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Alternating current electrical properties of argon plasma treated jute

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Abstract

Low temperature plasma (LTP) treatment, a kind of environment friendly surface modification technique, was applied to biodegradable and environment friendly jute fibre with the use of nonpolymerizing gas, namely argon, at various discharge power levels and exposure times with a definite flow rate. Scanning electron microscopy (SEM) microphotographs reveal that the roughness of the fibre surfaces increases with the increase of discharge power and exposure time. This is caused due to the bombardment of high energetic ions on the fibre surface and the fibres become sputtered. The capacitance and the electrical conductance of raw and LTP treated jute fibre were measured as a function of frequency at room temperature. The dielectric constant, conductivity, dielectric loss-tangent and the surface morphology of raw and LTP treated jute as a function of frequency were studied at room temperature. It was observed that for all the samples the dielectric constant almost constant at lower frequencies and then decreases gradually in the high frequency region. In addition, dielectric constant increases with the increase of plasma treatment time as well as discharge power. It is also observed for all the 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. The dependence of the dielectric loss-tangent with frequency at different treatment times and discharge powers for all the jute samples show small relaxation peaks in the very low frequency region. The dielectric loss-tangent decreases with the increase of both plasma treatment time and discharge power. In addition, the relaxation peaks are shifted to the higher frequency region as the plasma treatment time as well as discharge power increases. At the low frequencies relaxation peaks indicate the possibility of interfacial polarization.

Keywords: Jute, Plasma treatment, Dielectric constant, Conductivity, Loss-tangent

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1. 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 (Sinha et al., 2008). 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. (Mishra et al., 2003). 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_2). The jute plant is defined as an environmental friendly crop because of its high CO_2 assimilation rate, which means that it cleans the air by consuming large amount of CO_2 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% (Bledzki et al., 1990), 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 (Bledzki and Gassan, 1999). The literature abounds with references to the surface modification of fibres by chemical, physical and physiochemical means (Mwaikambo and Ansell, 1999; Modibbo et al., 2007; Corrales et al., 2007; Gassan and Bledzki, 1999 and Mukherjee et al., 1993).

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 (Luciu et al., 2008). 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 (Seki et al., 2010). 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 (Seki et al., 2010 and Qiu et al., 2002). 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 (Demir et al., 2011; Pandiyaraj and Selvarajan, 2008). 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 (Iriyama et al., 1990). 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 (Yasuda, 1985). 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 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 plasma using different discharge powers and exposure times. The ultimate goal of the research work was designed to inspecting jute surfaces at very high magnifications of the LTP treated jute fibre by scanning electron microscopy (SEM), electrical conductivity as well as the electrical conduction mechanism in jute, dielectric constant and dielectric loss-tangent as well as their mechanism as a function of frequency at room temperature.

2. Experimental details

2.1. Low temperature plasma treatment

Jute fibres were collected from the local market in Bangladesh. The jute fibres were introduced into a bell jar type capacitively coupled glow discharge reactor, which was made up of a cylindrical Pyrex glass bell-jar having 0.15 m in inner diameter and 0.18 m in length. It consists of a pair of circular stainless steel electrodes with their planes parallel to each other and perpendicular to the axis of the chamber. To sustain a glow discharge i.e. for getting proper and uniform plasma, the conductive electrodes separated 0.035 m apart from each other and were connected to high-tension AC power supply (3.5 kV, 2.8 kW). In order to exposed all through uniform LTP treatment on the samples surface, the fibres (length of each fibre: 0.08 m) were inserted between the two metallic electrodes by a carrier. After placing the jute fibres between the pair of electrodes, the glow discharge chamber was evacuated by a rotary pump (Vacuubrand, Vacuubrand GMBH and Co, Germany) at a pressure of 1.33Pa. Ar was considered as plasma gas for treating the jute fibre. The discharge powers were adjusted at 50, 75 and 100 W at a line frequency of 50 Hz with the duration of exposure times were 5, 10, 15 and 20 min. of LTP treatment of the surface of jute fibres. In all treatments, Ar gas was introduced into the reaction chamber by a flowmeter (Model: 1355K4BQ8F6CG, Emerson electric, Pennsylvania, USA) at a flow rate of 0.2 L/min. which was maintained by a needle valve. Figure1 shows a flow-chart of a plasma treatment system which was used in this experiment.

