech Republic).

2.3 Characterization Techniques

2.3.1 Tensile Testing

The tensile strength of jute fabric was measured in warp direction using Testometric M350-5CT instrument according to ISO 13934-1 standard[20].

2.3.2 Surface Morphology

The SEM photographs of jute fibres were taken using a scanning electron microscope TS5130-Tescan SEM at 20 kV accelerated voltage. The surfaces of the jute fibres were coated with gold by means of a plasma sputtering apparatus prior to SEM investigation and were investigated at ×2000 magnification to observe the surface morphological changes caused by different treatments.

2.3.3 ATR-FTIR Study

The raw and treated jute fibres were analyzed by ATR-FTIR spectroscopy. A Thermo Fisher FTIR spectrometer (model Nicolet iN10) was used in this study. The spectrometer was used in the absorption mode with a resolution of 4 cm^{-1} in the range of 4000–700 cm^{-1}.

2.3.4 Thermal Analysis

The thermal analysis of jute fibres, with and without treatments, was made by differential scanning calorimetry (DSC 6 Perkin Elmer instrument) using Pyris^{TM} software under a nitrogen atmosphere in the temperature range from 25 °C to 450 °C (heating rate 10 °C/min). Samples (6.5-8.5 mg) were placed in aluminum pans and sealed before DSC analysis. Thermogravimetric analysis (TGA/SDTA 851 METLER TOLEDO) of jute fibre samples (8.0-10 mg) was done under a nitrogen atmosphere (20 mL/min) in the temperature range from 30 °C to 450 °C (heating rate 10 °C/min).

3 Results and Discussion

3.1 Tensile Testing

The extent to which the treatment methods affect the tensile strength of treated jute fabrics is determined using tensile test (Fig. 1). A little reduction in the breaking strength of enzyme treated jute fibres is observed. This may be due to the partial removal of cementing materials binding the individual fibre cells, resulting in a little reduction of tensile strength. No significant difference in breaking strength of other treated fibres is observed when compared with untreated jute fibre.

3.2 SEM Analysis

In this study, morphological changes that occur after different treatments are examined (Fig. 2). Significant changes in surface morphology are observed after treatments. Figure 2(a) shows the multicellular nature of untreated jute fibre with a rather smooth surface, whereas a rough and fragmented surface morphology can be observed for enzyme treated fibres [Fig. 2(b)].

This may be due to the partial removal of cementing materials from the fibre surface after this treatment. Figure 2(c) displays the thermal degradation of surface fibres after laser treatment giving a porous and rough surface of fabric. The increase in roughness and cracks are noticeable on the surface of ozone treated jute fibre [Fig. 2(d)]. Plasma treatment causes a minor increase in fibre surface roughness. Overall, SEM micrographs give a strong indication that all treatments have changed the surface morphology of jute fibres.

3.3 FTIR Analysis

The FTIR spectra of untreated and treated jute fibres are shown in Fig. 3. A broad and intense peak at 3342 cm^{-1} suggests hydrogen-bonded O–H stretching vibration of cellulose and lignin structure of the fibre[21].

