

**Research Article**

Effect of Dielectric Barrier Discharge Parameters on Certain Properties of Natural Polymeric Material (Cotton) Fiber

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Abstract

Dielectric Barrier Discharge (DBD) plasma reactor, operated at atmospheric pressure was used for modifying the properties of bleached plain woven cotton fabrics. Cotton fabric was treated at 3.0, 3.5 and 4.0 kV electrode current for 2.5, 5.0, and 7.5 min in the presence of air plasma, in order to establish the chemical effect on cotton fiber. Some properties such as wick-ability, absorbency, tensile strength, tear strength, dye-ability; FT-IR analysis and surface morphology analysis were carried-out as well. The outcomes revealed that, plasma treatment resulted in surface etching, generation of carboxylic and carbonyl group contents in cotton fiber. The increased carbonyl and carboxylic group content resulted in enhancement of fiber wettability and rate of fabric vertical wicking. The reactive dyes uptake of treated samples were increased almost linearly but decreased with increased treatment time and intensity of voltage across the electrodes. A slight increase in tensile and tear strength was found in air plasma treated fabrics. Yellowness of the fabric was increased after plasma treatment. The SEM micrographs confirmed the atmospheric plasma could modify the fiber surface outwardly.

Keywords: Cotton; Dielectric Barrier discharge; Wicking height; Colour yield; FT-IR; SEM

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1. Introduction

Cotton is one of the most important textile fiber in the world due to their excellent properties, such as breathability, moisture, heat conduction, softness, hypoallergenic and antistatic properties [1-5]. It is a unique biological object; because of it is gigantic biological cell of micro-molecule of microscopic linear dimensions [6]. However, it contains several unwanted impurities and removal of impurities is obvious to make raw fabric white and absorbent to prepare them for dyeing, printing or chemical finishing [7, 8]. In recent years, plasma related techniques have been increasingly used in the etching, deposition, or other modifications of textile materials to improve their surface properties [9, 10]. Because, plasma treatment modifies or removes the fibers hydrophobic outer layer, improving dye-fiber interaction and increasing the flux of dye molecule through the fiber surface into the fiber bulk [11]. During the treatment bombardment of active species causes surface atoms ejection when, transferred energy is greater than atom binding energy. A large number of cracks and grooves are generated on fiber surface by etching which, modifies surface properties physically. Furthermore, during the treatment or immediately after plasma treatment on exposure to the atmosphere free radicals insert new hydrophilic carboxylic and hydroxyl derivatives (e.g. $-C=O$, $-C-O$, $-COOH$, $-OH$) on fiber surface [12, 13]. Polymer chains are partially decomposed during the plasma treatment and new functional are formed and these newly formed polar groups act as reactive centers for subsequent chemical modification or as an anchor for the other agents [14]. Dielectric barrier discharge plasma reactors, operate at elevated pressure and glow discharges at low pressure in a vacuum system, are used to modify the fibers and textiles as some existing chemical processes. They can modify the surface nature of hydrophobic polymers without remarkable deterioration to their bulk properties by etching and polarization [1]. The different types of gas or mixture of gases that can be used for plasma treatment of textile such as argon, carbon dioxide, sulphur dioxide, water and tetra-flouro-methane, and oxygen. Oxygen and oxygen containing plasma are most frequently and are very effective in increasing the surface energy of textiles [15]. Nitrogen and nitrogen containing plasmas are used to produce nitrogen functionality such as amino groups of polymer of polymer surface [16]. Fluorine and fluorine containing plasma are used to decrease the surface energy and to increase the hydrophobicity of polymer surface. Each gas produces a unique modified surface [17]. In oxygen plasma two processes occurs simultaneously; etching, of polymer and textile surface through the reaction of atomic oxygen with the carbon atoms, giving volatile reaction products, and the formation of oxygen functional groups at the textile and polymer surface through the reaction between active species from the plasma and the surface atoms. The balance of these two processes depends on the operation parameters such as the nature of textile substrate, the temperature of the substrate, electrode materials, pressure, power level, and gas flow rate [18].

In the present work an attempt has been made to enhance the function properties of cotton fiber with the use of least expensive air as a plasma gas, in a DBD reactor. Optimum treatment parameters such as treatment time and voltage across the electrodes on properties of cotton fiber such as whiteness, yellowness, tensile and tear strength, wicking height and colour yield of reactive dyes were well reported in this paper.

2. Experimental detail

2.1 Materials

Commercially available plain woven, scoured, bleached, 100% cotton fabric (warp count - 40 Ne and weft count - 40 Ne) with average weight (130 g/m^2), supplied by Piyush Syndicate (Mumbai, India) was used in this study. Before the plasma treatment, to minimize the chance of contamination, samples were washed with 0.5 % non-ionic detergent (Enkamole LFS supplied by Yogeshwar Chemical Ltd.) solution at $80 \text{ }^\circ\text{C}$ for 30 min, then rinsed with plain water for another 15 min, and dried at ambient temperature before plasma treatment.

