

# Fourier Transform Infrared Spectroscopic Analyses of Argon and Oxygen Plasma Treated Jute

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**Abstract**— Low temperature plasma (LTP) treatment, a kind of environmental friendly surface modification technique, was applied to biodegradable and ligno-cellulosic jute fibre with the use of two nonpolymerizing gases, namely argon (Ar) and oxygen (O<sub>2</sub>) at various discharge power levels of 50, 75 and 100 W, and exposure times 5, 10, 15 and 20 min. with a flow rate of 0.2 L/min where W, min. and L/min stands for discharge powers, exposure times and liter per minute for the LTP treatment of the fibres. Fourier Transform Infrared (FTIR) spectra of both raw and low temperature Ar and O<sub>2</sub> plasma treated jute were studied at various discharge power levels and exposure times. The analyses of the chemical structure by FTIR spectra of raw jute and plasma treated jute fibre showed similar spectra with the change in the peak intensity and shift in the peak position. From FTIR spectra, the changes at the bands around 1730, 1650, 1235, 1077, 1030 and at 890 cm<sup>-1</sup> revealed surface chemistry of fiber changes after plasma treatment. FTIR has given evidence of moisture removal from the fibre.

**Index Terms**— Fourier Transform Infrared, Chemical Structure, Discharge Power, Exposure Time and Plasma.

## I. INTRODUCTION

Spectroscopy is the study of the interaction of electromagnetic radiation with matter. There are many forms of spectroscopy, each contributing useful information to identify substances and to determine various characteristics of their structure [2]. FTIR spectroscopy is the spectroscopy that deals with the infrared region of the electromagnetic spectrum, that is light with a longer wavelength and lower frequency than visible light. On the immediate high energy side of the visible spectrum lies the ultraviolet, and on the low energy side is the infrared. The portion of the infrared region most useful for analysis of organic compounds is not immediately adjacent to the visible spectrum, but is that having a wavelength range from 2,500 to 16,000 nm, with a corresponding frequency range from  $1.2 \times 10^{14}$  to  $1.9 \times 10^{13}$  Hz [7].

Photon energies associated with this part of the infrared are not large enough to excite electrons, but may induce

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vibrational excitation of covalently bonded atoms and groups. The covalent bonds in molecules are not rigid sticks or rods, such as found in molecular model kits, but are more like stiff springs that can be stretched and bent. In physical and analytical chemistry, FTIR spectroscopy is a technique used to identify chemical compounds based on how infrared radiation is absorbed by the compound's chemical bonds [6]. Plasmas are ionized gases. An ionized gas consists mainly of positively charged molecules or atoms and negatively charged electrons [4]. A gaseous complex that may be composed of electrons, ions of polarity, gas atoms and molecules in the ground or any higher state of any form of excitation as well as of light quanta is referred to as plasma [5]. The ionization degree can vary from 100 % (fully ionized gases) to very low values (partially ionized gases). The presence of a non-negligible number of charge carriers makes the plasma electrically conductive so that it responds strongly to electromagnetic fields. Plasma therefore has properties quite unlike those of solids, liquids or gases and is considered to be a distinct state of matter [8].

Jute is a golden fibre as well as a major cash crop of Bangladesh. A great advantage of jute fibre is that it is environment friendly natural fibre. This natural fibre earns a lot of foreign currency by its export and its various products. Jute plays a very important role in the socio-economic activities of Bangladesh [1, 7]. Prospect for producing a wide variety of jute products and thus maximum utilisation of jute in the possible fields of textile sectors as well as thermal sectors are very encouraging. At present jute is facing tough competition from the convenient and competitive synthetics counter parts in the world market. The only way to save jute is through its uses in various diversified ways. Hence for better performability and to explore diverse use of jute, study of thermal properties of jute fibre is very important.