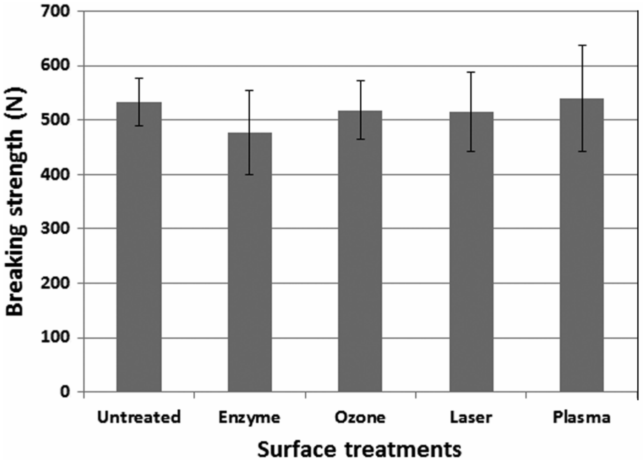

Fig. 1—Effect of treatments on tensile strength of jute fabrics

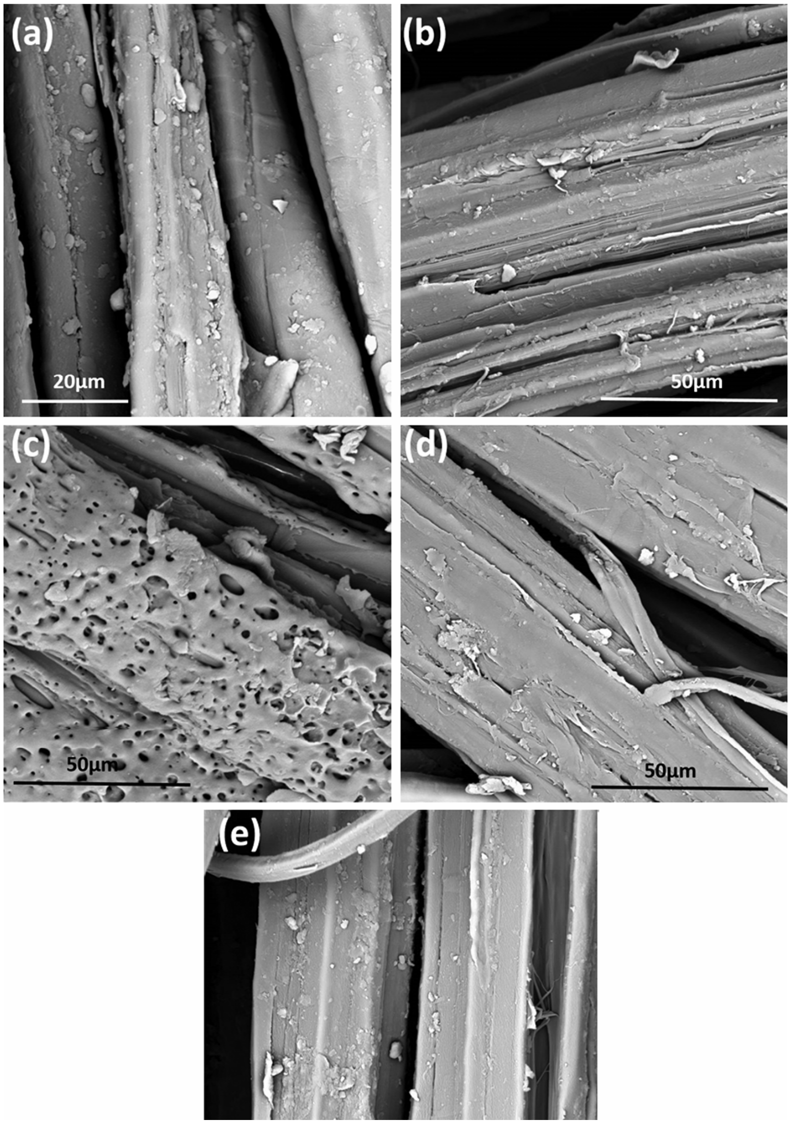

Fig. 2—Surface morphology of jute fibres (a) untreated, (b) enzyme treated, (c) laser treated, (d) ozone treated, and (e) plasma treated

