

# Variation of Electrical Conductivity with Frequency of Argon and Oxygen Plasmas Treated Jute

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**Abstract**— Jute fibers, a cellulosic and an environmentally friendly fibers, are treated with low temperature Argon (Ar) and Oxygen (O<sub>2</sub>) plasmas for 5, 10, and 15 minutes exposure times and at various discharge power levels of 50, 75 and 100 W with a flow rate of 0.2 L/min. Low temperature plasma treatment, a kind of environmentally friendly surface modification technique. The electrical conductivity of both raw and low temperature Ar and O<sub>2</sub> plasmas treated jute as a function of frequency were studied at room temperature. It is observed for all the types of samples that the conductivity increases as the frequency increases with a lower slope in the low frequency region and with a higher slope in the higher frequency region. In addition, the conductivity decreases with the increase of plasma exposure time as well as discharge power. The conductivity increases with frequency due to the hopping mechanism of electrons.

**Index Terms**— Electrical conductivity, Jute fibre, Plasma treatment, Exposure time and Discharge power.

## I. INTRODUCTION

In recent years, an increasing concern and awareness for the environment has given an impetus to research on lignocellulosic fibres for total or partial substitution of petroleum based synthetic fibres which are neither biodegradable nor renewable [1]. Plant fibres such as jute, hemp, flax, coconut fibre etc. have some interesting characteristics, e.g., cost effectiveness, renewable, available in huge quantities, low fossil-fuel energy and low cost compared to synthetic fibres such as glass, carbon etc. [2], [3], [4]. Thus, natural fibres have attracted attention for their applications in science and engineering, where lightweight is required. In addition to these advantages, there are also significant environmental advantages of using fibres that come from a continually renewable resource and utilize atmospheric carbon dioxide (CO<sub>2</sub>). The jute plant is defined as an environmental friendly crop because of its high CO<sub>2</sub> assimilation rate, which means that it cleans the air by consuming large amount of CO<sub>2</sub> that is a significant contributor to the green house effect. However, all plant fibres are hydrophilic and their moisture contents can reach up to 3%–13% [5], which limits their life span. The applications of natural fibres as reinforcements in composite materials require a strong adhesion between fibre and the synthetic matrix. But the hydrophilic nature of natural fibre causes weakening in the adhesion. Physical and chemical treatments can be used to optimize this interface [6], [7].

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The literature abounds with references to the surface modification of fibres by chemical, physical and physiochemical means [8], [9]. Traditionally, fibres are modified by chemical methods. These processes may imply numerous chemical substances, some of them being toxic for human and unsafe for environment. Additional problems appear also because of degradation and weakening of the treated fibres. Other disadvantages are high costs and sometimes a high process temperature [10]. However, in recent years, increasing concern about environmental pollution problems has limited wide industrial application of chemical surface treatments.

In contrast to chemical treatments, the cold plasma techniques are considered as dry and clean processes [11]. In the past few years interest has increased in the use of low temperature plasma (LTP) technique which is a promising approach for surface modifications of human made as well as natural fibres [12]. As a type of environmentally friendly physical surface modification technique, LTP treatment is one of the methods used to modify surfaces in a dry process [13].

Advantages of this technique, compared to a conventional wet process, are: (i) because of the very thin treatment layer, only the surface is modified without interfering the bulk properties and (ii) the process is simpler- fewer steps and less time are required, involving no chemicals. Inert gases, such as, Argon (Ar), Neon (Ne) and Helium (He) plasmas can cause some chemical and physical reactions on the surface of substrates because of highly energetic species such as free radicals, ions, photons and ultraviolet (UV) radiations [14]. The high-energy electrons and low-energy molecular species in non-thermal plasma can initiate reactions in the plasma volume without excessive heat causing substrate degradation [15]. Non-thermal plasmas are particularly suited to apply to textile processing because most textile materials are heat sensitive polymers. In addition, it is a versatile technique, where a large variety of chemically active functional groups can be incorporated into the textile surface. Plasma treatment is controlled by applied power for gas discharge, nature of the gas, position of the fibres inside plasma and exposure time. Plasma treatments using an inert gas such as Ar, effect on the fibre surface by physically sputtering and chemical etching. A lot of literatures have been published on the plasma treatment of natural fibres for improving surface, mechanical, physical and thermal properties as well as composites where jute was used as a reinforcing material. However, research work so far has yet not done on comparative study concerning the impact of plasma treatment upon the changes of alternating current electrical conductivity and dielectric behavior of plasma treated jute as a function of frequency. In the present work, jute fibres were treated with low temperature Ar and O<sub>2</sub> plasmas using different discharge powers and exposure times. The ultimate goal of the research work was designed to inspecting the electrical conductivity as well as the electrical

