

## **Altering the Wettability and Dyeability Properties of Cotton Fabric using Low Energy Hydrogen Ion Showers**

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### **Abstract**

*Low energy hydrogen ion shower discharge was produced using a gas discharge ion source to alter the wettability and dyeability properties of cotton fabric. Ion showers of 400 and 600 eV were used and samples were immersed in the discharge for 15, 30, 45 and 60 minutes. Wettability was measured via the capillary rise method by calculating the velocity of the rising liquid front. Results were then crosschecked using the Lucas-Washburn equation by calculating the advancing contact angles. Color properties such as reflectance, tinctorial strength (K/S) and CIEL\*a\*b\* values were measured. Treatment led to various physicochemical alterations depending on beam energy and processing times. High energy shower led to fiber abrasion which gave rise to decreased water-sorption property. Low energy shower and longer treatment duration enhanced hydroxyl groups (O-H) promoting surface activation. Both treatment levels strengthened carbon triple bonds (C≡C) resulting in better dyeability.*

**Keywords:** *Low energy hydrogen ion shower; Wettability; Dyeability; Lucas-Washburn equation; FTIR; SEM; CIEL\*a\*b\* system*

### **1. Introduction**

The ever growing need for a cleaner environment has forced the textile industry to seek out alternative processing technologies that are more “ecosystem- friendly”. Finding solutions to the environmental problems brought about by the textile industry is not a simple undertaking as it includes numerous production sectors which utilize a huge amount of hazardous, non-biodegradable compounds. High water and energy consumptions compounded by the generation of a sizable amount of effluents steep in toxic chemical compounds are only some of the complex problems brought about by the textile industry. The industry should proceed towards sustainable technologies, developing environmentally benign methods of processing and finishing fabrics. However, due to the costs related to research and production of new fibers, the focus now is on the modification of existing fibers to impart the desired aesthetic and unique functional properties. For example, one of the methods to modify textile fibers so as to increase the dyeing rate was performed by immersing hemp fabric in low pressure capacitively-coupled RF-induced air plasma [1]. As a result, enhanced dyeability of textiles downsizes the consumption of chemicals crucial in minimizing toxic wastes. Using plasmas to tailor-fit textile functionalities is a novel approach that reduces air, water and land pollution in comparison to conventional methods of wet chemistry [2]. Plasmas modify the surface layer of the textile substrate causing changes to a limited depth only, thereby leaving the bulk characteristics unaffected [3, 4]. Depending on the choice of plasma conditions, the treatment can lead to hydrophilization or hydrophobization of textiles, improvement of dyeability; printability; adhesion properties;

antibacterial and insect repellent characteristics; thermal and flame resistance and the improvement of thermo-physiological comfort.

However, one of the drawbacks in using plasma technology is the high initial capital investment on vacuum equipment, scaled-up reactors, advanced instrumentation for precise process control, *etc.* Hence atmospheric plasma systems for tailoring textile properties are now the trend to sidestep initial startup costs [5, 6]. On the other hand, low pressure plasma treatment offers much better stability, control and reproducibility compared to atmospheric treatments. In the long run, because low pressure reactors offer reproducible, highly flexible product functional design, this technology saves production cost for the entire process thus increases profitability. The expenditures in equipment can be recompensed rapidly in the form of environment-related savings resulting in a “greener”, eco-friendly more viable technique coupled with higher sale profits from better value-added products.

In this study, unlike conventional-plasma based surface modification where the textile material is placed in a plasma environment (*i.e.*, the target material is exposed to both electrons and ions), the current work focuses on the use of ions solely as the surface modifier. Low energy hydrogen ion showers (LEHIS) with energy range  $\leq 600\text{eV}$  were targeted onto cotton cloth to investigate changes in wettability and dyeability. The effects of  $\text{H}_n^+$  ion shower irradiation were analyzed vis-à-vis changes in fiber surface morphology and chemical composition.