2.2 Plasma treatment

Cotton fabric was treated in a DBD plasma reactor designed by, Facilitation Center for Industrial Plasma Technologies (India) was used. The main components of the reactor includes, an Al_2O_3 ceramic electrode with active area $40 \times 50 \text{ cm}$ and a high power supply unit (500 W). The gap between the electrodes could be adjusted in the range of 0.5 – 2.5 mm. However, the distance between the electrodes was 2 mm during the treatment and air was used as reactive gas and system was worked at atmospheric pressure. Cotton fabric samples with an average thickness of 0.3 mm passed between the electrodes continuously, and both sides of the fabric were exposed to the plasma treatment. The cotton fabric was treated with 3.0, 3.5 and 4.0 kV electrode current, for 2.5, 5.0 and 7.5 min. After LTP treatment, the fabrics were conditioned in 65% RH (Relative humidity) at $27 \pm 2 \text{ }^\circ\text{C}$ before being used.

2.3 Dyeing

Dyeing of untreated and DBD treated cotton samples was carried out by classical exhaustion method in a laboratory Rota dyer equipment. Samples were dyed with Reactive Red 195 and Reactive blue 171, supplied by the Coloutex Industries Ltd, (India). The dye solutions were prepared by dissolving the 1gm of dye powder in 100 ml hot distilled water and then samples were dyed in 1.5 % o.w.f. (on weight of fabric) shade with liquor ratio of 1:30. The dyeing was started at $40 \text{ }^\circ\text{C}$ and temperature was raised to $80 \text{ }^\circ\text{C}$ at the rate of $3 \text{ }^\circ\text{C}$ per min. The samples were allowed to remain in that condition for 45 min, and then rinsed with distilled water and air dried.

3. Testing and Analysis

3.1 Absorbency

To explore the effect of DBD treatment at different electrode voltage for different duration on absorbency behavior of cotton fabric, the wettability of cotton fabrics was evaluated according to the AATCC test methods: 79:2007.

3.2 Wicking Height

The wicking behaviour of plasma treated and untreated cotton fabrics were measured according to the German standard method DIN 53924.

3.3 Whiteness and Yellowness

The whiteness and yellowness indexes of untreated and DBD treated cotton fabric at different parameters were measured by Macbeth colorage[®] 3000 spectrophotometer with 10° observer under the D₆₅ light source and calculated according to the CIE, ASTM D1925 and TAPPI 452/ISO 2470 equations.

3.4 Tensile Strength

Tensile properties of untreated and plasma treated cotton fabric was determined according to the ASTM D5035 (1995), with Instron tester after conditioning the fabric samples at 65 ± 2 % RH and 27 ± 2 °C temperature for 24 hours.

3.5 Tear Strength

Tear strength of untreated and DBD treated cotton fabric was measured with Elmatear tear strength tester (James and Heal Co. UK) according to the IS 6489 -1971 test method.

3.6 Colour Yield (K/S)

The colour yield of untreated and DBD treated cotton samples was determined by Macbeth colorage[®] 3000 spectrophotometer with colour lab software. The fabric was folded two times before measurement for ensuring opacity and measured twice, i.e. measured on both the warp and weft directions to obtain average results. The colour yield expressed as a K/S value ranging from the wavelength of 400 to 700 nm with 20 nm interval within the visible spectrum. The K/S values were summed up according to equation (1).

$$K/S = \frac{(1-R)^2}{2R} \quad (1)$$

Where, K= Absorption coefficient depending upon the concentration of colorants, S= Scattering coefficient caused by the dyed substrate, and R= Reflectance of coloured sample.

3.7 FT - IR Analysis:

In order to determine the surface functional group changes caused by air plasma treatment, Fourier transform infrared spectroscopy (FTIR) assessment were carried out using Nicolet Avatar 8400 FTIR model, with 4

cm⁻¹ resolution. An average of 15 scans was recorded in the ATR mode.

3.8 Surface Morphology

The change in surface morphology of plasma treated cotton fabric was investigated using scanning electron microscope (SEM 5400, Japan). Samples were observed 5000 to 10,000 times of magnifications.

4. Results and Discussion

4.1 Absorbency

Absorbency measurement of air plasma treated fabric in DBD reactor was conducted immediately after treatment and compared with untreated sample and, results represented in Table 1. From the table it is clear that, the wettability of air plasma treated cotton was improved. The absorbency time of treated cotton decreased from 4 sec to less than 2 sec. However, the treatment voltage and exposure duration showed the significant differences in absorbency behavior i.e. when the treatment voltage was 3.0 kV the improvement in absorbency was observed at 5.0 to 7.5 min exposure duration whereas, identical effect was found with 3.5 and 4.0 kV for 2.5 min exposure duration. The decreased absorbency duration attributed to the surface ablation and formation of new polar groups. The enhanced surface roughness resulted in decreased barrier between water drop and surface caused by the vapor formed between the fiber and water drop. Water vapor traps at the bottom of the grooves of enough depth and water comes in direct contact with raised areas of groove and penetrates inside the fiber interstices immediately [19].

Table 1 Absorbency behavior of cotton before and after DBD treatment.

Voltage across electrodes (kV)	Plasma treatment time (min)	Absorbency time (sec)
Untreated Cotton	----	4
3.0	2.5	3
	5.0	2
	7.5	2
3.5	2.5	2
	5.0	3
	7.5	3
4.0	2.5	2
	5.0	3
	7.5	3

DBD generates an active gas plasma consisting of free electrons, positive ions, and other chemical species. These active species can be used to modify the material surface physically and chemically. The enhanced

wettability might be due to the etching effect of DBD emission on the cuticles of cotton fibers [20]. This results in partial degradation and removal of hydrophobic non-cellulosic. In addition the surface of cotton fiber would be oxidised by oxygen radicals in the plasmas and produces more oxygen-containing groups, this might also contribute to the improvement of wettability of treated cotton samples [1]. However, excessive treatment did not remarkably improve the wettability.

