

A comparative study on chemical treatment of jute fiber: potassium dichromate, potassium permanganate and sodium perborate trihydrate

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Abstract Studies have been carried out on chemical treatments of jute fibers in order to hinder moisture absorption, which causes incompatibility with a non-polar polymer, and to increase the surface roughness for mechanical interlocking. The objective of this research is to improve the interfacial adhesion between jute fibers and polypropylene by oxidative treatments. On this basis, jute fibers were treated with potassium dichromate (PD), potassium permanganate (PM) and sodium perborate trihydrate (SP). Fourier transform infrared spectrometry (FTIR), X-ray photoelectron spectroscopy (XPS) and atomic force microscopy were used to characterize jute fibers. The effects of chemical treatments were also revealed by assessment of moisture absorbability, yarn tensile properties and interfacial shear strength with polypropylene. FTIR and XPS analyses confirmed oxidative modification of jute fibers using any of the surface treatments. It was observed that the proportion of O=C groups increased, whereas that of O–H groups decreased after oxidative modifications. Tensile strength and elasticity modulus results decreased after oxidative treatments, whereas PD, PM and SP enhanced the interfacial shear strength values by 25, 61 and 71 %, respectively. Only SP treatment

influenced moisture absorbability results significantly. The surface roughness of untreated jute fibers shows increments after chemical treatments due to partial removal of surface cementings. According to the findings obtained from surface characterization methods and physical tests, the highest interfacial adhesion with better compatibility with polypropylene was achieved after SP treatment by providing the highest surface roughness values and hydrophobic character of jute fiber.

Keywords Interfacial shear strength · Jute · Oxidative agent · Sodium perborate · Surface treatment

Introduction

Lignocellulosic fibers such as flax, hemp, jute and sisal have attracted attention because of their composite application in engineering and the transport industry where light weight is essential. With growing environmental awareness and substantial interest in ecological concerns, in recent years natural fibers have almost displaced conventional synthetic fibers such as aramid, carbon and glass fibers in fiber-reinforced polymeric composite materials. In spite of their beneficial properties such as biodegradability, recyclability, low density, high specific tensile performance and sustainability, poor compatibility with nonpolar polymers due to their high moisture

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absorption capacity has restricted their usability as a reinforcement. They also have some shortcomings such as quality variations and low thermal stability (Sanadi et al. 1994; Rowell et al. 1997; Sever et al. 2012).

Natural fibers are highly hydrophilic because of the presence of hydroxyl groups (OH) of anhydroglucose repeating units in the cellulose structure (Sawpan et al. 2011). Plant fibers are covered with pectin and waxy substances, thus hindering hydroxyl groups from reacting with polar matrices and forming mechanical interlocking adhesion with non-polar matrices (Seki 2009). In order to remove non-cellulosic constituents to produce reactive hydroxyl groups for interacting with the polymers, the surface characterization of plant fibers needs to be modified by chemical treatments (Liu and Dai 2007; Corrales et al. 2007; Sinha and Rout 2009) and/or physical treatments (Morales et al. 2006; Bozacı et al. 2009; Seki et al. 2010; Sever et al. 2011). The surface treatments can increase the surface roughness of the fiber and also alter the crystalline structure of the cellulose as well as fiber tensile properties (Mwaikambo and Ansell 2002; Wang et al. 2003; Sawpan et al. 2011; Bozacı et al. 2009). Chemical surface modifications of plant fibers including coupling agents (Singh et al. 2000; Hong et al. 2008), dilute polymer solutions (Van de Weyenberg et al. 2003), chemical treatments (Das and Munshi 2009), graft polymerization (Ouajai et al. 2004) and also steam treatments (Das et al. 2000) were investigated to enhance wettability and interface adhesion with non-polar polymers by a number of researchers. Among the different surface treatments, the usability of oxidative agents to decrease the hydrophilic character of cellulose by the oxidation of alcohol groups has recently increased in efficiency. The primary and secondary alcohol groups of cellulose are susceptible to the action of oxidizing agents (Khan et al. 2010). Physical characteristics such as mechanical performance, thermal behavior and electrical properties of cellulose materials can also change during the oxidation reaction.