After plasma treatment has been finished, the vacuum chamber was vented. Jute samples were then removed and handled carefully in order to avoid possible surface contamination to the fibres. The plasma treated fibres were immediately placed into a desiccator with silica gel.

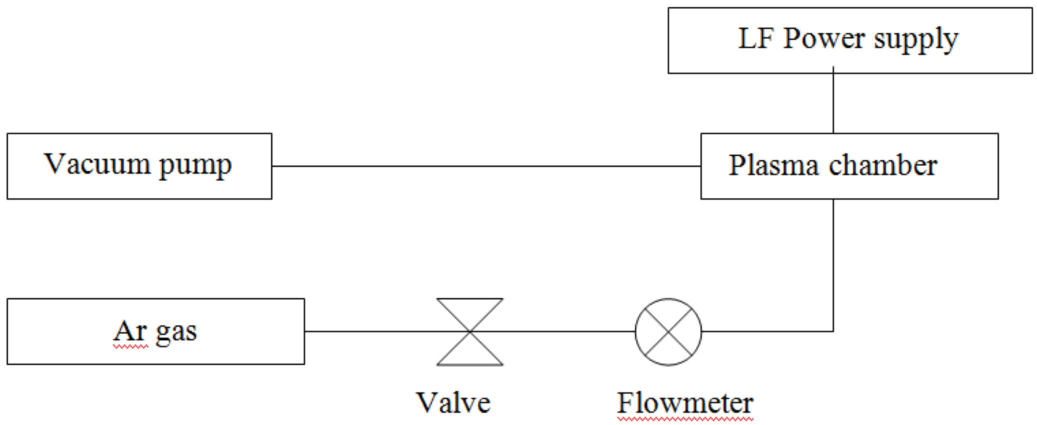

Figure 1. A schematic diagram of the plasma treatment set-up

2.2. Characterization of fibres

2.2.1. Scanning electron microscope

The surface morphology of the raw and LTP treated jute fibres were examined with a scanning electron microscope (Model: S50, FEI Qunta Inspect, The Netherlands) operated at 25 kV in low vacuum control at 50 Pa. The surfaces of the jute fibres were examined at 2000× magnification.

2.3. Sample preparation for the electrical measurements

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 seived 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 1.5mm respectively. In this way thirteen 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. For the dielectric parameters measurements suitable electrical contacts were made by coating opposite faces with silver paste. Thereby forming parallel plate capacitor geometry. All the tablets were oven-dried at 100 °C before characterization of the samples.

2.4. Electrical measurements

The dielectric behavior of a material is usually described by using the equation $\varepsilon^*(\omega, T) = \varepsilon' - j\varepsilon''$, where ε^* is the complex dielectric permittivity, ε' is the real part of complex dielectric permittivity and simply called the dielectric constant and ε'' is the imaginary part of the complex dielectric permittivity and is equal to $\varepsilon'\tan\delta$ where, $\tan\delta$ is the loss-tangent or dissipation factor.

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

2.4.1. Dielectric constant

The dielectric constant (ε_r') was calculated using the relation

$$\varepsilon_r' = \frac{C_p d}{A\varepsilon_0}$$

where, C_p is the parallel capacitance (in F), ε_0 is the permittivity of free space (8.854×10^{-12} F/m), d is the thickness (in m) of the tablet and A is the cross-sectional area (in m^2) of the tablet.

2.4.2. A.C Conductivity

The AC conductivity (σ_{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.

2.4.3. Dielectric loss-tangent

The dielectric loss-tangent ($\tan\delta$) can also be calculated using the relation

$$\tan\delta = \frac{G_p}{2\pi f C_p}$$

where, C_p and G_p are the parallel capacitance (in F) and parallel conductance (in Siemens) respectively, f is the applied frequency.