## 2. Materials and Methods

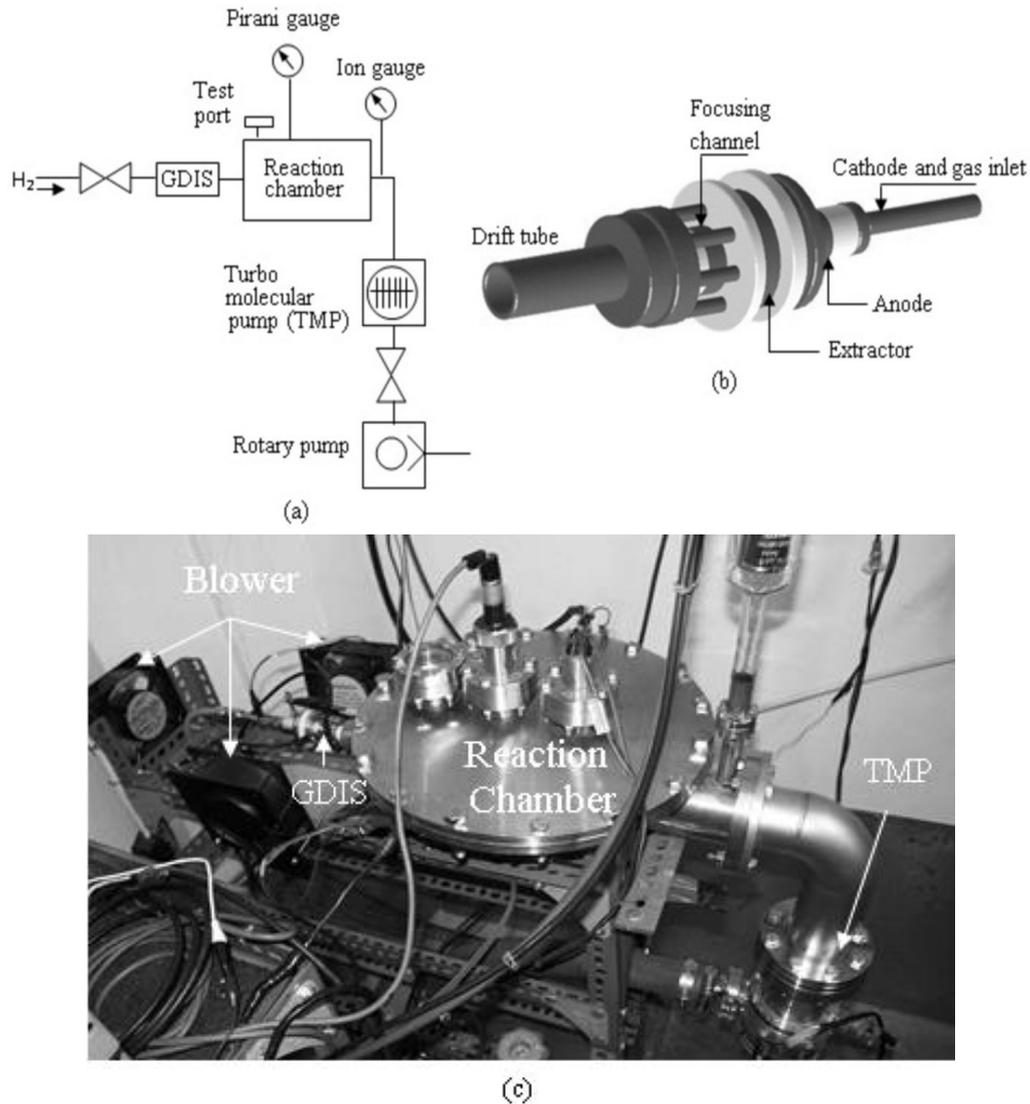
LEHIS treatment of the cellulose samples took place in a Gas Discharge Ion Source (GDIS) facility. Figure 1(a) shows the schematic of the overall facility and 1(b) the 3-D figure of the GDIS. The device has been reported in several papers [7-14]. The GDIS has a negative focal strength, which is the distinguishing characteristic of an ion source generating divergent ion showers. Generated beams are diffused, homogenous and round with energy range below 1 keV most appropriate for surface transformation of organic polymers. Prior to plasma ignition, the facility was evacuated up to a background pressure of  $1 \times 10^{-5}$  Torr. Ion shower treatment was set at 3 mTorr  $\text{H}_2$  gas feed, 400-600V discharge.

Samples to be treated consisted of cellulose in the form of pure cotton cloth, cut into uniform strips and wrapped around stainless steel (SS) sheets which served as substrate holders. These SS sheets measuring  $3 \times 3 \times 0.1$  cm<sup>3</sup> were pre-sanitized with methanol and air-dried. Mounted on a substrate holder, the cloth samples were oriented perpendicular to the incoming  $\text{H}_n^+$  ion shower. The samples were irradiated using 400 and 600 eV showers for 15, 30, 45 and 60 minutes.

To provide a qualitative description of the effect of the ion shower treatment on the surface morphology of the cloth samples, scanning electron microscopy was employed (Leica S440 Electron Microscope with Wavelength Dispersive System). Chemical changes in the samples were examined using Fourier Transform Infrared spectroscopy (Nexus 670 FTIR ESP with Nicolet Continuum Microscope).