Sodium perborate, a nontoxic and stable oxidizing agent, is synthesized by allowing it to crystallize out of an H_2O_2 -containing solution of sodium metaborate or by the reaction of sodium peroxide with boric acid or borates (Eagleson 1993; Huang and Sun 2003). Sodium perborate, a source of active oxygen, can produce hydrogen peroxide, which is responsible for

the oxidation when dissolved in water. Sodium perborate can be a good alternative agent for surface treatments of cellulose fibers.

The purpose of this research is to modify the surface characterization of jute yarns with oxidative agents to improve the compatibility with a non-polar polymer, polypropylene, when used as reinforcement. Surface treatments of jute fibers with oxidative agents (Khan and Ahmad 1996; Paul et al. 1997; Khan et al. 2010, 2012) have been performed by many researchers. However, a literature review provided no knowledge on surface treatment of jute fibers with sodium perborate trihydrate. This study also aims to compare the efficiency of sodium perborate trihydrate on jute fiber treatment with other commonly used oxidative agents. The effect of surface treatments such as potassium dichromate (PD), potassium permanganate (PM) and sodium perborate trihydrate (SP) on jute was investigated by Fourier transform infrared spectrometry (FTIR), atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS). Also a tensile test, moisture absorption properties and pull-out test (interfacial shear strength, IFSS) were evaluated.

Experimental

Materials

Jute yarns, having 445 tex linear density, were supplied by Atlantik Hali A.Ş., Turkey. PM (KMnO_4) and SP ($\text{NaBO}_2 \cdot \text{H}_2\text{O}_2 \cdot 3\text{H}_2\text{O}$) were supplied by Merck Corp. PD ($\text{K}_2\text{Cr}_2\text{O}_7$) was provided by Emir Kimya, Turkey. Polypropylene (PP), used in powder form with a melt flow index value of 35 g/10 min, was obtained from Petkim A.Ş.

Methods

Surface treatments

Jute yarns were immersed in 0.02 % w/v PD aqueous solution for 2 min at room temperature. The yarns were washed with distilled water several times to remove the chemical residues. Then, the jute yarns were oven-dried at 105 °C for 6 h and kept in a desiccator. In addition, the same procedure was applied for PP and SP treatments of jute yarns.

Determination of jute fiber content

The chemical composition of jute fiber was determined according to the gravimetric method (Mylsamy and Rajendran 2010). Prior to the chemical analysis for cellulose, hemicellulose and lignin, the fiber samples were oven-dried at 105 °C for at least 4 h and kept in a desiccator.

For the determination of cellulose content, 1 g of jute fibers in powder form was treated with 1.72 % sodium chlorite solution with a few drops of concentrated H₂SO₄. After carrying out the refluxing process for an hour, excess fluid was removed. The concentrated ammonia solution was added and then washed with distilled water, and finally the sediment was dried. The sediment content gives the cellulose content.

For the determination of the hemicellulose content, 1 g of jute fibers in powder form was dipped in 10 % NaOH solution for 1.5 h at room temperature and then cured with 5 % HCl solution. Excess fluid was sucked out, and the residue was oven-dried. The decrease in the initial weight gives the amount of hemicellulose.

For the determination of the lignin content, 1 g of jute fibers in powder form was treated in 12.5 ml of 72 % H₂SO₄ solution for an hour at room temperature. Then the solution with the sample was diluted with 300 ml of distilled water, and the sample was treated for 2 h. Excess fluid was removed, and the resulting sample was dried in the oven. The sediment was lignin.

Yarn tensile test

In order to examine the effects of chemical treatments on tensile properties of jute yarns, yarn tensile tests were carried out using the Instron Tensile Testing Machine at a cross-head speed of 20 mm/min and gauge length of 40 mm (Sever et al. 2012; Bozacı et al. 2009; Wang et al. 2003). The tensile test data were analyzed with a *t* test.