conduction mechanism in jute as a function of frequency at room temperature.

### 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 was 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.

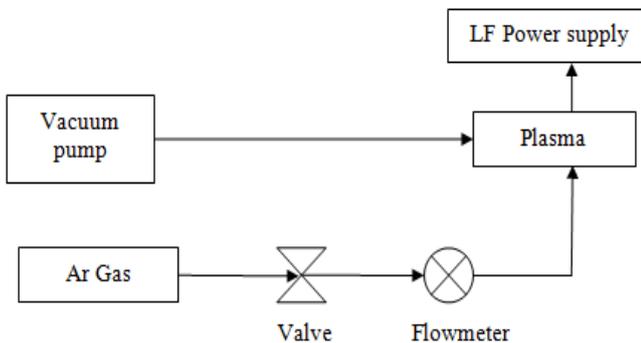


Fig. 2 Flow chart of the plasma treatment set-up

After plasma treatment has been finished, and the vacuum chamber was vented, jute samples were then removed and handled carefully in order to avoid possible surface contamination to the fibres. 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. The powdered form jute of about 200 mg. was then put in a specially prepared high-pressure die. In order to make the tablets from jute powder, a high pressure (14000 psi) was applied by a hydraulic press (Model: X30659, 0-16000 psi, Mold Pressure, P.S.I: 1" and 5/4" Mold, Will Corporation, NY, USA). The diameter and the thickness of each equipped tablet was 13.5 and 1.5 mm respectively. In this way twenty five types tablets (one tablet was for raw jute and another twelve were for LTP treated jute) were prepared with treated jute samples of different discharge powers and exposure times. All the tablets were oven-dried at 100 °C for 20 minutes before characterization of the samples.

#### C. AC electrical measurements

The dielectric measurements were carried out at room temperature using a Precision Impedance Analyzer (Model: 6500B, Wayne Kerr, Made in UK) (figure 3) over the frequency range 100 Hz-120 MHz. The frequency dependent values of parallel capacitance ( $C_p$ ) and conductance ( $G_p$ ) of the tablet formed jute samples were noted directly at different frequencies at room temperature

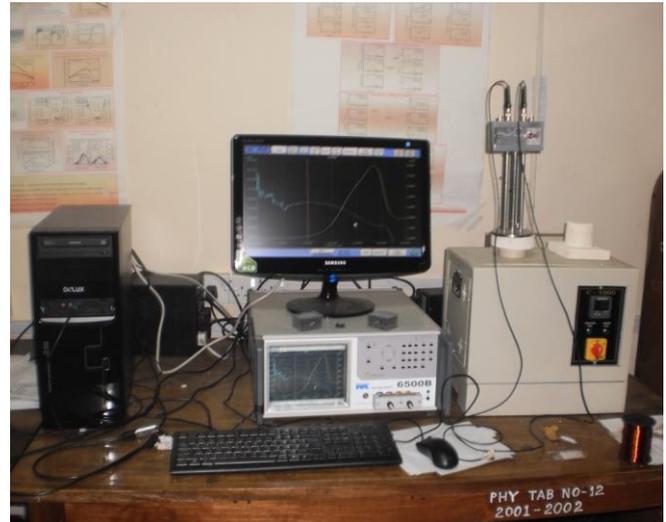


Fig. 3 Arrangement for AC measurement

#### Calculation of AC conductivity of different types of jute samples

The AC conductivity ( $\sigma_{ac}$ ), of the tablet formed jute samples can be calculated using the relation,

$$\sigma_{ac} = \frac{G_p d}{A}$$

where,  $G_p$  is the parallel conductance (in Siemens),  $d$  is the thickness (in m) of the tablet and  $A$  is the cross-sectional area (in  $m^2$ ) of the tablet.