Wettability is actually very easily described by contact angle measurements. However, in the case of textiles, and in particular cellulose fibers which are naturally hydrophilic, the porosity of the fabrics elicits a strong suction effect on the water droplet which ultimately prevents the formation of the liquid's drop-shape [15]. The swiftness of the droplet's penetration into the fabric made it impossible to measure static contact angles [16]. Hence, the study resorted to fabric imbibition wherein the process involved bringing the fibrous system in contact with the wetting liquid and the height of the liquid front is measured as a function of time. This is known as the capillary rise measurement. The test fabrics (both treated and pristine) were vertically dipped to a depth of 5 mm into deionized water contained in a sealed transparent glass container to prevent evaporation.

The whole process of water uptake was captured through video. Still snapshots were then extracted from the video for every second of the process and the height reached by the water per second was subsequently recorded. The height of water-uptake was then plotted against time. The slope of the linear trend line indicated the rate at which water was being absorbed by the cloth sample. A steeper slope would mean a higher water-sorption rate.



**Figure 1. (a) Schematic Diagram of the Overall Processing Facility. (b) 3-D Figure of the Gas Discharge Ion Source. (c) Panoramic View of the Whole Facility**

Fortunately, the kinetics of water uptake allows us to calculate for the contact angles of porous materials. According to Poiseuille's law, the rate of liquid capillary rise in porous media is given by [16]:

$$\frac{dh}{dt} = \frac{R_D}{8\eta h} \left( \frac{2\gamma \cos\theta}{R_S} - \rho gh \right) \quad (1)$$

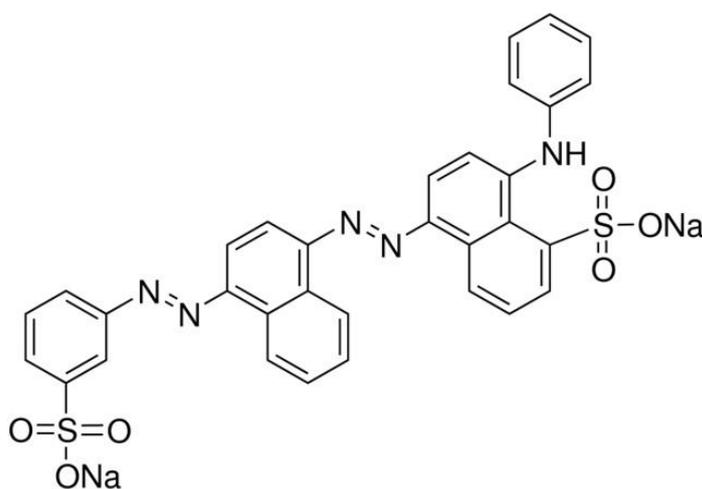
Where  $h$  is the height reached by the liquid at time  $t$ ,  $R_D$  and  $R_S$  are the mean hydrodynamic and static radii of the pores.  $R_S$  equals the geometrical radius of the pores as verified experimentally [17], while  $R_D$  depends on the tortuosity of these pores.  $\theta$  is

the contact angle between the liquid and capillary surface,  $\eta$  viscosity,  $\gamma$  surface tension,  $\rho$  density of the liquid, and  $g$  the acceleration due to gravity. The bracketed expression is the difference between capillary pressure ( $2\gamma\cos\theta/R_s$ ) and hydrostatic pressure ( $\rho gh$ ). Generally for cotton fabric, the average radius of the fibers is such that  $\rho gh \ll 2\gamma\cos\theta/R_s$ . This means that the hydrostatic pressure can be neglected so that upon integration, Eq. 1 reduces to the well-known Lucas-Washburn equation:

$$h^2 = \frac{r\gamma\cos\theta}{2\eta} \quad (2)$$

where  $r = (R_D)^2/R_s$ , which in fiber networks is the equivalent radius of the capillary porous structure. Contact angle calculated using Eq. 2 together with the velocity of the liquid front (*i.e.*,  $dh/dt$  or rate of water uptake) provided the parameters for a quantitative description of the wettability property. It has to be noted however that the Lucas-Washburn equation yields advancing contact angles ( $\theta_A$ ) and not static contact angles ( $\theta_S$ ). Static contact angles are measured when a sessile droplet is standing on the surface at equilibrium and the three phase boundary (where liquid, gas and solid intersect) is not moving. Advancing contact angle is the maximum value that static contact angle can reach, therefore  $\theta_A > \theta_S$ .

Dyeability in its simplest sense is how much a reactive dye adheres to the fabric. There are various methods to evaluate dye uptake such as dye exhaustion, fastness to washing and color measurement [18]. The percentage of dye exhaustion is determined by comparing the concentration of the dyeing solution before and after dyeing. Fastness to washing is a measure of the integrity of the dye against several laundering cycles. Color measurement involves noting how intense or deep the color of the dyeing solution turns out to be. This latter method was used and thus, dyeability in this study was restricted to color measurement only. A commercially available dye known as Acid Blue 113 was used to perform the dyeability test. It is a diazo-containing acid dye, dark blue to black in powdered form and soluble in water. Figure 2 shows the dye structure. 500 mg of the dye was dissolved in 100 mL of double distilled water. A sessile drop of this mixture was applied onto each sample and allowed to diffuse and dry up completely.