Determination of moisture absorption properties

The moisture content and regain of the untreated and treated jute yarns were tested in accordance with ASTM D 629-08. The moisture absorption test data were analyzed with a *t* test.

Fourier transform infrared spectrometer (FTIR)

FTIR spectra of the untreated and treated jute fibers were obtained using a Fourier transform infrared spectrometer (Perkin Elmer 100); 1 mg of fiber was ground into powder with high purity infrared grade KBr powder (100 mg) and pressed into a pellet for measurement. Each spectrum was recorded in the range of 600–4,000 cm⁻¹ with a resolution of 2 cm⁻¹. Spectra of the samples were obtained from 25 scans.

Atomic force microscopy (AFM) observation

The surface characterization of untreated and treated jute fibers was investigated by means of AFM using Digital Instruments MMSPM Nanoscope IV. Measurements were carried out in contact mode with a silicon nitride tip in air.

Yarn pull-out test

The yarn pull-out test is an experimental method that has been used to characterize the fiber-matrix interface (Sever et al. 2012). Interfacial shear strength (IFSS) was determined via pull-out test by a Shimadzu Autograph AG-IS series universal testing machine (contact speed 0.1 mm/min) after embedding the untreated and treated jute yarns in the PP matrix for 180 min at room temperature. The debonding force F_{\max} , the diameter d and the embedded length of the fibers l_e were determined, and the IFSS τ_d was calculated from the following equation (Bozacı et al. 2009; Sever et al. 2012):

$$\tau_d = \frac{F_{\max}}{d \pi l_e} \quad (1)$$

X-ray photoelectron spectroscopy (XPS)

The surface chemistry of jute fibers was investigated using XPS analysis. The XPS spectra were obtained using a PHI ESCA instrument equipped with a monochromatic Al K α radiation source. Gaussian-Lorentzian functions were used for curve fitting (80 % Lorentzian) of C1s and O1s spectra to determine the functional groups and their concentration.

Results and discussion

Chemical composition of jute fiber

The chemical composition of the untreated jute fibers is tabulated in Table 1. The contents of cellulose, hemicellulose, lignin and other components were obtained at 79.0, 12.6, 8 and 0.4 %. As seen in Table 1, the principal constituents of jute fibers are cellulose, hemicellulose and lignin, normally considered to be the main binder.

Yarn tensile properties

The results of evaluation of the breaking strength and elasticity modulus of untreated (J) and treated jute yarns (PDJ, PMJ and SPJ) are summarized in Fig. 1a–b. It can easily be seen that tensile strength and elasticity modulus values decreased after any of the surface treatments. Oxidizing agents mainly affect the amorphous regions and the surface of the crystallites of cellulose fibers (Khan et al. 2010). The opening of the pyranoside ring as a consequence of the oxidation of primary and secondary alcohol groups of cellulose and the shortening of the macromolecular chains produced by this chemical attack can deteriorate the tensile properties of jute fibers (Khan et al. 2010). In addition, with the removal of surface impurities after chemical treatments, cellulose becomes more susceptible to the action of oxidizing agents (Khan et al. 2010). The highest and lowest decrements in the breaking strength of jute yarns were observed after PD and PM treatments, respectively (Fig. 1a). According to the statistical analysis, there are significant differences among the breaking strength and the elasticity modulus values of J, PDJ, PMJ and SPJ.

Determination of moisture content and moisture regain

Figure 2 shows the effects of chemical treatments on moisture absorption properties of jute yarns. The

Table 1 Chemical composition of untreated jute fibers

Component	Content (%)
Cellulose	79.0
Hemicellulose	12.6
Lignin	8
Others	0.4

oxidation reaction of alcohol groups of cellulose negatively influences the moisture content of jute fibers because this chemical reaction reduces the total amount of polar hydroxyl groups of jute fibers (Khan et al. 2010; Wetz et al. 2010). The partially removal of surface impurities that consist of hydroxyl groups may also affect the moisture content of jute fibers. Although PMJ has a higher moisture content than untreated jute yarns, as presented in Fig. 2, there is no statistically significant difference between the