3. Results and discussion

3.1. Surface morphology

SEM microphotographs of raw jute fibre and LTP treated jute fibres by Ar gas at various discharge powers (50, 75 and 100 W) and exposure times (5, 10, 15 and 20 min.) were taken in this experimental work. Only raw jute and 10, 20 min. LTP treated jute with 50 and 100 W discharge powers are presented here in figure 2(a)-(e).

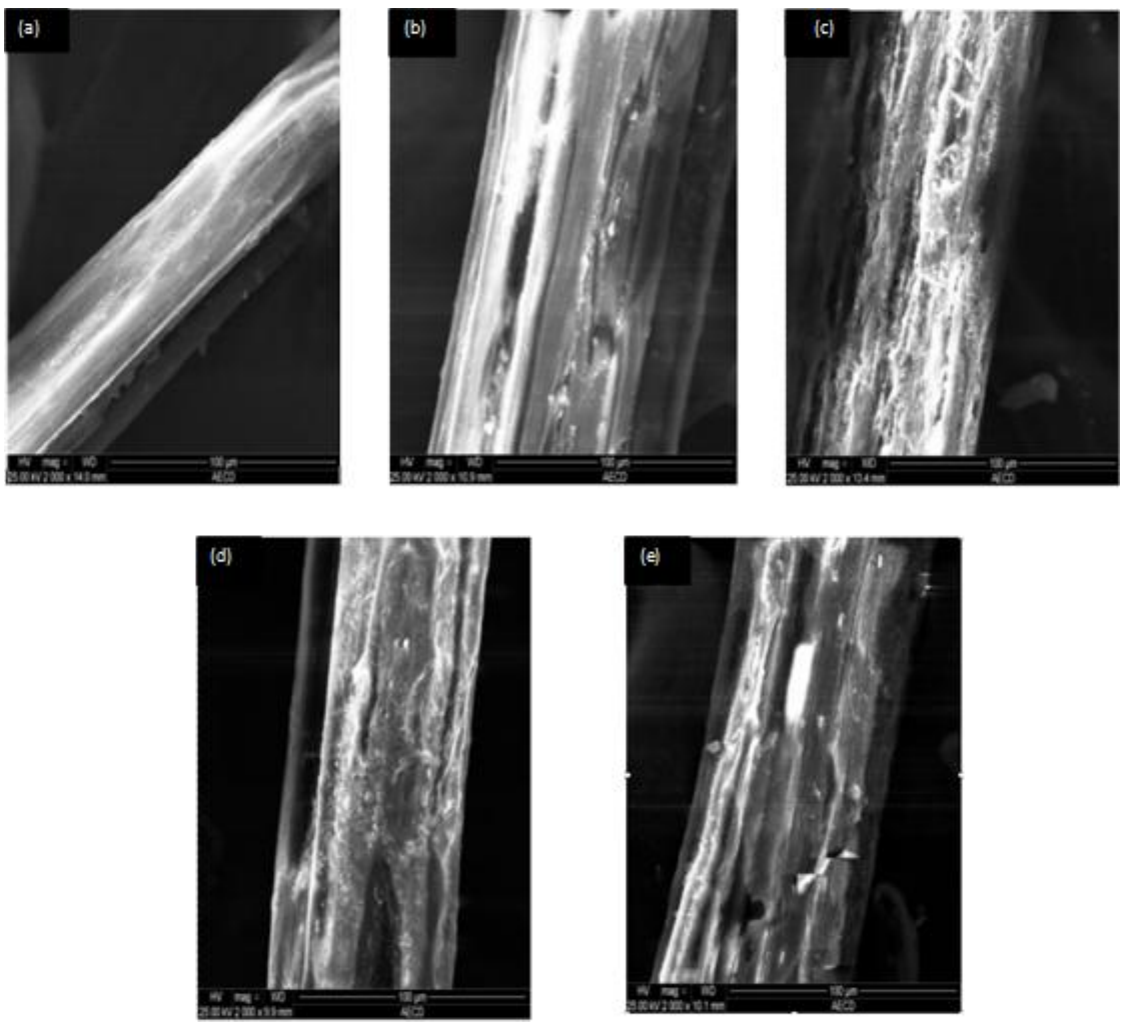

Figure 2. SEM microphotographs of different jute fibres (a) raw jute (b) 50 W, 10 min. (c) 50 W, 20 min. (d) 100 W, 10 min. and (e) 100 W, 20 min. LTP treated jute fibre by Ar gas