III. RESULTS

The electrical conductivity,  $\sigma_{ac}$ , against frequency,  $f$ , curves of raw and LTP treated jute for different gases, exposure times and discharge powers are shown in figure 4

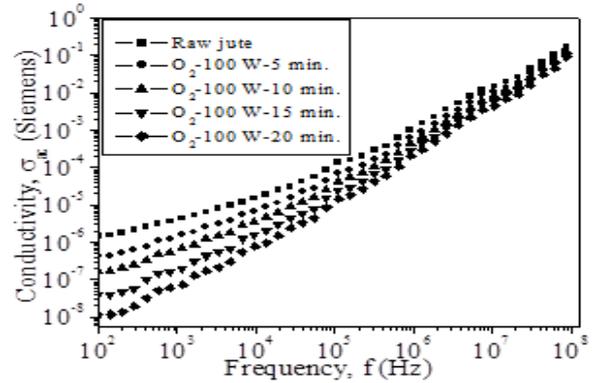
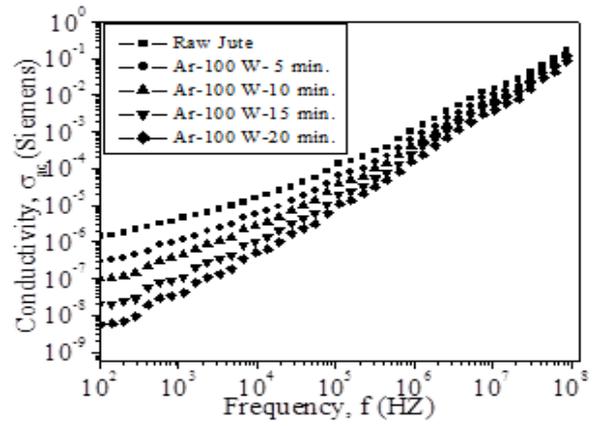
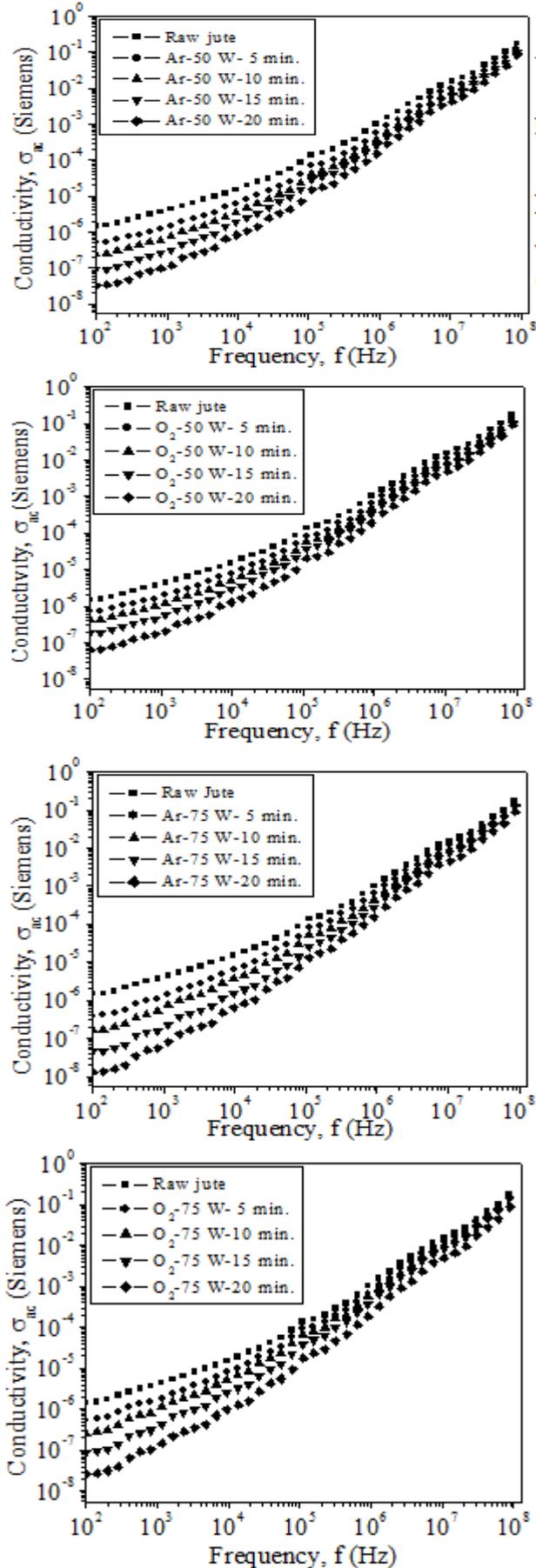
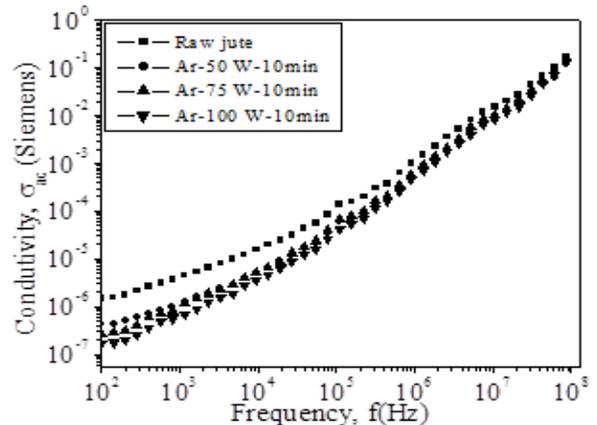
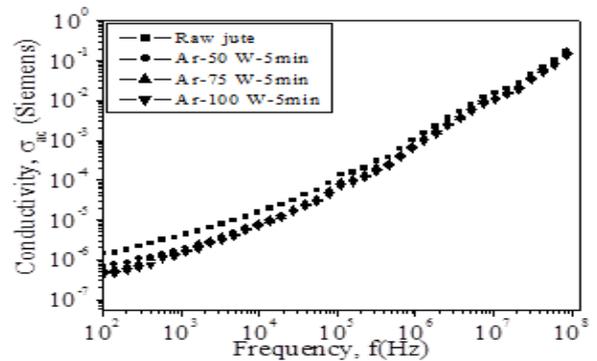