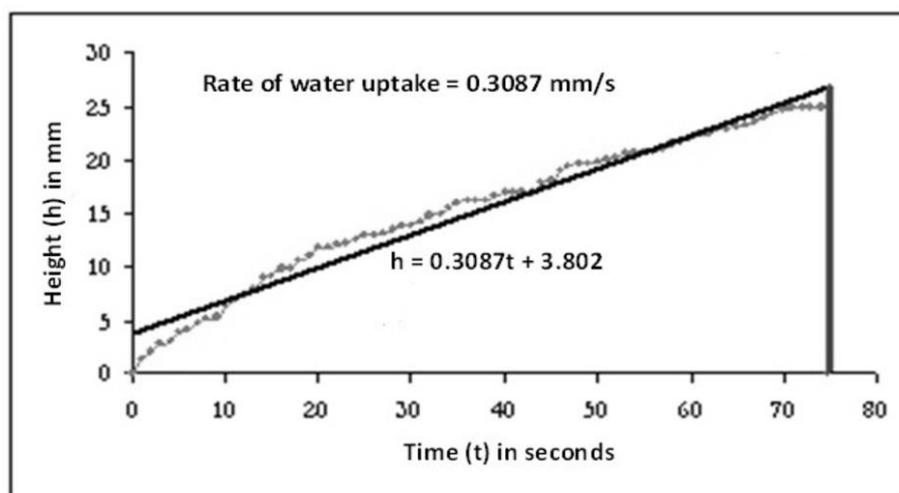
**Figure 2. (a) Dye Structure of Acid Blue 113. It has a Molecular Formula of  $C_{32}H_{21}N_5Na_2O_6S_2$**

Results of the dyeing parameters namely  $K/S$ ,  $CIEL^*a^*b^*$  values were measured using the Premier Color Scan Machine SpectraScan 5100+. Tinctorial or color strength ( $K/S$ ) is related to absorption property measured using reflectance via the Kubelka – Munk

equation:  $K/S = (1-R)^2/(2R)$ , where R is the decimal fraction of the reflectance of the dyed fabric, K is the absorption coefficient and S is the scattering coefficient of the colorant (or dye). Higher K/S values indicate higher strength of the colorant. The basis of all color measurement is the C I E system of color specification introduced by the Commission International de l' Ecclairage (CIE).  $L^*$  values indicatedepth of shade, it represents the difference between light (where  $L^* = 100$ ) and dark (where  $L^* = 0$ ). When comparing values,a sample with lower  $L^*$  means it has a darker shade compared to one with higher  $L^*$ .The values of  $a^*$  indicate greenness or redness of shade, ( $+a^*$ ) if the sample is red and ( $-a^*$ ) if the sample is green.  $b^*$  values indicate the blueness or yellowness of shade, ( $+b^*$ ) if the sample is yellow and ( $-b^*$ ) if the sample is blue.

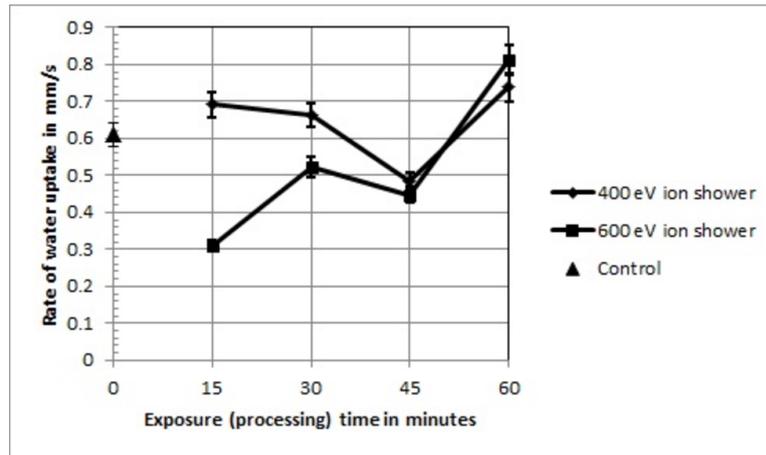
### 3. Results and Discussion

A typical plot of water uptake is shown in Figure3, which is a graph of the capillary rise data for the 600 eV treated sample processed at 15 minutes. The slope of the linear trend gives the rate of water uptake. In comparing rates of different samples, the lower the rate, the less water-absorbing the material.