It can be visually verified that the surface of raw jute at figure 2(a) is smooth and shows no roughness. Figure 2(b)-(e) illustrate the action of plasma treatment with Ar on the fibre surface for different exposure times and discharge powers. Figure 2(b)-(c) associated with 50 and 100 W discharge powers of 10 min. treatment time presents a surface like rough and fragmented. Figure 2(d)-(e), associated with 50 and 100 W discharge powers of 20 min. treatment time shows more roughness and also shows degradation on fibre surface. Besides, figure 2(e) shows the formation of pits on the fibre surface, likely by the ablation (sputtering and etching) mechanism of plasma causing more degradation on the fibre surface (Yasuda, 1985).

3.2. Variation of dielectric constant with frequency

Figure 3 is the representative graph of the variation of dielectric constant with frequency of raw jute and LTP treated jute with different exposure times at 50 W.

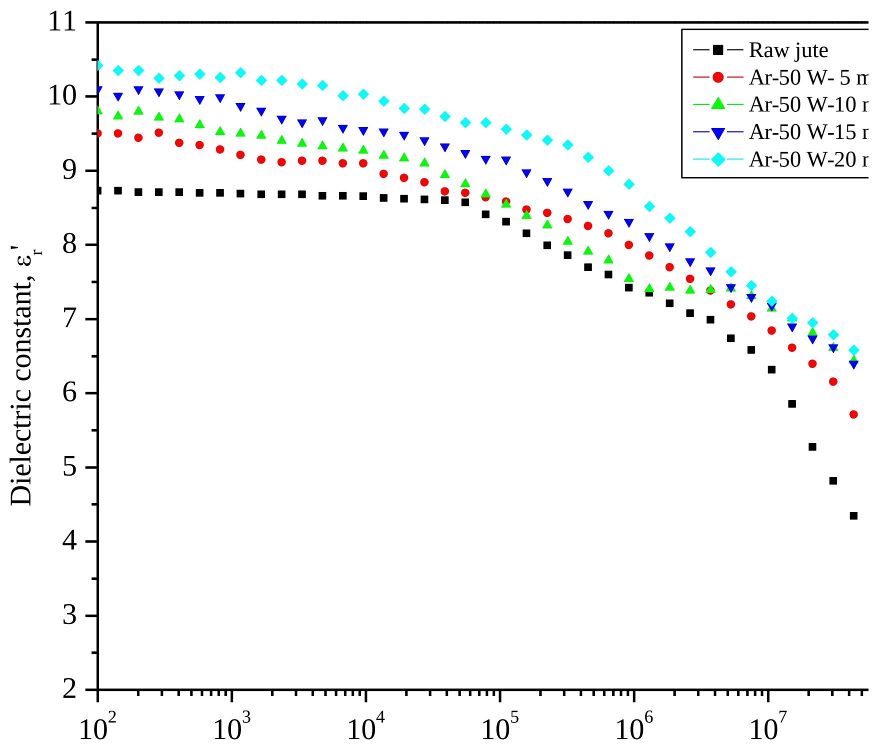

Figure 3. Variation of dielectric constant, ε_r', with frequency, f of various treatment times for 50 W

Figure 4 shows the representative graph of dielectric constant versus frequency of raw jute and LTP treated jute with different discharge powers at 20 min. exposure time.