Fig. 4(a) Electrical conductivity,  $\sigma_{ac}$ , as a function of frequency of raw jute and LTP treated jute for various treatment times and discharge powers of Ar and O<sub>2</sub> plasmas.



## Variation of Electrical Conductivity with Frequency of Argon and Oxygen Plasmas Treated Jute

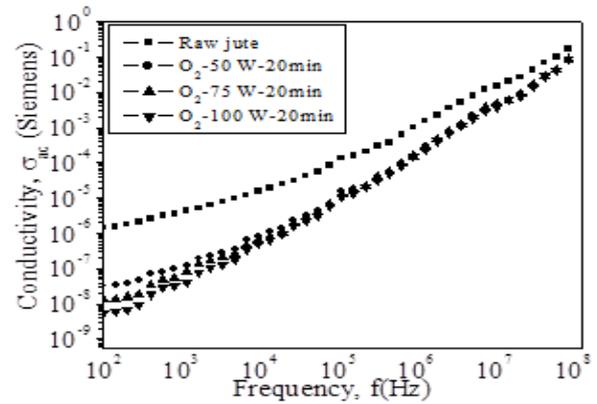
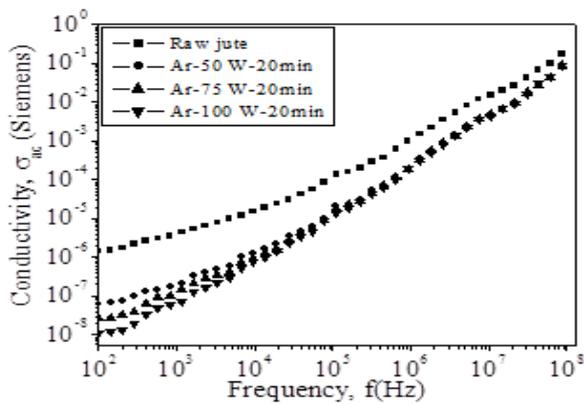
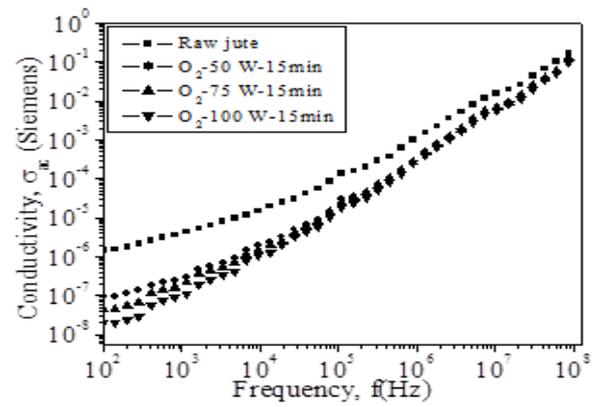
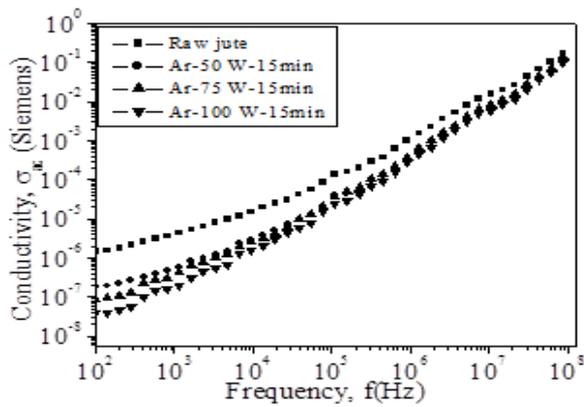


Fig. 4(b) Electrical conductivity,  $\sigma_{ac}$ , as a function of frequency for raw jute and LTP treated jute for various discharge powers at a constant treatment time of Ar plasma.

Fig. 4(c) Electrical conductivity,  $\sigma_{ac}$ , as a function of frequency of raw jute and LTP treated jute for various discharge powers at a constant treatment time of O<sub>2</sub> plasma.

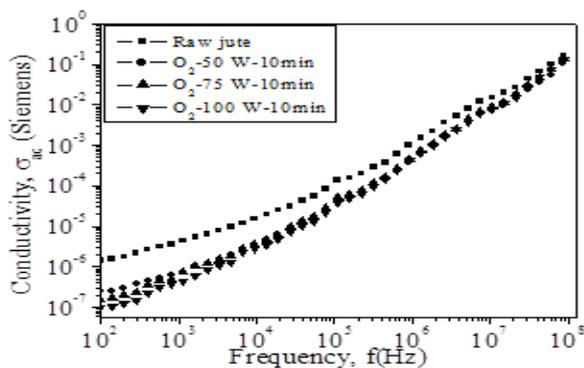
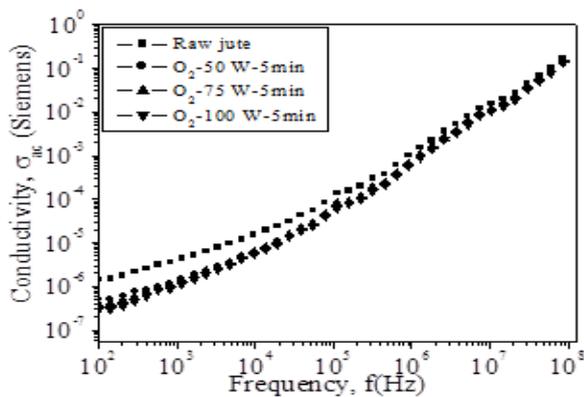