## II. MATERIALS AND METHODS

### A. Low Temperature Plasma Treatment

Jute fibres (Corchorus Olitorius or Tossa jute) were collected from the local market in Bangladesh. The fibres were introduced into a bell jar type capacitively coupled glow discharge reactor as shown in figure 1

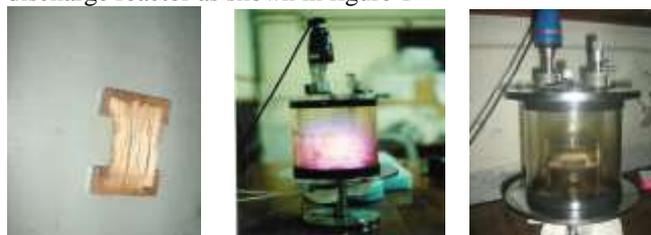


Fig. 1 Schematic diagram of jute fibre and position of it in the glow discharge reactor

To sustain a glow discharge i.e. for getting proper and uniform plasma, the conductive electrodes are separated 0.035 m apart from each other. In order to exposed all through uniform LTP treatment on the samples surface, the fibres (length of each fibre: 0.08 m) were inserted in between the two metallic electrodes by a carrier. After placing jute fibres between pair of electrodes, the glow discharge chamber was evacuated by a rotary pump at a pressure of 1.33 Pa. Ar and O<sub>2</sub> were considered as plasma gas for treating the jute fibre. In all treatments, both process gases were introduced separately into the reaction chamber by a flowmeter at a flow rate of 0.2 L/min. which is maintained by a needle valve. The discharge powers were adjusted at 50, 75 and 100 W at a line frequency of 50 Hz with the duration of exposure times of LTP treatment of fibres were 5, 10, 15 and 20 min. Figure 2 shows a flow chart of a plasma treatment system which was used in this experiment.

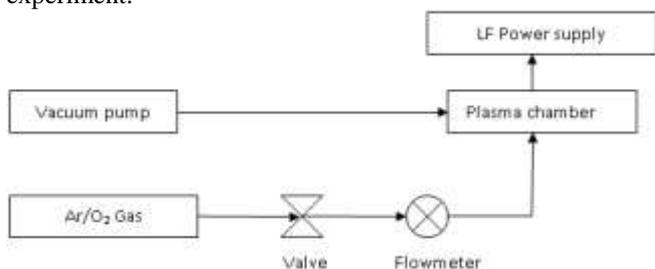


Fig. 2 Flow chart of the plasma treatment set-up

After the completion of the plasma treatment jute samples were removed. Later, the plasma treated fibres were immediately placed into a desiccator with the silica gel.

**B. Sample Preparation**

In preparing the samples, both raw and plasma treated jute fibres were cut into small pieces of sizes of about 1.0-2.0 mm. By mortar and pestle these small pieces of jute were ground, crushed and mixed in order to convert into powder form. Finally, the jute powders were sieved by a very fine and thin net to make the powder finer.

**C. Fourier Transform Infrared Spectroscopy**

The FTIR spectra were recorded to investigate the chemical structure of raw jute and LTP treated jute at different discharge power and exposure time. The FTIR spectra of the different samples were recorded at room temperature by using a double beam FTIR spectrophotometer (SHIMADZU FTIR-8900 spectrophotometer) in the wavenumber range of 4000 - 400 cm<sup>-1</sup>. Figure 3 shows a photograph of a FTIR machine which was used in this experiment.



Fig. 3 A photograph of a FTIR spectrometer

The FTIR spectrums of all the samples were obtained by putting these samples in a potassium bromide (KBr) measuring cell. Both raw jute and LTP treated jute powder mixed separately with KBr were prepared for recording the FTIR spectra of the samples. The FTIR spectra of all the types of jute samples were recorded in transmittance (%) mode.