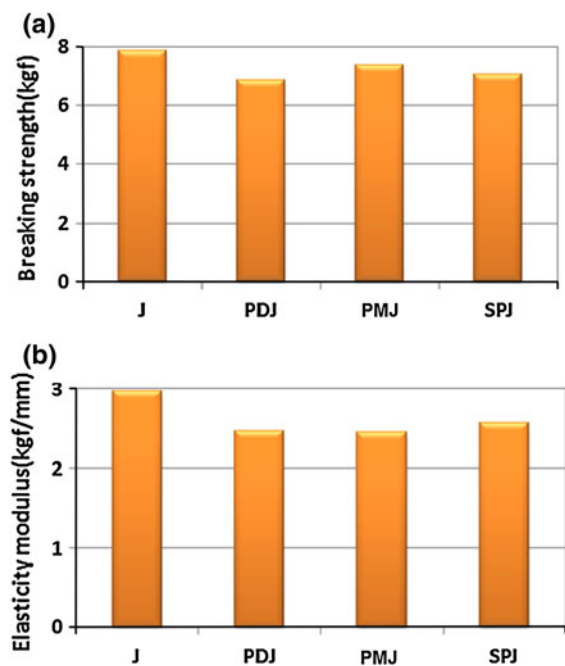


Fig. 1 The effects of surface treatments on the **a** breaking strength and **b** elasticity modulus

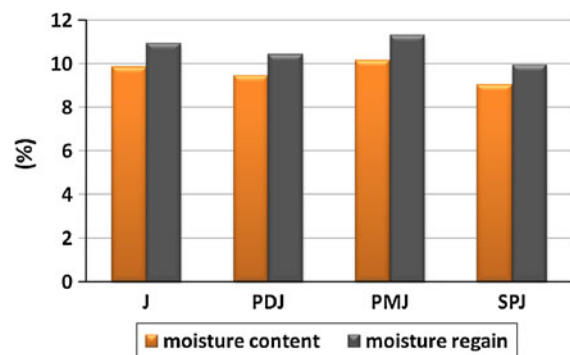


Fig. 2 The effects of surface treatments on moisture content and moisture regain

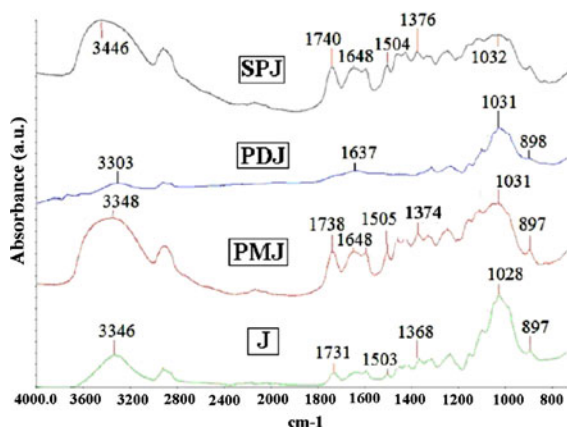


Fig. 3 FTIR spectra of the jute fibers

moisture content and moisture regain values of J, PDJ and PMJ. However, SP treatment decreased the moisture content and moisture regain values of jute yarns significantly.

FTIR analysis

FTIR analysis was conducted to investigate chemical interaction between jute fibers and chemical agents. FTIR spectra of untreated and treated jute fibers are presented in Fig. 3. A peak was obtained at the $3,336\text{ cm}^{-1}$ absorption band in the spectrum of untreated jute assigned to O–H stretching vibrations. After PD surface treatment, O–H stretching shifted this band to $3,303\text{ cm}^{-1}$. However, after SP treatment, this band shifted to $3,446\text{ cm}^{-1}$. The peak intensity in the absorption band at about $2,900\text{ cm}^{-1}$ assigned to CH_2 and CH_3 stretching vibrations was remarkably decreased after PD treatment (Seki et al. 2013).