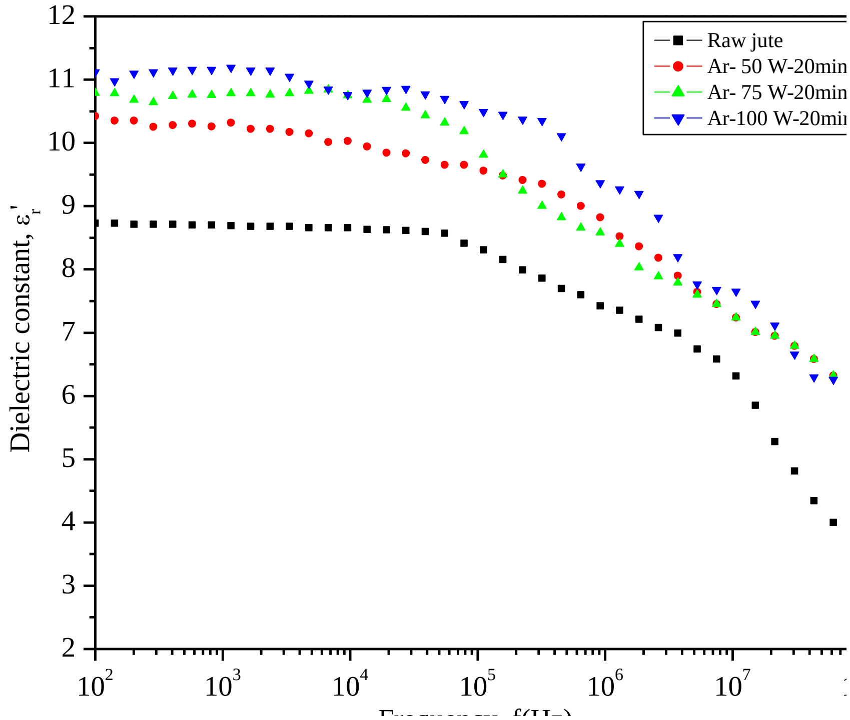

Figure 4. Dielectric constant, ε_r', vs. frequency, f curve of raw jute and LTP treated jute with various discharge powers for 20 min. exposure time

Table 1 represents the values of dielectric constant at various discharge powers and LTP exposure time at 1 kHz and 1 MHz frequency.

Table 1. Values of dielectric constant, ε_r', at various power and time at 1 kHz and 1 MHz frequency

Power (in Watt)	ε_r' (for 1 kHz frequency) (for1 kHz)				ε_r' (for 1 MHz frequency) (for1 MHz)			
	5 min.	**10 min.**	**15 min.**	**20 min.**	**5 min.**	**10 min.**	**15 min.**	**20 min.**
50	9.26	9.59	9.90	10.29	7.90	8.05	8.25	8.75
75	9.8	9.86	10.33	10.75	8.42	9.46	9.72	9.86
100	9.86	10.33	10.75	11.17	9.46	9.72	9.86	9.95

It is seen from figure 3 and figure 4 that the values of dielectric constant of both raw jute and LTP treated jute almost constant in the low frequency region (<10^5 Hz) and above this frequency dielectric constant falls gradually with the increase of frequency. It is also seen from the above figures that the values of dielectric constant of LTP treated jute increases compared to the raw jute sample. Furthermore, with the increase of plasma treatment time and the discharge power the values of the dielectric constant increases gradually within the measured range of frequency, which can be seen in table1 too. The maximum value of dielectric constant for raw jute sample is 8.75. In addition, the values of dielectric constant of LTP treated jute lie between 9.0 and 10.50 for 50 W, 9.75 and 10.75 for 75 W and 10.1 and 11.1 for 100 W discharge powers.

The longer the duration of physical sputtering of LTP treatment, the more severe the modification of the fibre surface which are shown by SEM microphotographs (figure 2). In addition, the higher the discharge power applied the more kinetic energy the plasma species will carry. Hence, there will be a change in the total amount of the excited particles inside the plasma and their energy level accordingly. As a result strong intensity of plasma action happened on the fibre surfaces (Sinha, 2009 and Bozaci et al., 2009). It is noted that jute fibre contains organic molecules. Gaseous plasma may introduce degradation and chain scission in fibre (Morshed et al., 2010). Plasma gas may also react with some polymer constituents inside the fibre. These effects may increase the dipole rotation of the dielectric according to the increase of exposure times as well as discharge powers. These may cause dielectric constant of LTP treated jute to increase with the increase of exposure time and discharge power.