**III. RESULTS**

The FTIR spectroscopic analyses were performed on the raw jute and the LTP treated jute fibres. The FTIR spectra of the raw and the LTP treated jute fibres are illustrated in figures 4, 5, 6, 7, 8 and 9 for the case of 50 W, 20 min.; 75 W, 20 min.; 100 W, 20 min.; 100 W, 10 min.; 100 W, 15 min. and 100 W, 20 min. respectively. It is seen from the FTIR spectra that all the spectra have similar bands with the change in the relative peak intensity and shift in the peak positions. The spectrum of raw jute fibre shows absorption bands for chemical groups characteristic of the fibre components that are cellulose, hemicellulose and lignin. Originally, these components came from alkenes, aromatic and different oxygen containing functional groups of ester, ketone and alcohol. The positions of major absorption peaks of LTP treated and raw jute fibres are listed in table 1.

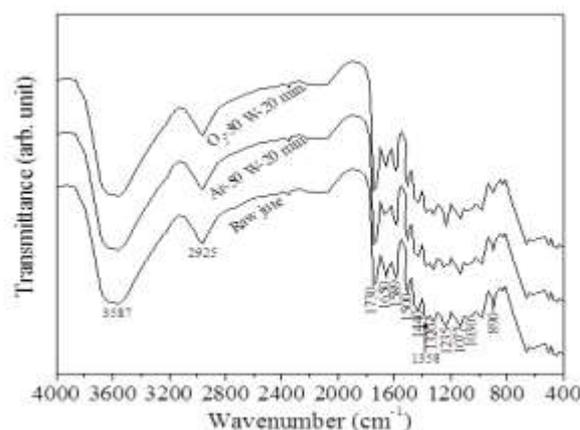


Fig. 4 FTIR spectra of raw, Ar and O<sub>2</sub> LTP treated jute at 50 W, 20 min.

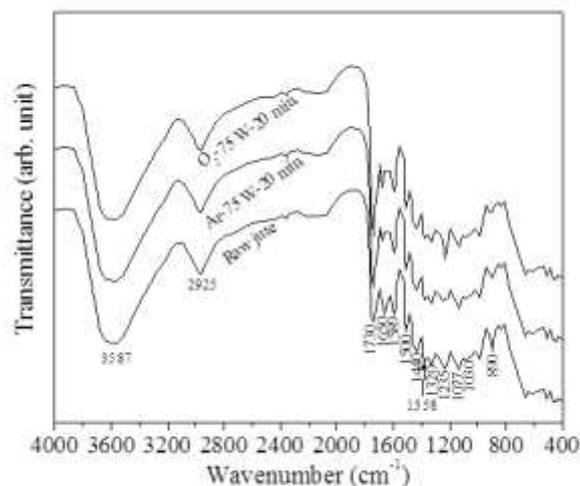


Fig. 5 FTIR spectra of raw, Ar and O<sub>2</sub> LTP treated jute at 75 W, 20 min.

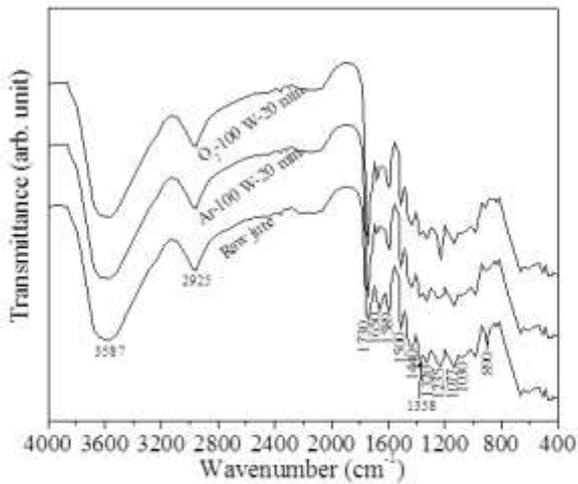


Fig. 6 FTIR spectra of raw, Ar and O<sub>2</sub> LTP treated jute at 100 W, 20 min.