The absorption band at $1,731\text{ cm}^{-1}$ assigned to C=O groups (Jabasingh et al. 2012) in the spectrum of untreated jute was observed to increase to $1,738$ and $1,740\text{ cm}^{-1}$ after PM and SP treatment, respectively, due to the oxidation of alcohol groups to carbonyl groups. It should also be mentioned that the intensity of the peak showing C=O groups of untreated jute fiber increased after PM and SP treatment. However, this could not be seen after PD treatment. This may be due to the removal of the molecules consisting of C=O groups.

The absorption band at $1,028\text{ cm}^{-1}$ in the spectrum of untreated jute was assigned to cyclic alcohol groups (Seki 2009). The intensity of the peak decreased after

any of the treatments. This may be due to the oxidation of alcohol groups. The peaks in the range of $1,600\text{--}1,650\text{ cm}^{-1}$ absorption bands for PMJ, SPJ and PDJ indicated the absorbed water in crystalline cellulose (Seki 2009).

The spectra of J, PMJ and SPJ exhibited absorption bands at $1,503\text{--}1,505\text{ cm}^{-1}$, which were attributed to the presence of aromatic rings in lignin (Seki et al. 2013; Jabasingh et al. 2012; Arshad et al. 2011). After PD modification, these bands could not be seen. The spectra of J, PMJ and SPJ exhibited a band at $1,368\text{--}1,374\text{ cm}^{-1}$, assigned to C–H bending in hemicellulose and lignin (Mwaikambo and Ansell 2002; Oлару et al. 2011). After PD modification, this peak also could not be seen.

AFM observation

The surface morphology of untreated and treated jute fibers derived from AFM in contact mode are presented in Fig. 4, and the average surface roughness values of jute fibers are given in Table 2. The average roughness values (R_a) of untreated jute fibers increased by 17, 33 and 39 % after PD, PM and SP treatments, respectively. The fibrillation of the jute fibers after any of the surface treatments was observed. This may confirm that the partial removal of surface impurities and binder materials of fiber elements by chemical treatments can increase the surface roughness of jute fibers.

Yarn pull-out test

The chemical surface treatments provide mechanical interlocking between the fiber and polymer by increasing the surface roughness of jute fibers. In addition, the surface modifications also provide numbers of chemical bonds formed in the interface by increasing the number of functional groups on the fiber surface. IFSS data can be used to determine how the surface treatments affect the fiber/matrix interfacial adhesion and the mechanical properties of the composite, and also the performance of the fiber as reinforcement material (Sever et al. 2012; Park et al. 1998; Wang et al. 2000). Table 3 gives the IFSS results of the jute yarns and polypropylene matrix. It can be clearly understood that the IFSS result of the untreated jute yarn was enhanced by 25, 61 and 71 % by PM, PD and SP treatments, respectively. In