4.2 Wicking Height

The increased wicking of untreated and DBD treated cotton is represented in figure 1. Compared to the untreated fabric, the rate of wicking was very rapid initially and the same speed was maintained up to a height of 12.8 cm at 3.0 kV for 2.5 min in warp direction, and in weft direction was 12.3 cm at 4.0 kV for 2.5 min on the cotton stripe. However, no significant impact of electrode voltage and treatment duration on wicking height of cotton fabric was observed in both warp and weft directions.

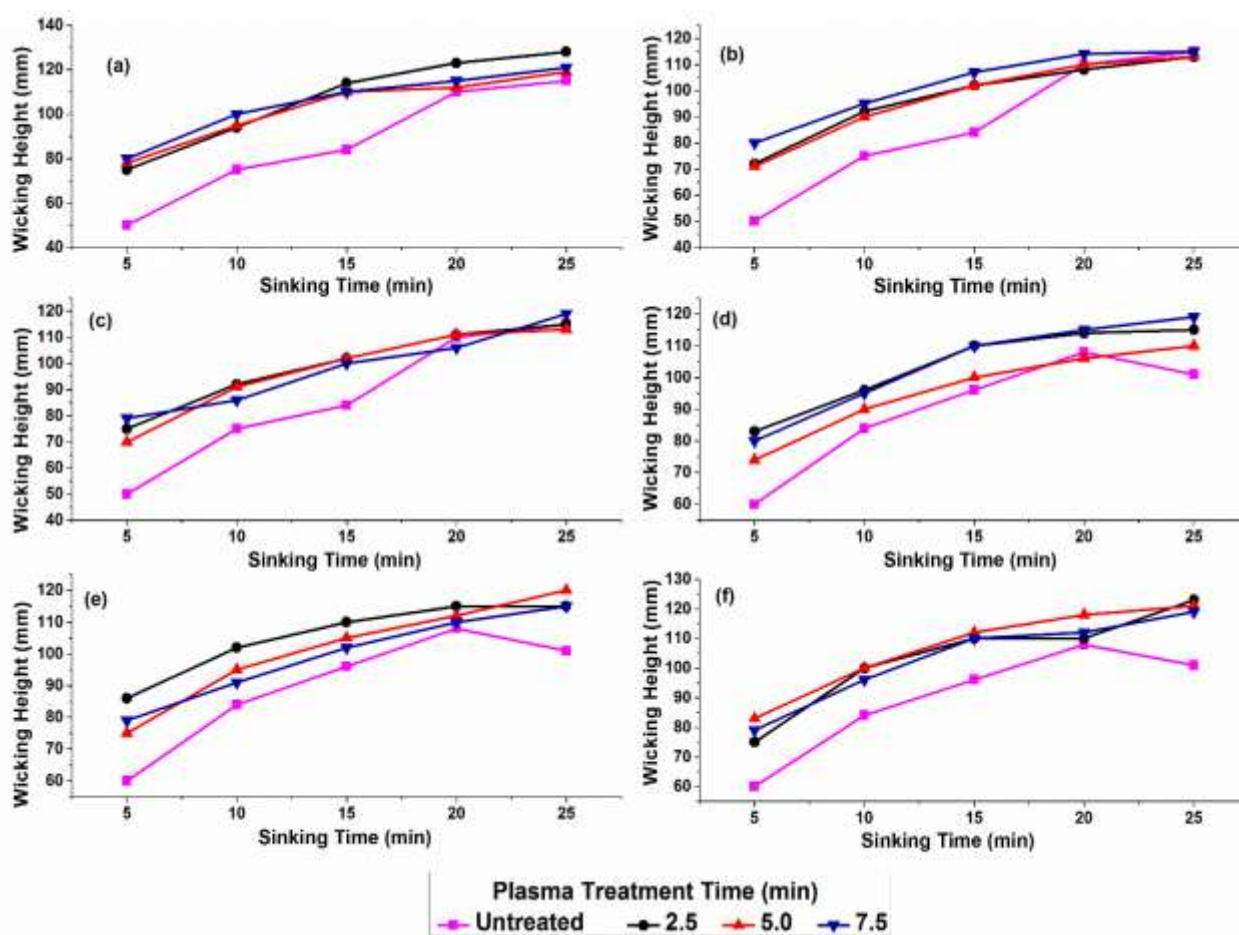


Figure 1 Wicking height in warp direction of untreated and air plasma treated cotton fabric with 3.0 kV (a), 3.5 kV (b), and 4.0 kV (c), and wicking height in weft direction of untreated and air plasma treated cotton fabric with 3.0 kV (d), 3.5 kV (e), and 4.0 kV (f), in warp direction (a) and weft direction (b) at different parameters.

This observation suggests that with increasing voltage across the electrodes, the cellulosic fibers may become slightly more hydrophilic since wettability is a prerequisite for wicking; a liquid that does not wet fibers cannot wick into a fabric. Moreover, due to plasma etching effect the effective pore size present cotton fibers may increase and adversely reduce the capillary pressure thus increasing the wicking ability [21, 22]. This can be explained by the facts that increasing the discharge power result in an increase of the amount of reactive plasma species. The presence of more plasma species can leads to increased fiber wettability and/or an increased wicking ability due to a more intense bombardment on the fiber surface [23]. Both this aspects will be further explored by performing SEM and FTIR measurements in order to determine the chemical and physical properties of the plasma treated cellulosic fibers.