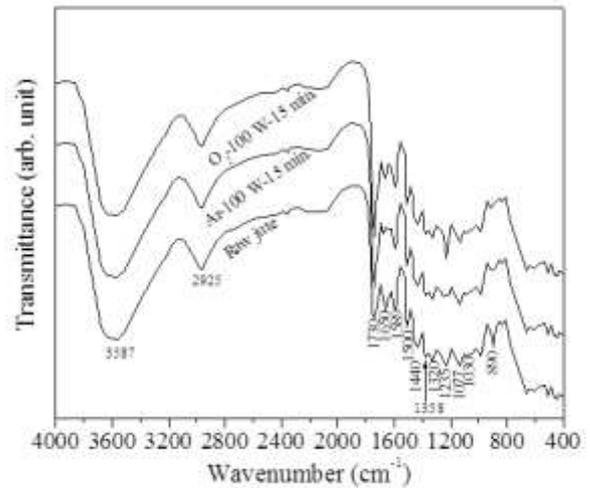


Fig. 8 FTIR spectra of raw, Ar and O<sub>2</sub> LTP treated jute at 100 W, 15 min.

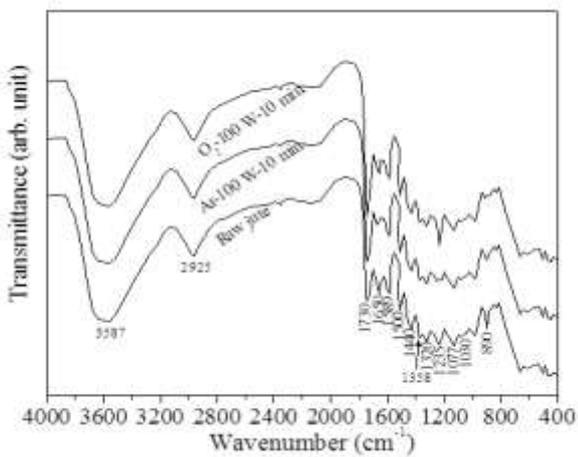


Fig. 7 FTIR spectra of raw, Ar and O<sub>2</sub> LTP treated jute at 100 W, 10 min.

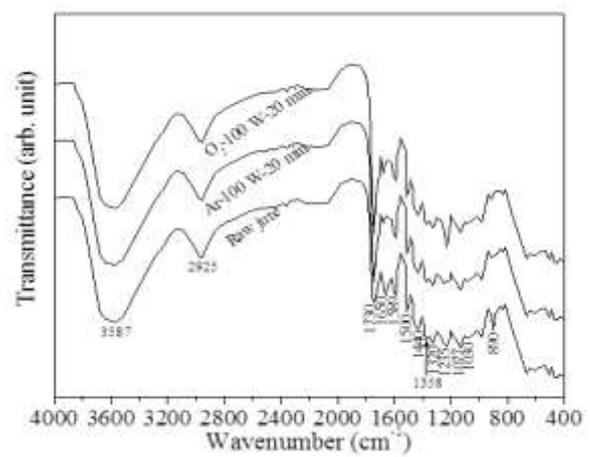


Fig. 9 FTIR spectra of raw, Ar and O<sub>2</sub> LTP treated jute at 100 W, 20 min.

Table 1 Standard and observed FTIR absorption peak position for different compounds of jute