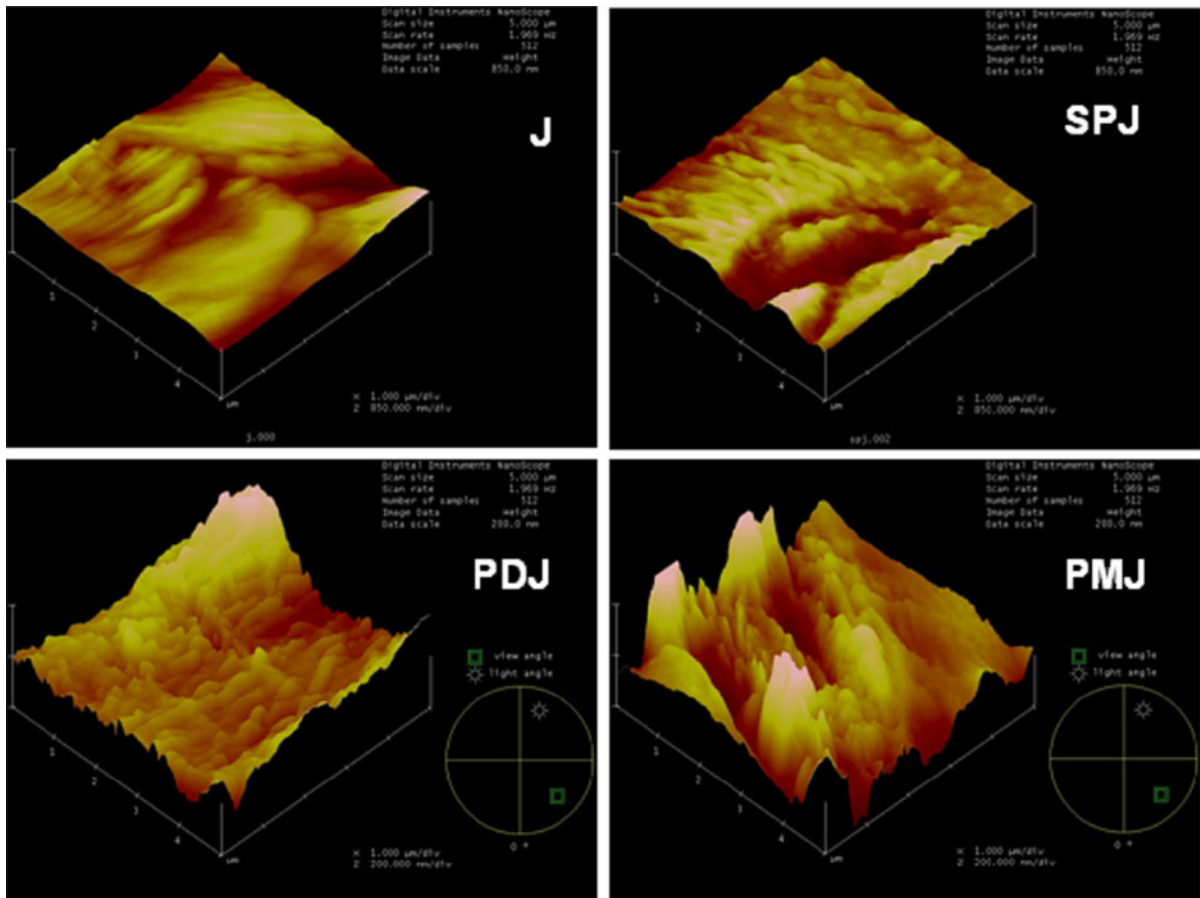


Fig. 4 AFM images of jute fibers

Table 2 Surface roughness values of the jute fibers

Sample	R_a (average roughness value)
J	33.43
PDJ	39.05
PMJ	44.33
SPJ	46.47

addition, there is a statistically significant difference between the IFSS values of untreated and treated jute yarns.

The IFSS result of SPJ was in accordance with the surface roughness value given in Table 2. As mentioned before, the surface roughness contributes to the fiber/matrix interfacial adhesion. The higher surface roughness leads to higher interfacial shear strength by increasing mechanical interlocking (Sever et al. 2012, 2011). However, the IFSS also depends on functional

Table 3 IFSS of the jute yarns

	IFSS (MPa)
J	5.39 ± 0.50
PDJ	8.65 ± 0.26
PMJ	6.71 ± 0.85
SPJ	9.22 ± 0.37

groups on the fiber surface, confirmed with XPS results.

XPS analysis

The surface chemical composition of untreated and treated jute fibers was investigated by using XPS analysis. Table 4 gives the atomic concentrations and C/O ratios for untreated and PD, PM and SP treated jute fibers. As can be seen from Table 4, the C content

Table 4 Surface chemical compositions (%) of jute fibers

	C	O	C/O	Si	N	Ca
J	64.3	29.7	2.16	2.8	1.8	0.4
PDJ	69.1	24.8	2.78	1.0	3.1	1.4
PMJ	73.5	22.4	3.28	1.2	1.6	0.8
SPJ	69.5	26.5	2.62	1.2	2.1	0.6