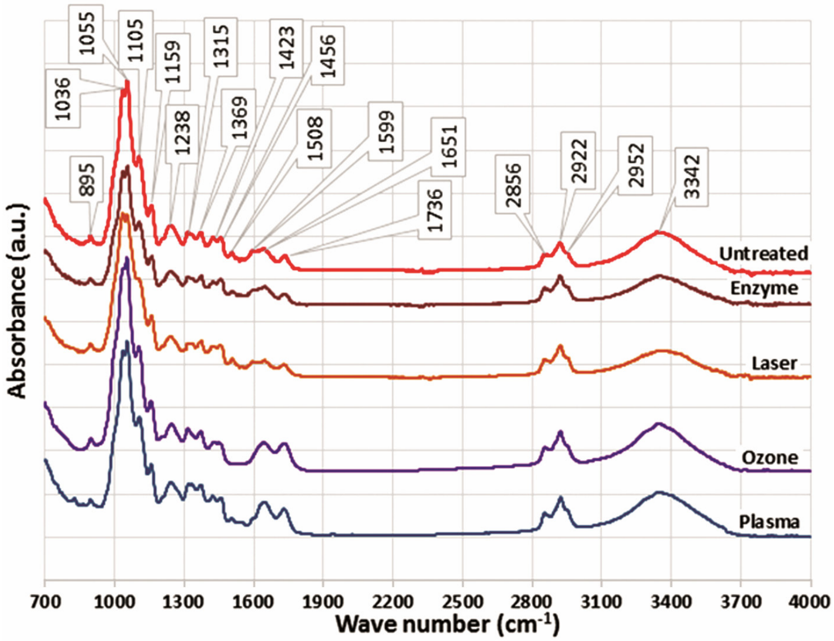

Fig. 3—ATR-FTIR spectra of untreated and treated jute fibres

The bands in the range of 2750–3000 cm^{-1} are related to the CH stretching in saturated hydrocarbons[22]. It is reported that the peaks at 2922 cm^{-1}and 2856 cm^{-1} are the characteristic bands for the C–H stretching vibration of CH and CH$_2$ in cellulose and hemicellulose components[23, 24]. The peak at 1736 cm^{-1} is due to stretching vibration of C=O bonds in carboxylic acid, ester components of cellulose and hemicellulose[25], and also non-conjugated carbonyls in lignin[26, 27]. This peak is slightly reduced for enzyme treated fibres which shows the partial removal of hemicellulose and lignin components upon treatment. However, the intensity and peak height at 1736 cm^{-1} are increased by ozone and plasma treatments. The peaks at 1599 cm^{-1} and 1508 cm^{-1}correspond to the aromatic ring vibrations in lignin. The increase in the intensity of peak at 1736 cm^{-1}, the reduction of peak after plasma treatment and disappearance of peak after ozone treatment at 1508cm^{-1}are possibly due to the oxidation of lignin[28]. The absorption at ~1650 cm^{-1} is probably associated with absorbed water in crystalline cellulose[27] and the lower intensity of this peak for laser treated fibre indicates a decrease in the amount of water absorbed. The absorbance bands at 1456, 1423, 1369 and 1315 cm^{-1} are possibly due to a CH$_3$ bending, CH$_2$and CH wagging in lignin. Some researchers pointed out that the band due to the C–O–C asymmetric stretching vibration for cellulose and hemicelluloses is observed at around 1238 cm^{-1}. This band is found more prominent in plasma treated fibres, which may be due to cellulose oxidation[25].

It has been indicated that the C–O–C symmetric glycosidic stretching or ring stretching mode at around 1100 cm^{-1} arise from the polysaccharide components (large cellulose) [22]. The absorption bands at 1055 and 1036 cm^{-1} are attributed to the C–O and O–H stretching vibration, which belong to polysaccharide in cellulose[24, 29]. The reduction in the shoulder height at 1105 cm^{-1} and peak height at 1055 cm^{-1} for IR laser gives a strong evidence that this treatment can alter the fibre surface structure.

In addition, the increased intensity of peak at ~3200–3600 cm^{-1} for ozone and plasma treated fibres gives an indication of reaction of hydroxyl bonds with the carboxyl group[30, 31], and reduction in peak at the same wavenumber range may be ascribed to the decrease in hydroxyl and carboxyl groups on the surface of laser treated jute fibre due to thermal degradation. As a result, there is strong evidence that the proposed treatments have altered the surface chemistry of jute fibres.

3.4 DSC Analysis

The differential scanning calorimetry (DSC) technique is used to compare the thermal behavior of untreated and treated jute fibres. The DSC curves presenting the endothermic processes of jute fibres are expressed in terms of heat flow per unit mass of fibres (Fig. 4). The results of DSC analysis are also presented in Table 1. The DSC curves of all samples show large endothermic peaks below 100°C which correspond to the heat of vaporization of water absorbed in the fibres[32] and may be associated with the loss of water molecules from the surface or interstitial spaces within the fibres[33]. The second small and broad endothermic peaks are observed in the temperature range of 160–300°C. It is reported[34] that hemicelluloses in lignocellulosic fibres degrade at around 200°C while other polysaccharides, such as cellulose, degrades at higher temperature. The degradation temperature of hemicelluloses is observed to decrease from 254°C for untreated fibre to lower temperatures for all treated specimens, especially the fibres treated with laser (241.5°C) and plasma (236°C). This may be explained due to decrease in both phenolic and secondary alcoholic groups[9] or oxidation of hemicelluloses by the formation of inter monomeric bonds in them[35]. The third endothermic peaks for cellulose degradation at around 365°C for treated fibres (359-365 °C) show no substantial difference from that of untreated jute fibre (366°C) as shown in Fig. 4 and Table 1.

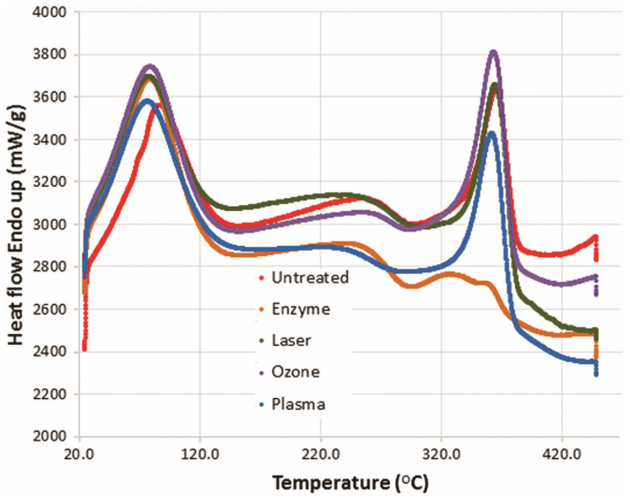

Fig. 4—DSC thermogram of untreated and treated jute fibres

Table 1—Thermal parameters of untreated and treated jute fibres obtained from DSC and DTG analysis