4.3 Whiteness and Yellowness

The dependence of whiteness and yellowness on the electrode voltage and treatment time with air plasma is shown in figure 2. It is clear that, dielectric barrier discharge emission caused a slight yellowing compared with the untreated cotton fabric. When the exposure time of DBD treatment was prolonged, the degree of yellowness increased and whiteness decreased accordingly. This might result from pyrolysis and oxidation of the surface components [20]. The untreated fabric sample had the lowest yellowness, while the fabric sample treated by LTP treatment for 7.5 min at 4.0 kV electrode voltage got the highest yellowness index values. However, the overall changes in yellowness and whiteness were not so obvious when the parameters of DBD treatment were changed from 3.0 to 4.0 kV electrode voltage and time from 2.5 to 7.5 min.

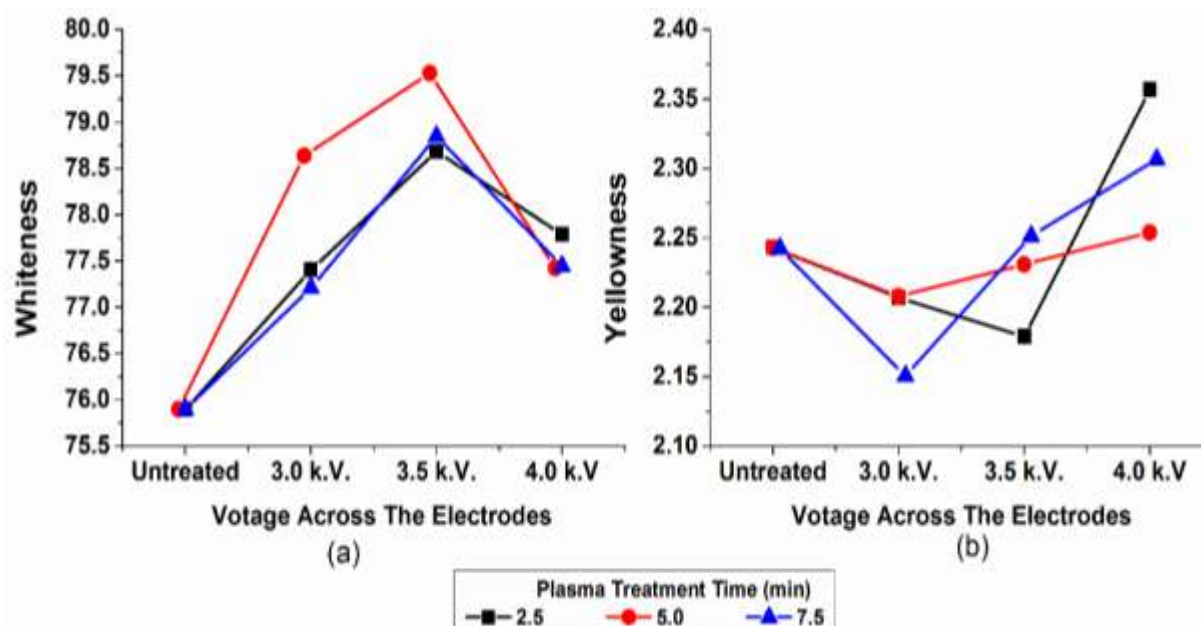


Figure 2 Effect of air plasma treatment on whiteness (a), and yellowness property (b) of cotton treated in dielectric barrier discharge plasma reactor.

DBD treatment it seemed that the oxygen (present in air) damage on the fabric surface after a certain period of time. The longer the LTP exposure time, the greater damage of fiber surface. Since, whiteness of textile

substrate depends upon the surface smoothness i.e. more smooth surface leads to the reflection of incident light whereas rougher surface leads to the diffuse reflection of incident light and cause yellowness of the fibers. Treatment at higher electrode current for longer duration leads to the serious etching of the fiber surface and hence overall yellowness was increased when the electrode current was 4.0 kV for 5.0 to 7.5 min. As a result of the degree of yellowness and whiteness was dependent on the plasma treatment conditions such as treatment time, plasma gas and voltage across the electrodes [24]

4.4 Tensile Strength

Tensile strength is one of the important properties in assessing the quality of treated fabric. Hence, the tensile strength of plasma treated fabric was compared with that of the untreated fabric. It is observed that on several occasions the tensile strength of plasma treated sample is higher than that of the control sample. Figure 3 represents the effect of parameters on tensile strength. It is evident from the figure, with increase in electrode voltage from 3.0 to 4.0 kV for 2.5 to 5.0 min exposure time, tensile strength was increased up to 4 % but longer exposure time for 7.5 min at higher voltage from 3.5 to 4.0 kV is found to be decreased up to 2.2 %. From SEM micrographs it is evidenced that, air plasma treatment leads to the ablation of fiber surface and micro cracks was formed. The newly formed micro cracks might be the root cause for the increased inter fiber cohesion and thus prevents the sliding action on application of force.