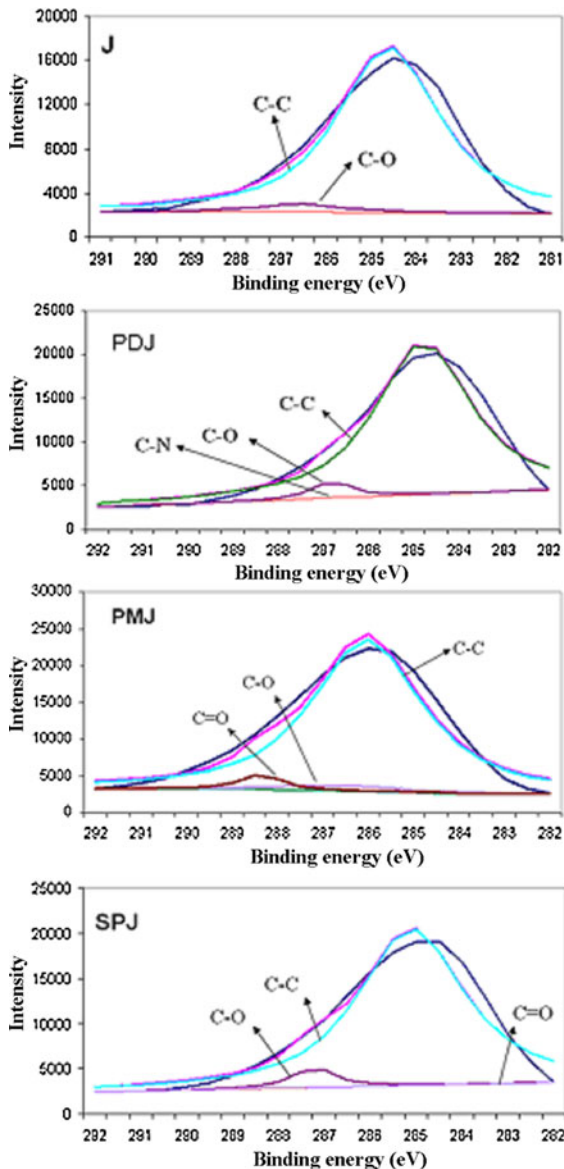


Fig. 5 XPS spectra showing the deconvoluted C1s envelope for the jute fibers

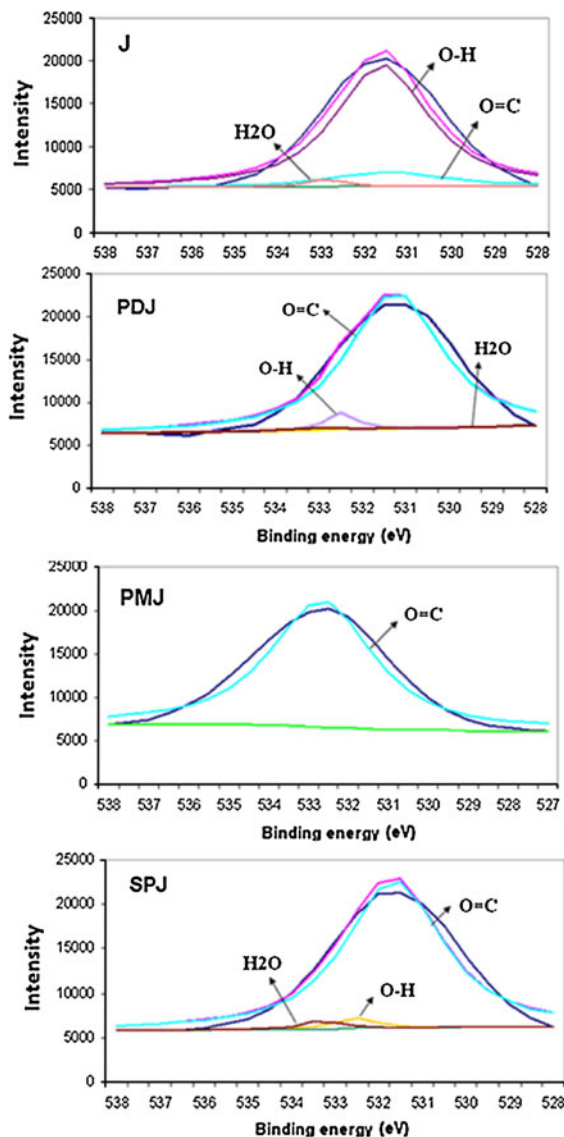


Fig. 6 XPS spectra showing the deconvoluted O1s envelope for the jute fibers

of untreated jute fiber increased by 14, 8 and 7 % after PM, SP and PD treatments, respectively. But the O content was observed to decrease after any of the surface treatments. C/O ratios of J, PDJ, PMJ and SPJ were calculated to be 2.16, 2.78, 3.28 and 2.62, respectively. A low O/C ratio (or a high C/O ratio) modified the wood surface from hydrophilic to more hydrophobic (Sernek 2002). According to this, it was determined that PM, SP and PD chemical treatments changed the surface of untreated jute fiber from hydrophilic to more hydrophobic.

Table 5 The concentration of functional groups on the surface of jute fibers (Sever et al. 2012; Seki et al. 2012)

	C–C, C–H	C–O	C=O, O–C–O	C–N	O=C	O–H	H ₂ O
<i>J</i>							
C1s							
eV	284.6	286.6					
%	95.3	4.17					
O1s							
eV					531.5	532.5	533
%					12.11	85.83	2.06
<i>PDJ</i>							
C1s							
eV	284.3	286.3		285.6			
%	95.37	4.31		0.319			
O1s							
eV					531.25	532.5	532.97
%					95.76	3.63	0.62
<i>PMJ</i>							
C1s							
eV	285.52	286.2	287.84				
%	90.89	3.88	3.29				
O1s							
eV					532.67		
%					99.8		
<i>SPJ</i>							
C1s							
eV	284.6	286.7	287.5				
%	94.91	4.75	0.34				
O1s							
eV					531.64	532.5	533.27
%					92.96	5.06	1.998

Deconvolution analysis of C1s and O1s peaks was carried out to obtain the functional groups on the surface of untreated and treated jute fibers (Figs. 5, 6). Table 5 shows the peak assignments and the concentration of functional groups on the surface of the jute fibers. Table 5 shows that a great proportion for jute fibers belongs to C–C groups. As can also be seen in Table 5 and Fig. 5, the C–C and C–O chemical bonds remain almost