The greater the polarizability of the molecules, the higher is the dielectric constant of the material. The dependence of ε on frequency might be explained with space charge accumulation at the structural interface. The charges present by the plasma treatment on the surface of the tablet formed jute sample can migrate under the influence of an electric field. As the frequency increases, dipoles begin to lag behind the field, and ε gradually decrease. It is possible that they get blocked or cannot discharge at the electrode dielectric interface, which leads to space charge or interfacial polarization.

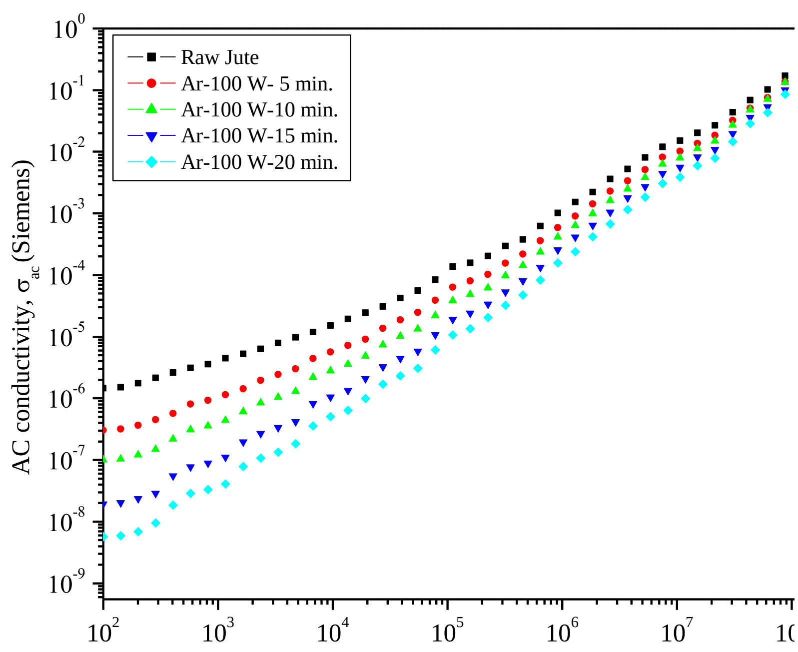

Figure 5. Conductivity, σ_{ac}, as a function of frequency, f of the raw and LTP treated jute for 100 W

3.3. Variation of electrical conductivity with frequency

The conductivity, σ_{ac}, against frequency (from 100 Hz to 120 MHz) curve of raw and LTP treated jute for the case of 100 W discharge power at various exposure times are shown in figure 5 as a representative one.

Figure 6 displays the representative graph of σ_{ac} versus applied frequency of raw jute and LTP treated jute with different discharge powers and at 15 min. exposure times.