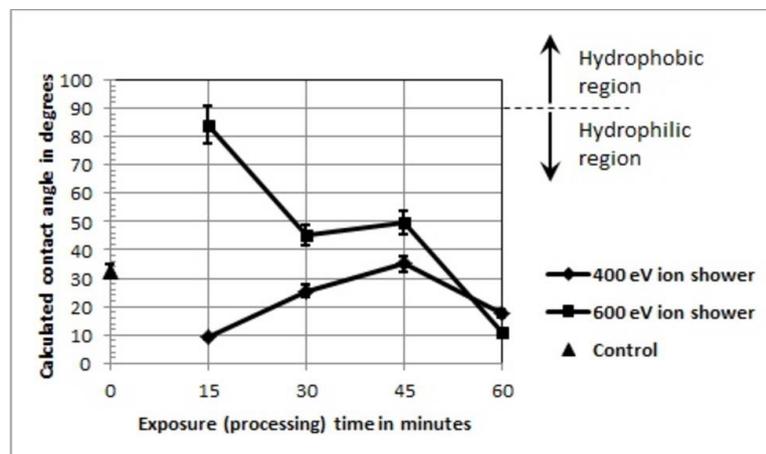
**Figure 3. Typical Plot of Water Uptake. Capillary Rise Graph for the Sample Treated with 600 eV Ion Shower Processed at 15 Minutes**

Figure 4 summarizes the wetting behavior of the test samples based on the rates of water uptake. Samples treated with higher beam energies (600eV) at short exposure times (namely 15 min, 30 min and 45 min) became less absorbent. Eq. 2 (Lucas-Washburn equation) was used to crosscheck the wetting behavior, this time based on contact angles. It is worth noting that the control yielded a contact angle of  $32^\circ$ , which is far from the expected value of about  $0^\circ$ , cotton being hydrophilic by nature. One reason for this as mentioned earlier is that Eq. 2 gives an advancing contact angle ( $\theta_A$ ) which is bigger than the equilibrium or static contact angle ( $\theta_S$ ). The second reason involves hysteresis effects. In capillary rise systems where liquid flow is relatively fast, the effect of dynamic advancing contact angle ( $\theta_{AD}$ ) becomes significant. The difference between  $\theta_{AD}$  and  $\theta_S$  increases even more as the liquid speeds up [19].



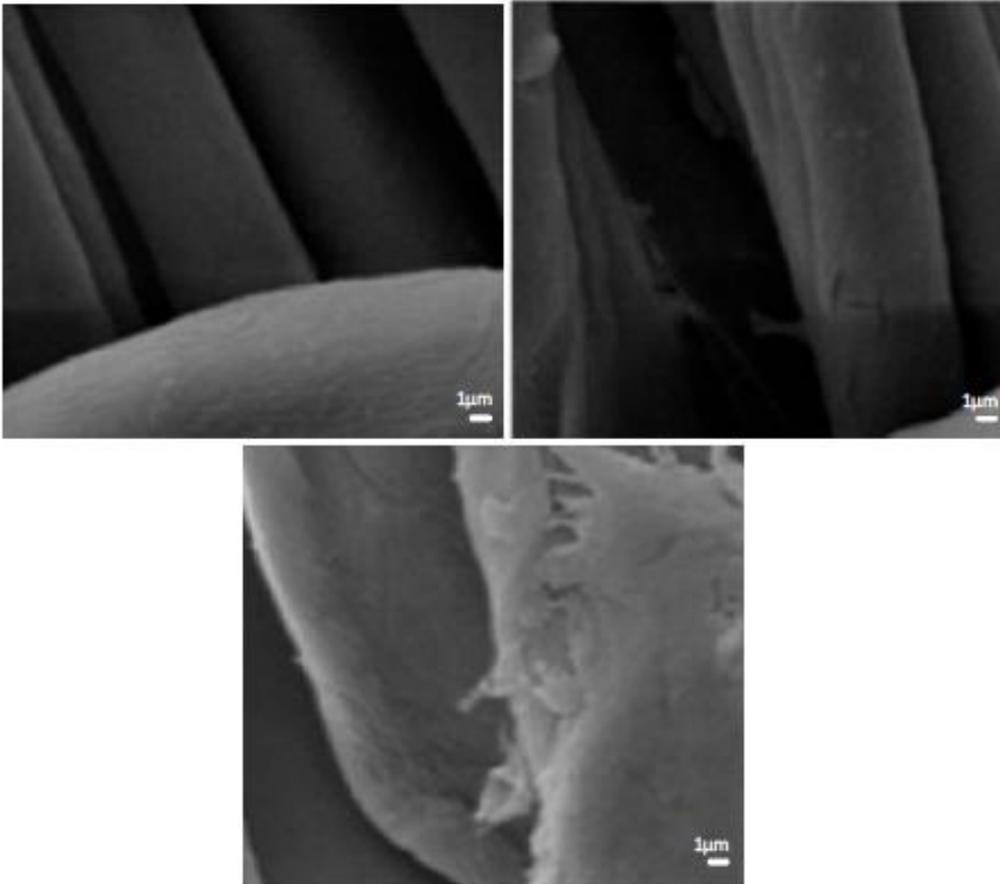
**Figure 4. Rate of Water Uptake for the Control and Treated Samples at Varied Exposure Times**