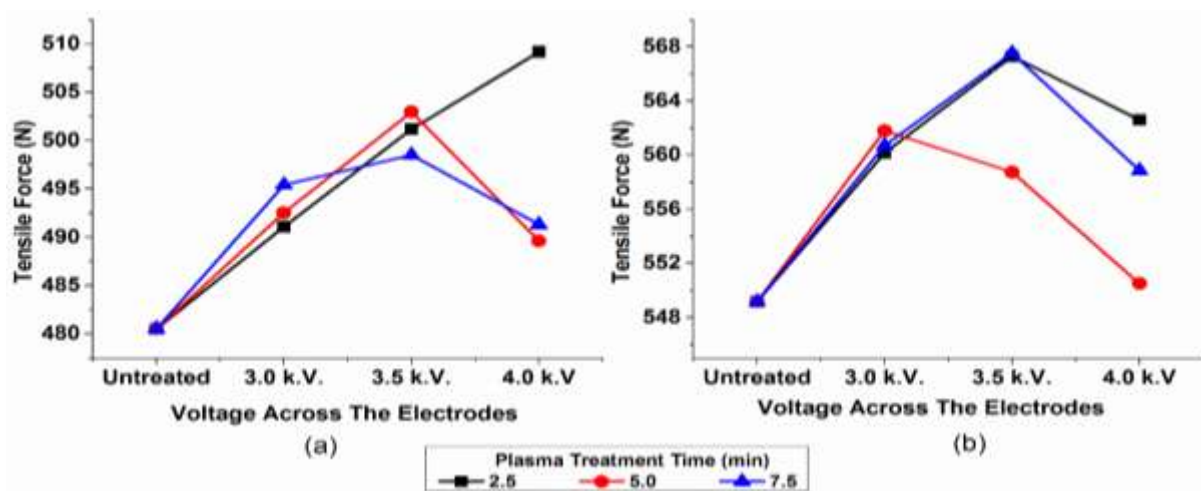


Figure 3 Tensile strength of cotton before and after air plasma treatment in warp direction (a), and weft direction (b) in dielectric barrier discharge plasma reactor.

Apart from change in surface topography air plasma leads to the formation of polar groups by the action of plasma in the cuticle of fibers; these newly formed polar groups are responsible for an increase in inter-fiber cohesion and leads to the better mechanical properties. However, at higher intensity of electrode current leads to the damage of cuticle and cellulose degradation due to serious bombardment of active plasma species and leads to the reduction in tensile strength [25, 26].

4.5 Tear Strength

The tear strength of untreated and DBD treated cotton fabric under different conditions are represented in figure 4. The outcome revealed that the tear strength of cotton fabric was increased 0.25 to 0.45 % both warp and weft directions, when compared to untreated samples. The maximum strength was observed when duration of treatment was 2.5 to 5.0 min for 3.0, 3.5 and 4.0 kV respectively. This may be attributed to the increased inter yarn and inter-fiber friction because of the surface modification by the plasma treatment, restrict the sliding action of yarn during tearing and thereby increases the fabric's tearing strength [27].

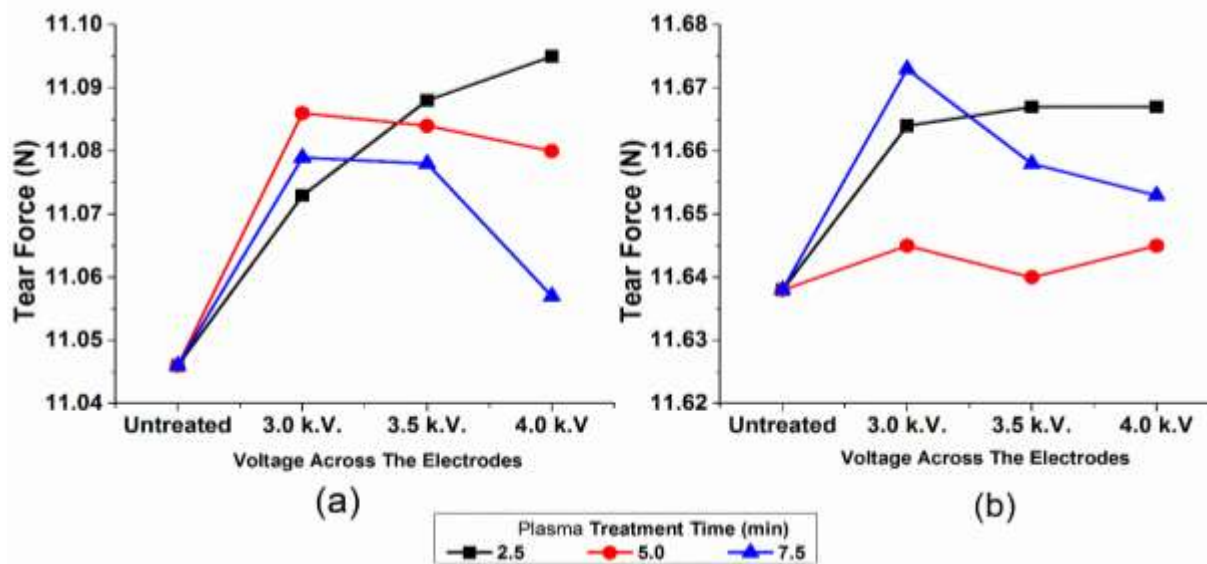


Figure 4 Tear strength of untreated and air plasma treated cotton fabric in warp direction (a), and weft direction (b) in dielectric barrier discharge plasma reactor.