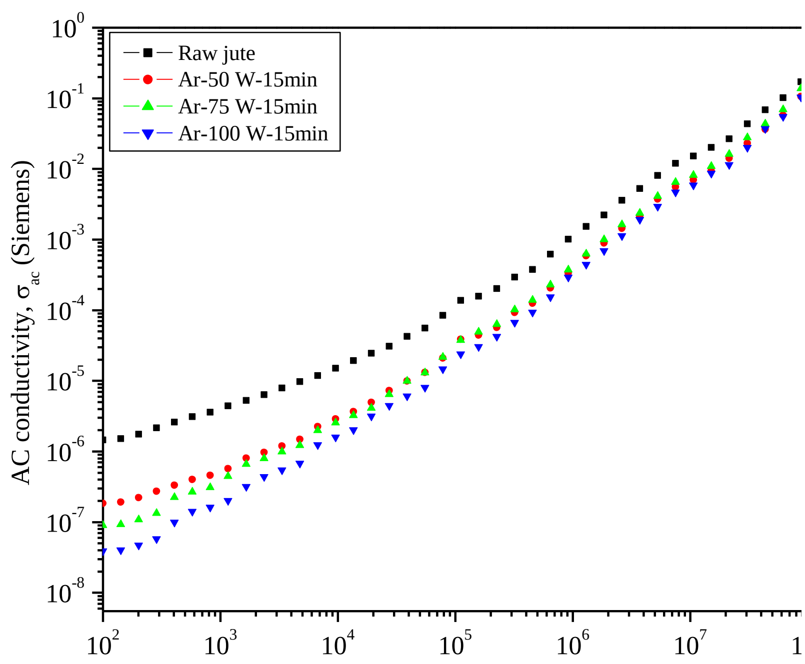

Figure 6. Conductivity, σ_{ac}, as a function of frequency, f of the raw and LTP treated jute at various discharge power for 15 min. exposure time

Table 2 represents the values of conductivity at various discharge power and LTP exposure time at 1 kHz and 1 MHz frequency.

Table2. Values of Conductivity, $\sigma_{ac,}$ at various power and time at 1 kHz and 1 MHz frequency

Power (in Watt)	σ_{ac} (in micro Siemens) (for1 kHz frequency)				σ_{ac} (in micro Siemens) (for 1 MHz frequency)			
	5 min.	10 min.	15 min.	20 min.	5 min.	10 min.	15 min.	20 min.
50	1.861	1.116	0.530	0.198	666.698	513.357	361.404	222.178
75	1.736	0.927	0.355	0.120	753.564	597.199	400.363	202.774
100	1.459	0.640	0.187	0.068	668.485	479.078	316.457	216.645

It is observed from figure 5 and figure 6 that the σ_{ac} increases as applied frequency increase 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 σ_{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 (Mott and Davis, 1971)

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

where A is a proportionality constant, ω (=$2\pi f$, 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 5, figure 6 and table 2 that the values of electrical conductivity decrease as the exposure time as well as the discharge power increase. The reasons behind the decreases of electrical conductivity 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 (Modibbo et al., 2007). 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 fibre decreases due to the surface modification of the fibres. In the LTP process, water (H_2O) dissociates into H and –OH species by energetic gaseous ion bombardment. The temperature sensitive jute fibres were dried more effectively in plasma without damaging its constituents and also improved crystallinity of the fibres (Morshed et al., 2011). Furthermore, by the LTP process the temperature increase inside the plasma reactor gradually with the increase of both treatment time as well as discharge power. These in turn, jute fibres become hydrophobic and the electrical conductivity decreases as the exposure time and the discharge power increase.

3.4. Variation of dielectric loss-tangent with frequency

Figure 7 is the representative graph of the variation of dielectric loss-tangent with applied frequency of raw jute and LTP treated jute with different exposure times and for 75 W. Figure 8 displays the representative graph, which shows the dependence of tanδ on the applied frequency of raw jute and LTP treated jute with different discharge powers for 10 min. exposure time.

It is seen from the figure 7 and figure 8 that the values of loss-tangent of raw and LTP treated jute increases as the applied frequency increase and reaches a maximum within the frequency range of 10^3 to 10^4 Hz. Above this frequency range the loss-tangent has a decreasing tendency up to the frequency range of 10^5 Hz and then increases gradually with the increase of frequency. When the hopping frequency is nearly equal to the frequency of externally applied electric field, a relaxation peak is observed. The increase of dielectric loss in low frequency region is dominated by interfacial or ionic polarization. It is also observed from the figure 7 and figure 8 that the values of loss-tangent decreases as the exposure times as well as the discharge power increase. This is the good agreement with the results found by electrical conductivity analysis in the previous section. In addition, it is seen from the figure 7 and figure 8 that the relaxation peaks are broaden and they are shifted towards the higher frequency side with the increase of exposure time and discharge power. This may be due to the increases of dipole rotation of dielectric material with the increase of exposure times and discharge powers.