similar after chemical treatments. While the proportion of O=C groups (carbonyl groups) is increasing, that of O–H groups is decreasing (Table 5; Fig. 6). This is taken as evidence of oxidative modification (Levine et al. 2000). Oxidative treatment modifies the fiber surface more hydrophobic by reducing the number of O–H groups and develops compatibility between jute fiber and a non-polar polymer.

Conclusion

In this study, effects of oxidative treatments on interfacial adhesion of jute to polypropylene and physical properties of jute yarns were investigated. XPS and FTIR analyses indicated oxidative modification with any of the treatments by reducing alcohol groups and increasing O=C groups. This means that PDJ, PMJ and SPJ have a more hydrophobic surface character with higher C/O ratios compared with untreated jute fiber. The pull-out test supports this view that any of the treatments increased IFSS results of untreated jute yarn by providing better compatibility with polypropylene. The average surface roughness values of untreated jute fibers derived from AFM show increments after chemical treatments due to partial removal of surface impurities. SP treatment

brings about 71 % increments in the IFSS as the greatest improvement by providing the highest surface roughness and hydrophobic surface character compared with untreated jute yarn. The tensile properties deteriorated after any of the treatments. In addition, only SP treatment was found to influence the moisture absorption properties significantly. According to the findings obtained from surface characterization methods and physical tests, the highest interfacial adhesion with polypropylene was achieved after SP treatment, providing the highest surface roughness values and also increasing the C/O ratio in the characterization of the hydrophobic surface character.

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