For many instance it was observed that, treatment for longer duration resulted in a considerable decrease in tear strength due to the serious oxidation fiber surface. A slight reduction in tear strength was observed when cotton samples were treated at 3.5 and 4.0 kV for 7.5 min. treatment at higher voltage for longer duration cause serious damage to the surface as well as loss of some polar groups of cotton fiber therefore, reduction in tear strength [26]

4.6 Colour Yield (K/S)

Effect of exposure time to the air plasma at different discharge currents on the colour yield of reactive dyes (Reactive Red 195 and Reactive Blue 171) on untreated and air plasma treated cotton fabric dyed in 1.5 % shade illustrated in figure 5. It is observed that, the colour yield of cotton fabric was improved after DBD treatment. The enhanced colour yield increase by plasma treatment may be explained by, the higher hydrophilicity of the fiber surface, which produces a faster adsorption and diffusion of the dyestuff from the dye bath to the core of the fiber structure and increased number of hydrophilic groups which may allow a higher number of molecules to bind the dye molecules in the fiber. [24, 28]. The treatment time and electrode voltage have a greater impact on the colour yield of reactive dyes on DBD

treated cotton fabric. It was increased with increasing treatment voltage but, decreased when treated for longer duration up-to 7.5 min at high voltage (i.e. 3.5 kV and 4.0 kV) The decreased colour yield might be attributed to the loss of polar groups for the dye molecules were might be lost and hence the dye-ability of treated cotton fabric was decreased. The maximum colour yield was obtained when the discharge current was in the range of 3.0 – 3.5 kV electrode. Hence, these voltage was considered as optimum discharge current in accordance to the dye ability of cotton fibre.

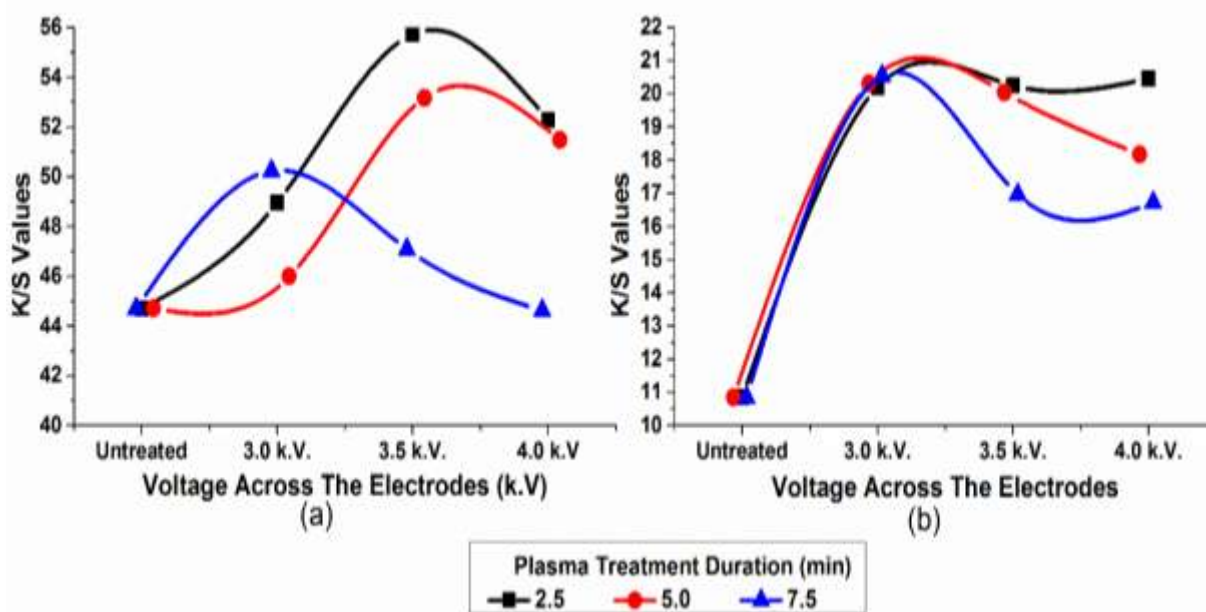
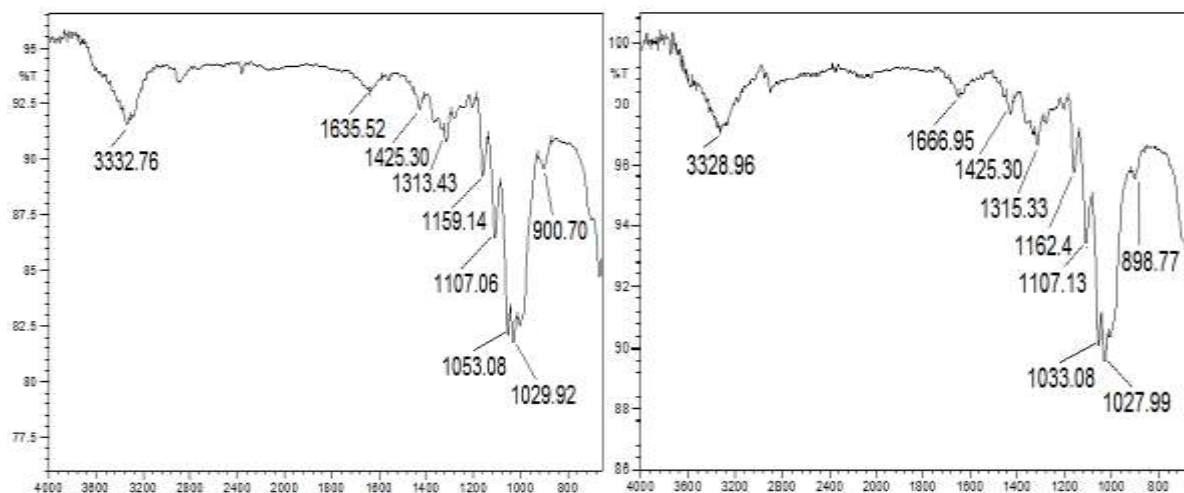


Figure 5 Colour yield of untreated and air plasma treated cotton fabrics with Reactive Blue171 (a), and Reactive Red 195.