Figure 5 shows the contact angles (calculated using Eq. 2) vs. treatment time. The results support the wettability curve of Figure 3 (based on water uptake rates). The high energy group with shorter processing times had higher contact angles reaching a high of 84.1°. The low energy group showed a low of 9.0° to a high of 35.2° while that of the control is 32.4°. For the high energy group, the decreased affinity to water is a consequence of the abrasive action of the reactive hydrogen ion species ( $H_n^+$ ) on the cellulose fiber surface. This may be attributed to the so called “Lotus Effect” wherein the surface of the fiber achieves a rough texture similar to that of a lotus leaf enhancing its superficial water retention.

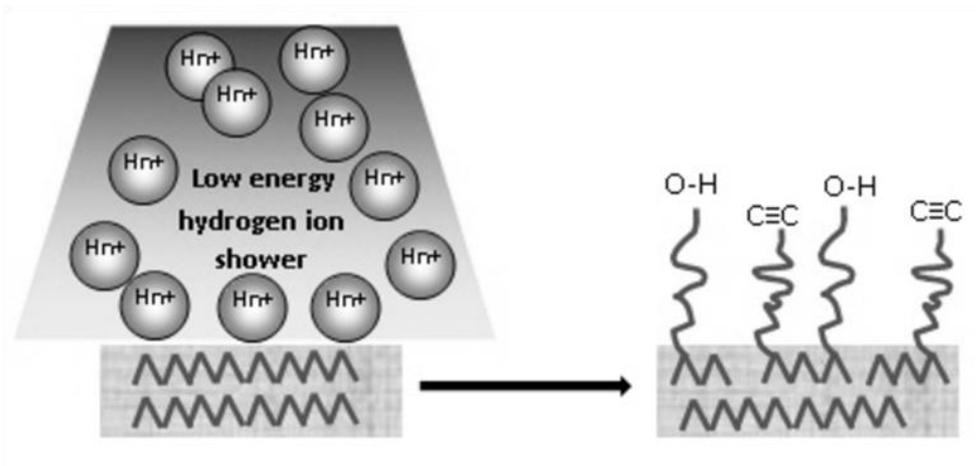


**Figure 5. Calculated Contact Angles for the Control and Treated Samples at Varied Exposure Times**

Figure 6 shows the progression of fiber abrasion at increasing beam energies. Noticeably, the degree of abrasion is pronounced at higher discharge. Figure 6(c) shows visible signs of roughening (abrasion) on the surface of the fibers. Interestingly though, at 60 min processing time, the processed sample under higher discharge became more moisture-absorbing than the control. Here, an interplay between fiber distortion and surface activation occurs with the latter ultimately overriding the former due to longer exposure time. The material undergoes repetitive scissions of polymeric chains by  $H_n^+$  ions resulting in a variety of functional groups left anchored on the surface as illustrated in Figure 7.



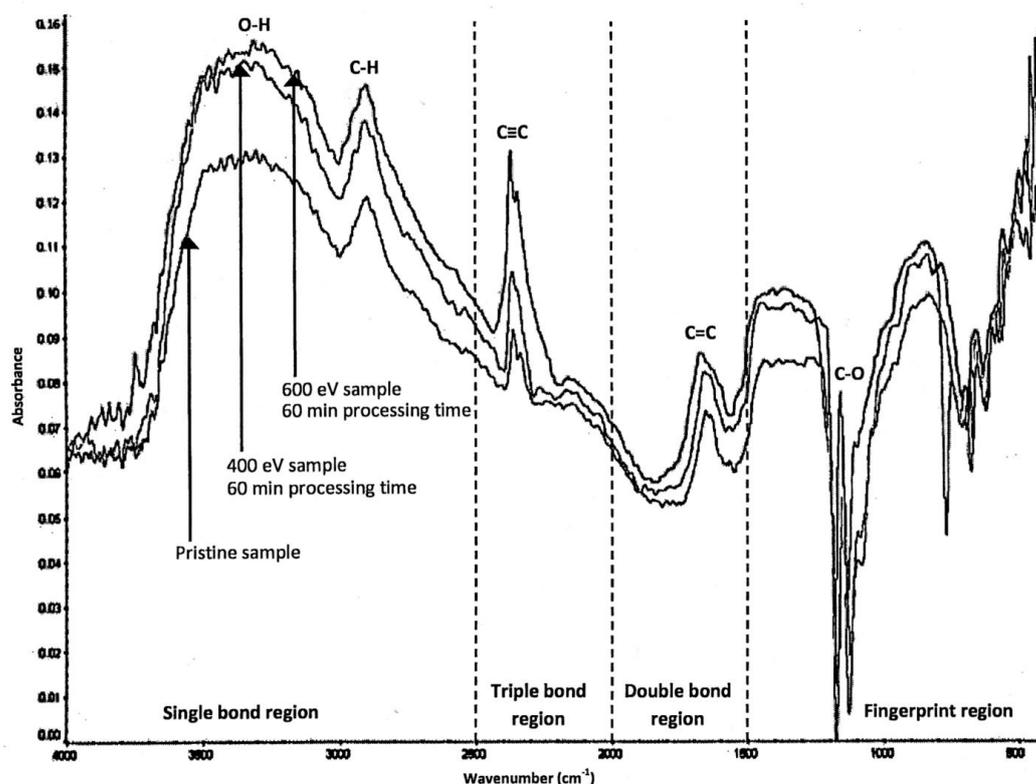
**Figure 6. SEM Images of the (a) Control, (b) Low Energy Treated Sample and (c) High Energy Treated Sample**