Table 1 Values of  $\sigma_{ac}$ , at various power and time at 1 kHz and 1 MHz

Power (in Watt)	$\sigma_{ac}$ (in micro Siemens) (for 1 kHz)							
	5 min.		10 min.		15 min.		20 min.	
	Ar	O <sub>2</sub>	Ar	O <sub>2</sub>	Ar	O <sub>2</sub>	Ar	O <sub>2</sub>
50	1	2	0.7	1.1	0.3	0.5	0.1	0.2
75	1	2	0.6	0.9	0.2	0.4	0.1	0.1
100	1	1	0.4	0.6	0.1	0.2	0	0.1

Power (in Watt)	$\sigma_{ac}$ (in micro Siemens) (for 1 MHz)							
	5 min.		10 min.		15 min.		20 min.	
	Ar	O <sub>2</sub>	Ar	O <sub>2</sub>	Ar	O <sub>2</sub>	Ar	O <sub>2</sub>
50	590	666	397	513	291	361	176	222
75	700	753	468	597	300	400	167	202
100	627	668	444	479	274	316	170	216

## IV. DISCUSSION

It is observed from figure 4 that  $\sigma_{ac}$  increases as frequency increases with a lower slope in the low frequency ( $<10^5$  Hz) regions for all the samples. It is also seen from the above curves that  $\sigma_{ac}$  increases as frequency increases with a higher slope in the high frequency regions (above  $10^5$  Hz) for all the samples. Such behaviour can be described by the relation [16]

$$\sigma_{ac}(\omega) = A\omega^n$$

where,  $A$  is a proportionality constant,  $\omega (=2\pi f)$ ,  $f$  is the linear frequency) is the angular frequency and  $n$  is the exponent, which generally takes the value less than unity for Debye type mechanism and is used to understand the conduction/relaxation mechanism in polymeric materials.

It is also seen from figure 4 and table 1 that the values of  $\sigma_{ac}$  decrease as the exposure time as well as the discharge power increase. The reasons behind the decrease of  $\sigma_{ac}$  with the increase of exposure time and discharge power may be explained as follows

The lignocellulosic jute fibres have an affinity to water and are usually charged or have polar side groups to their structure that attract water. Due to the high content of hydroxyl and carboxyl groups on cellulose and hemi-cellulose structure, jute fibres are hygroscopic and hydrophilic in nature. The inherent polar and hydrophilic nature of jute fibres can absorb moisture from the atmosphere [17], [18]. When jute fibres are exposed to LTP, energetic charged particles inside the plasma are able to interact both physically and chemically with the surface to be treated. Such interactions can also affect the fibre properties and the moisture content of the treated jute decreases due to the surface modification of jute. These in turn, jute fibres become hydrophobic and the  $\sigma_{ac}$  decreases as the exposure time and the discharge power increase. It is also seen from table 1 that at various discharge powers and exposure times the  $\sigma_{ac}$  of jute fibre treated by  $O_2$  plasma is higher than that of jute treated by Ar plasma. This may due to the oxidation of the surface of lignin and hemicellulose when the jute treated by  $O_2$  plasma. Chemically jute possesses high content of semicrystalline and amorphous materials, such as cellulose, hemicellulose and lignin. Moreover, due to the presence of the hydroxyl and carboxyl groups on the fibre surface and in the amorphous region, the jute fibres can absorb moisture from the atmosphere under standard conditions of temperature and pressure [19]. Therefore, the jute are hygroscopic as well as hydrophilic in nature. When jute is exposed to LTP condition, energetic charged particles inside the plasma are able to interact chemically with the surface to be treated. Such interactions can also affect the material properties and the moisture content of the treated jute decreases due to the surface modification of the jute fibres. In the LTP process, the water ( $H_2O$ ) dissociates into H and OH species by energetic gaseous ion bombardment. The temperature sensitive jute was dried more effectively in plasma without damaging its constituents and also improved the crystallinity of jute [20], [21]. Therefore,  $\sigma_{ac}$  decreases with an increase of exposure times and discharge powers. It is also seen from the figures 4 that at various discharge powers and exposure times, the  $\sigma_{ac}$  of jute treated by  $O_2$  plasma is higher than that of jute treated by Ar plasma. This may due to the oxidation of the surface of lignin and hemicellulose when the jute treated by  $O_2$  plasma. In addition, cellulose-based jute are intrinsically polar in nature owing to the presence of hydroxyl and carboxyl groups in their structure. The  $O_2$  plasma treatment of the jute may lead to an increase in the polar component mainly due to the increase of the content of hydroxyl and carboxyl groups. Thus, the jute treated by  $O_2$  plasma is relatively less dry compared to the Ar plasma

treated jute. That is why  $\sigma_{ac}$  of jute treated by  $O_2$  plasma is higher than that of Ar plasma.