4.7 FT - IR Analysis

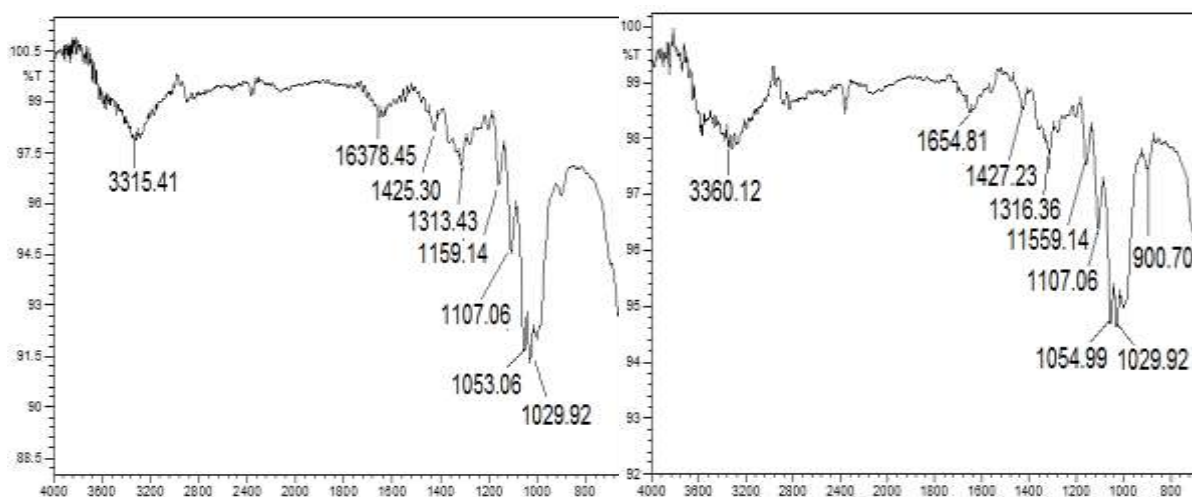
FTIR analyses were performed on untreated and DBD treated cotton samples with different parameters (electrode voltage and treatment time), and their corresponding spectra are shown in figure 6. The spectrum obtained from untreated and plasma treated cotton species are very similar, but the spectrum for DBD treated cotton showed some difference in peaks of 1635 and 3332 cm^{-1} corresponding to the C=O vibrations groups in the carbonyl structure and O-H stretching vibrations, respectively [29]. Bombardment of plasma on cotton fabric enables the initiations of chemical reaction and leads to alteration of chemical composition of the cotton fabric i.e. increase of hydrophilic groups such as -OH, -C=O, and -COO⁻ that can improve hydrophilicity of the fabric [30, 31]. Besides, transmittance intensity of plasma treated fabric was lower than of the untreated fabric. This mainly attributed to the number of

polar groups formed and bonded on the fabric polymer in which transmittance is inversely proportional to concentration of the corresponding groups [32].



(a)

(b)



(c)

(d)



(e)



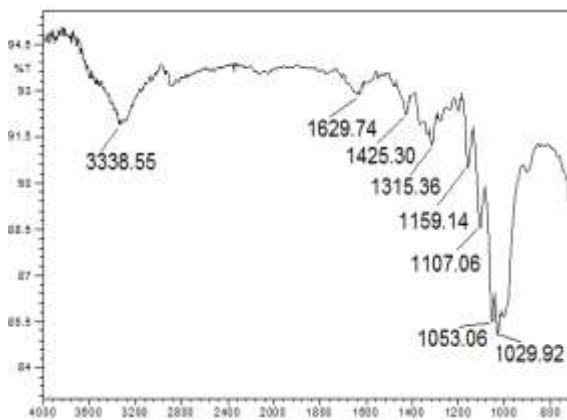
(f)



(f)



(g)



(h)



(i)

Figure 6 FT - IR spectrum of untreated cotton (a), air plasma treated cotton at 3.0 kV electrode voltage for 2.5 min (b), 3.0 kV for 5.0 min (c), 3.0 kV for 7.5 min (d), 3.5 kV for 2.5 min (e), 3.5 kV for 5.0 min (f), 3.5 kV for 7.5 min (g), 4.0 kV for 2.5 min (h), 4.0 kV for 5.0 min (i), and 4.0 kV for 7.5 min (j).

4.8 Surface Morphology (SEM)

As 3.0 kV for 7.5 min of DBD treatment time showed improvement in overall properties of cotton fabric (e.g. tear and tensile strength, dye ability, wick ability, and absorbency) in this study. Thus the SEM image of air plasma treated cotton fabric in dielectric barrier discharge reactor with 3.0 kV for 7.5 min treatment time was selected for study and their corresponding micrographs were shown in Figure 7. When compared, it was observed that the untreated cotton fiber surface had a smooth surface while the air plasma treated cotton fiber surface became wrinkled and roughened.