Structural bond	Standard peak position (cm <sup>-1</sup> ) [10]	Reported peak position (cm <sup>-1</sup> )[3]	Observed peak position (cm <sup>-1</sup> )	Observed intensity and peak height after LTP treatment
OH stretching	3600~3000	3510	3587	-
CH <sub>2</sub> , CH <sub>3</sub> stretching	3000~2800	2920	2925	-
C=O stretching	1750~1725	1740	1730	Intensity and peak height increased
H-O-H bending	~1650	1643	1650	Intensity and peak height decreased
Benzene ring stretching	~1580, ~1500	1589, 1520	1589, 1500	-
CH <sub>2</sub> deformation	1480~1435	1430	1440	-
CH <sub>3</sub> bending	1390~1340	1358	1358	-
CH <sub>2</sub> wagging	~1320	1320	1320	-
C-O-C asym. stretching	1275~1185	1230	1235	Intensity and peak are more prominent in O <sub>2</sub> plasma compared to Ar plasma
C-O stretching	1160~1000	1085	1077	Bands are slightly shifted toward the higher wavenumber
O-H deformation	1080~1030	1010	1030	Bands are slightly shifted toward the higher wavenumber
β-Glucosidic linkage	~885	890	890	Less prominent

These peak positions are compared with the standard peak positions of stretching/bending vibrations of different organic compounds of jute [8].

## IV. DISCUSSION

Figures 4 to 9 show that a very strong and broad band in the range of 3600 to 3000  $\text{cm}^{-1}$  is due to the stretching vibration of the O-H group of cellulose, hemicellulose and lignin. The bands in the range 3000 to 2800  $\text{cm}^{-1}$  are related to stretching vibration of hydrocarbon groups, i.e.,  $\text{CH}_2$  and  $\text{CH}_3$  characteristic of lactic and glycolic acids. The peak at around 1730  $\text{cm}^{-1}$  is due to the stretching vibration of the carbonyl (C=O) group which is characteristic of cellulose and hemicellulose. The intensity and peak height of carbonyl group C=O increased by LTP treatment which indicates that the crystallinity of cellulose improved by LTP treatment [3]. The band near at 1650  $\text{cm}^{-1}$  is principally associated with absorbed water in jute fibre [7]. These absorptions are found decreasing after LTP treatment as the increase of discharge powers and exposure times, which are shown in figure 4 to figure 9. This may be due to the temperature effect by LTP, generated inside the reactor by glow discharge mechanism upon the jute surface. This may also be due to the bond cleavage due to plasma exposure to form free radicals [3]. It is also seen from the spectra that the absorptions of water for the case of  $\text{O}_2$  plasma are higher than that of Ar plasma. It might have occurred due to the oxidation of the jute surface during the LTP treatment by  $\text{O}_2$  plasma. Therefore, jute surface remains comparatively more hydrophilic after the treatment by  $\text{O}_2$  plasma.

The absorption peaks at 1589 and 1500  $\text{cm}^{-1}$  in all the spectra are due to benzene ring stretching. The absorption peak at 1440  $\text{cm}^{-1}$  in all the spectra are due to  $\text{CH}_2$  deformation in lignin. The peaks near 1358 and 1320  $\text{cm}^{-1}$  are possibly due to a  $\text{CH}_3$  bending and  $\text{CH}_2$  wagging in lignin. The band due to the C-O-C asymmetric stretching vibration for celluloses and hemicelluloses are observed at 1235  $\text{cm}^{-1}$ . The peak at 1235  $\text{cm}^{-1}$  corresponding to C-O-C is more prominent in  $\text{O}_2$  plasma compared to Ar plasma due to cellulose oxidation. Two peaks appear at 1077 and 1030  $\text{cm}^{-1}$  for C-O stretching and O-H deformation vibration respectively and are characteristic of cellulose and lignin. These bands are slightly shifted toward the higher wavenumber for the plasma treated jute. In addition, remarkable change in the relative peak intensity of band at 890  $\text{cm}^{-1}$  is found for the spectra of plasma treated jute fibre. This band is very prominent in the spectrum of raw fibre, which is attributed to linkage between the sugar units of hemicelluloses. But the reduction in the relative peak intensity of this band with the increase of discharge power and treatment time after LTP treatment of fibre may be due to the expansion in the single bond molecular chain caused by the heating effect of plasma radicals, which comes under energetic species, which energies are high enough to alter all chemical bonds in the surface layer of natural polymeric substance [10].

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