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#### REFERENCE

- [1] E. Sinha, S. K. Rout and P. K. Barhai, "Study of the structural and thermal properties of plasma treated jute fibre", *J. Appl. Phys. A*, Vol. 92, 2008, pp. 283-290.
- [2] R. R. Atkinson, "Jute fibre to yarn", Chemical Publishing Company, New York, 1995, pp. 38-47.
- [3] T. C. Ranjan, "Handbook on jute", Oxford and IBH Publishing Com., New Delhi, 1973, pp. 55-69.
- [4] E. Sinha, "Effect of cold plasma treatment on macromolecular structure, thermal and mechanical behavior of jute fiber", *J. Indust. Text.*, Vol. 38, 2009, pp. 317-338.
- [5] Morton, W. E., and Hearle, J. W. S., 1974. *Physical Properties of Textile fibre*, The Textile Institute, Manchester, U.K, 122-132.
- [6] Gowda, T. N., Naidu, A. C. B. and Chaya, R., "Some mechanical properties of untreated jute fabric reinforced polyester composites", *Compos. Sci. Technol.*, Vol. 30, pp. 277-284, 1999.
- [7] Mohanty, A. K., Khan, M. A. and Hinrichsen, G., "Surface modification of jute and its influence on performance of biodegradable jute fabric/biopole composites", *Compos. Sci. Technol.*, Vol. 60, pp. 1115-1124, 2000.
- [8] Mohanty, A. K., Khan, M. A., Sahoo, S., Hinrichsen, G., "Effect of chemical modification on the performance of biodegradable jute yarn/bipol composites", *J. Mater. Sci.*, Vol. 35, pp. 2589-2595, 2000.
- [9] Moore, W. R., "An Introduction to Polymer Chemistry", Al-din Publishing Com. Chicago, Vol. 14, pp.215-17, 1990.
- [10] Browns, P.B., "The Chemistry of Lignin", Academic Press, New York, p. 242, 1952.
- [11] Biederman, H. and Osada, Y., "Plasma Polymerization Process", Elsevier Science, New York, 1992.
- [12] Yasuda, H., "Plasma Polymerization", Academic Press, New York, 1985.
- [13] Liston, E.M., Martinu, L., and Wertheimer, M.R., "Plasma Surface Modification of Polymers", Chapman and Hall: New York, 1994.
- [14] Inagaki, N., "Plasma Surface Modification and Plasma olymerization", Technomic Publishing Company: Lancaster, PA, 1996.
- [15] N. K. Pandiyaraj and V. Selvarajan, "Non-thermal plasma treatment for hydrophilicity improvement of grey cotton fabrics", *J. Mater. Process. Technol*, Vol. 199, 2007, pp. 130-139.
- [16] Mott, N.F. and Davis, E.A., "Electronic Processes in Non-Crystalline Materials", Clarendon Press, Oxford, 1971.
- [17] M. M. Morshed, M. M. Alam, and S. M. Daniels, "Plasma treatment of natural jute fibre by RIE 80 plus plasma tool", *Plasma Sci. Technol.*, Vol. 12, 2010, pp. 325-329.
- [18] G. Socrates, "Infrared and Raman Characteristic Group Frequencies", Wiley, West Sussex, England (3rd ed.), 2001pp. 58-62.
- [19] H.Yasuda, *Plasma Polymerization*, Academic Press, New York, 1985.
- [20] H. S. Sabharwol, F. Denes, L. Nielsen and R. A. Yuong, "Free-radical formation in jute from plasma argon treatment", *J. Agri. Food. Chem.*, Vol. 41, 1993, pp. 2202-2207.
- [21] B.K. Sharma, "Spectroscopy", Krishna prakashna Media Ltd, Meerut, India, 1993.