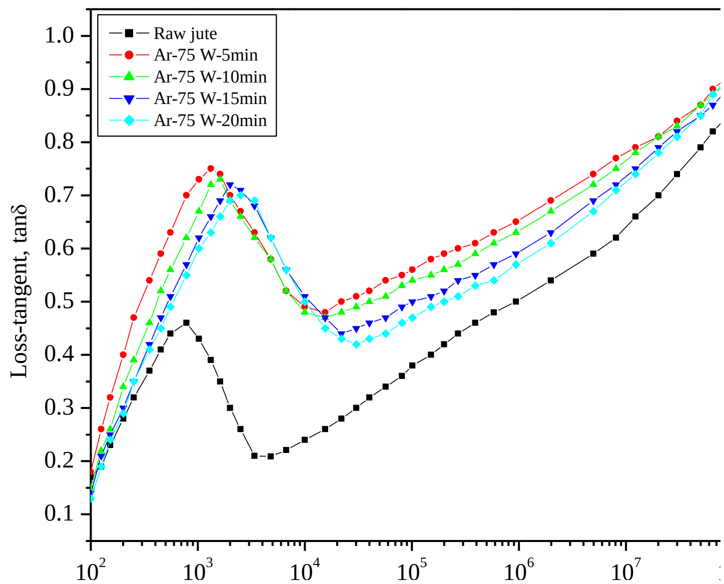

Figure 7. Variation of dielectric loss-tangent, tanδ, with frequency, f at various treatment times for 75 W

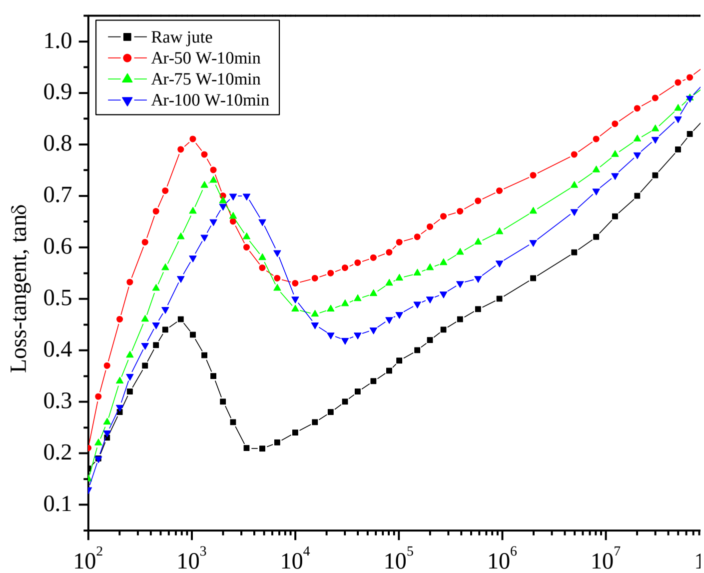

Figure 8. Variation of dielectric loss-tangent, tanδ, with frequency, f at various discharge powers for 10 min. exposure time

4. Conclusions

Once the plasma power was applied, surface morphological changes observed on the fibre surface with exposure times were obviously evident according to the treatment time and discharge power. Because of the physical sputtering and chemical etching effect, surface roughness of the fibres increases with increase of discharge powers and exposure times. LTP treated jute fibres by Ar plasma have high dielectric constant (9-11), and almost constant up to the frequency 10^5 Hz. So it may be a potential candidate to be used in power capacitor, electrical cables or in microelectronics in the frequency region $<10^5$ Hz. The conduction mechanism may be dominated by hopping of carriers between localized states. Conductivity of jute fibre decreases with the increase of plasma discharge power as well as treatment time. This happens because of the LTP process is a candidate process for moisture removal. Because of this, jute fibres become dry and hydrophobic. So, the electrical conductivity decreases with the increase of discharge power and treatment time. Dielectric loss-tangent, which was found in low frequency region, is dominated by interfacial or ionic polarization. Although the surface property alterations obtained with plasma treatment are very complex, the treatment is environmental friendly, it optimizes the surface properties of materials without altering their bulk characteristics. In addition, the consumption of chemicals is very low due to the physical process. The most important factor is that the substrate surface properties change significantly after a short plasma treatment. In addition, jute has low specific weight, low cost, renewable, biodegradable and the production requires little energy, CO_2 is used while O_2 is given back to the environment. These alone have significant implications for the jute industry.

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