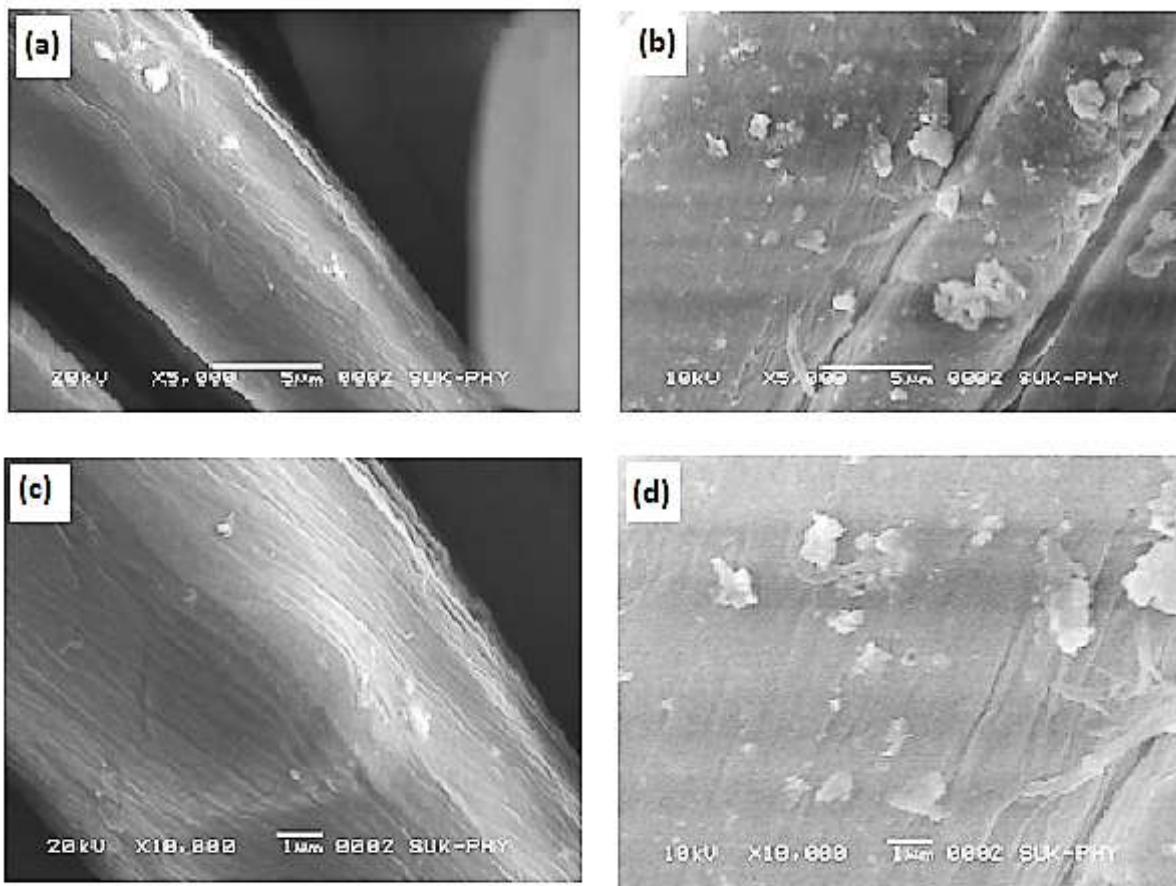


Figure 7 SEM micrograph of untreated cotton at X 5000 (a), and X10, 000 times (c), air plasma treated cotton fibre with 3.0 kV for 7.5 min at X 5000 (b), and X 10,000 times (d).

The change in the cotton fiber surface appearance might be due to the pyrolysis and localized ablation of the surface layer causing a surface damage due to active species in air plasma [33, 34]. During air plasma treatment ablation, the fiber surface was subjected to certain degree of etching. Presence of micro-pores on the fiber surface indicated this predominant effect of the interaction of air plasma (chemical etching) with the fiber surface. The differential etching of crystalline and amorphous regions might be the origin of the roughness. This process, lead to almost complete breakdown of relatively small number of molecules on the fiber surface into a very low molecular component, which would eventually vaporize into the low pressure

system along the fibers axis [24].

Conclusions

The effect of air plasma treatment on physiochemical properties of cotton fabric have been studied with respect to the treatment time and plasma power. As expected air plasma treatment generates greater change in the fiber and fabric properties under the experimental condition used. The plasma treatment leads to surface erosion of the cotton fibers, which generates an increase in tensile and tearing strength in certain degree and decreased slightly when, the voltage across the electrodes increased, the highest physical strength was obtained at 2.5 to 5.0 min DBD treatment when, treated with different voltages across the electrodes. The FT-IR spectrum clearly showed that, oxygen containing groups such as C–O, C=O, O–C–O and O–C=O was enhanced in DBD treated cotton fiber, which, might be the root cause for improvement in wetting and wicking properties. It was found that, the air plasma treated cotton fabric surface turned yellow due to pyrolysis of fiber surface when, compared with the untreated cotton fabric. The colour intensity of the reactive dyes on air plasma treated cotton fabric was increased with 3.0 – 3.5 kV electrode current. This is well explained by the fictionalization of the surface with oxygenated moieties, without any significant alteration in the surface topography of the fibers. SEM outcomes suggested that, atmospheric plasma treatment made the surface structure into a rougher state because of the etching effect. In other words, air plasma generated in DBD reactor have the potential of influencing not only the chemical properties but also the physical properties of the cotton fiber.

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