**Figure 7. An Illustration of a Textile Surface being Bombarded by Reactive Hydrogen Ions which Results in the Splitting of Polymeric Chains in the Fibers. Highly Polar Functional Groups such as O-H are Left Anchored on the Fiber Surface Increasing the Water-Sorption Property of the Treated Fabric**

Some of these dangling groups may be highly polar which increases the water-sorption attribute of the treated material. This is proven by the FTIR spectra of the sample exposed

to the higher discharge at longest exposure time (60 min) shown in Figure 8. A higher O-H absorption peak is seen, thus, the reason for the sample's reversion to a more hydrophilic state.



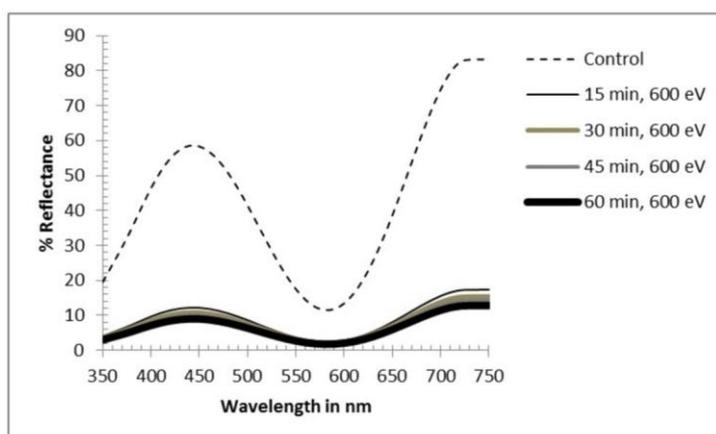
**Figure 8. FTIR Spectra of the Pristine and Treated Samples**

For the low energy group, a different mechanism seems to dictate the alteration of their wetting characteristic. The  $H_n^+$  constituents of the ion shower are non-abrasive at lower beam energies; hence do not produce any surface roughening. It would seem that the bombarding ions' dominant effect is in the strengthening of the material's hydroxyl bonds. Plausibly, a form of hydrogenation may have occurred which increased the bonding sites for water molecules. FTIR data shows that treated samples have strong O-H signals compared to the control thus promoting greater surface activation. Alternatively, from a physical standpoint, fabric capillary pores may also play a vital role in enhanced water uptake. It can be recalled from the Lucas-Washburn equation, the rate of liquid capillary rise in porous media is a function of the fiber radius ( $r$ ). It is suggested here that the high rates of water uptake are manifestations of enlarged " $r$ " or fiber swelling, perhaps due to the heating effects of ion bombardment. Irradiation in the low energy regime could have increased the radii of the pores without necessarily damaging the fibers. As can be gleaned in Figure 6, fiber radii of treated samples are relatively larger than that of the pristine sample.