Treatment	Peak	Nature of peak	Peak temperature, °C	Weight loss (%) at 450°C for each sample
Untreated	1st	Endo	85.646	85.311
	2nd	Endo	254.342	
	3rd	Endo	366.142	
Enzyme	1st	Endo	79.160	77.737
	2nd	Endo	247.531	
	3rd	Endo	359.541	
Laser	1st	Endo	76.998	81.527
	2nd	Endo	241.536	
	3rd	Endo	365.330	
Ozone	1st	Endo	78.812	82.196
	2nd	Endo	251.707	
	3rd	Endo	363.989	
Plasma	1st	Endo	78.013	78.347
	2nd	Endo	236.048	
	3rd	Endo	362.510	

The cellulose decomposition peak for enzyme treated fibres is slightly inverted to exothermic and reduced significantly (Fig. 4). Similar reduction/inversion, due to alkali treatment was reported by Mitra *et al*[36,37]. This reduction/inversion may be attributed to the partial removal of noncellulosic constituents such as hemicellulose and lignin, leading to the destruction of chemical linkages between the constituents which might have some influence in reducing the cellulose degradation peak[38].

3.5 Thermogravimetric Analysis

TGA is also a useful technique to study the thermal stability/decomposition of fibres. The rate of weight loss of the sample as a function of temperature is measured to analyze the thermal degradation behavior of the material. Thermogravimetry (TG) and differential thermogravimetry (DTG) curves of untreated and treated jute fibres are shown in Figs 5(a) and (b) respectively. Like DSC curves, the peaks below 100 °C in the DTG curves are attributed to loss of

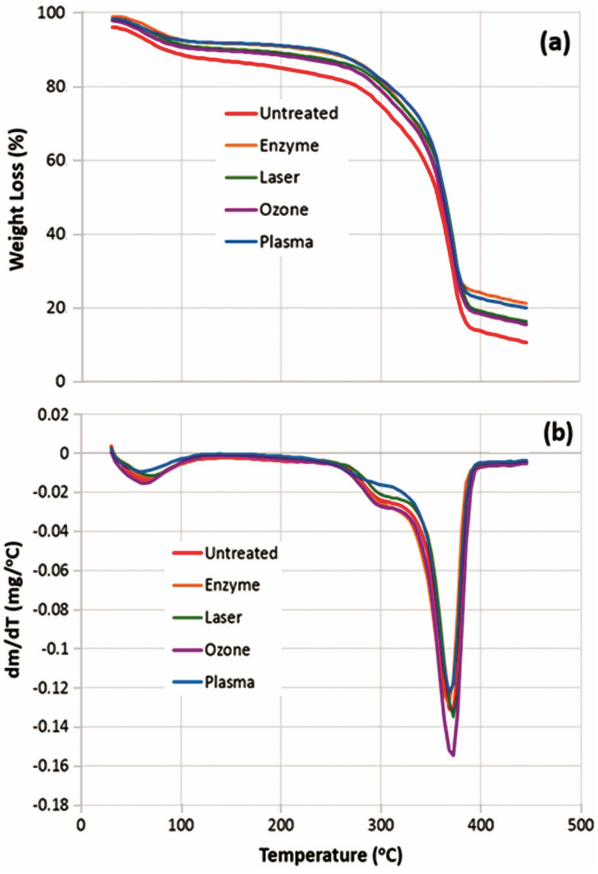

Fig. 5—TGA curves (a) TG and (b) DTG

moisture absorbed by the fibres, and strong sharp peaks at around 365 °C are due to the degradation of cellulose. No significant difference is observed in the degradation temperature of cellulose for all treated samples. These results are consistent with the above DSC analysis. From Fig. 5(a) and Table 1, it can be observed that weight loss (%) of untreated fibre (85.311%) is higher than that of treated jute fibres (77.737-82.196%) over the temperature range of 30-450 °C.

4 Conclusion

The study shows no noteworthy difference in the tensile strength of treated and untreated jute fibres. The FTIR analysis exhibits a change in surface chemical composition and SEM investigation shows an increase in surface roughness after treatments. DSC and TGA studies reveal no significant changes in the thermal stability of fibres. Hence, the selected novel treatment methods have potential for modification of lignocellulosic plant fibres without any substantial change in their tensile strength and thermal stability.

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