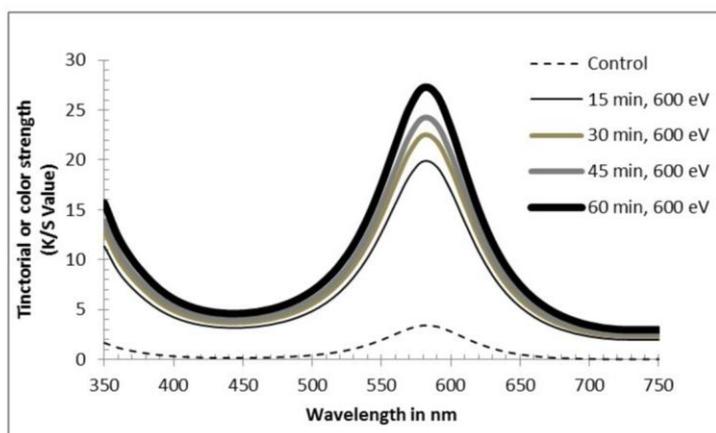
Results of color measurements using the SpectraScan 5100+ are shown in Table 1. Higher  $L^*$  values correspond to lighter shades while lower values show darker shades. Figures 9 (a) and (b) show representative spectral curves of reflectance ( $R$ ) and tinctorial strength ( $K/S$ ) over the visible spectrum from 400 – 750 nm. Compared to the control, treated samples showed significant decrease in reflectance increasing the color strength ( $K/S$  value) of the dye.

**Table 1. Color Properties of all Samples at Different Beam Energies and Processing Times**

Sample treatment	Treatment time in minutes	K/S	L*	a*	b*
Control (Untreated)	0	2.8	18.1	53.0	-99.5
400 eV treatment	15	7.6	15.6	44.1	-92.1
	30	5.2	6.3	34.0	-64.6
	45	6.8	2.7	17.6	-45.2
	60	8.4	11.2	42.3	-79.3
600 eV treatment	15	19.9	1.7	11.4	-36.4
	30	22.5	0.9	5.9	-24.5
	45	24.2	1.0	6.2	-25.6
	60	27.2	0.8	5.5	-23.4



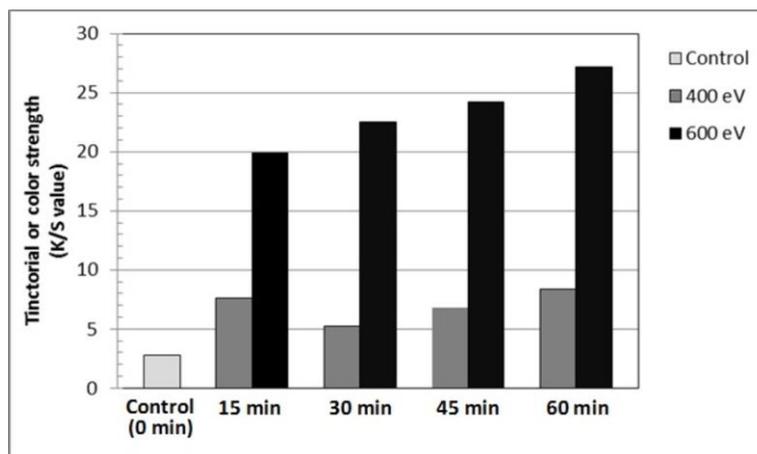
(a)



(b)

**Figure 9. (a) Representative Spectral Curves of Reflectance (R) for the High Energy group at Different Processing (Treatment) Times Plotted against the Visible Spectrum from 350 – 750 nm. (b) Tintorial Strength of the Same Samples. The Strength is Expressed in Terms of the Highest K/S Value**

The K/S values for all samples are shown in the bar graph of Figure 10. Generally the deepness of hue increases with increase in discharge and processing time. This shows that the dyestuff has better affinity to the irradiated samples as against the control.



**Figure 10. K/S Values for all Dyed Samples (i.e. control, both high and low energy groups) at Different Processing Times**

As seen in the FTIR spectra of Figure 8, the strong  $C\equiv C$  functional group at  $2370\text{ cm}^{-1}$  after treatment, accounts for the improvement in coloration. The carbon-carbon triple bond has two unpaired electrons, one for each carbon atom. The unpaired electrons will form covalent bonds with the anionic groups of the acidic dye [18]. The triple bonds provide more venues for covalent bonding. Referring to the FTIR spectra, the sample exposed to higher discharge condition has a stronger  $C\equiv C$  absorbance compared to both the control and the low energy treated specimen. Thus, a higher concentration of triple bond gives a more intense color yield. This is attributed to the chemical modifications brought about by the irradiation process. It is plausible that the treatment gave rise to molecular reorientation which strengthened the  $C\equiv C$  bonds.

#### 4. Conclusions

Fiber abrasion and disintegration are the morphological effects of low energy hydrogen ion shower treatment on cellulose. Water repellency and dyeability were both optimized with increase in discharge conditions. Among the identified functional groups that are attributed to the morphological effects of ion treatment are the carbon triple bonds at around  $2370\text{ cm}^{-1}$  and the hydroxyl bonds from  $3300\text{--}3500\text{ cm}^{-1}$ . A strong and broad O-H bond such as that shown by the low energy group makes them inherently compatible with water. The carbon triple bonds, having an unpaired electron per carbon atom, serve as avenues for covalent bonding with the anionic groups of the acidic dye. Hence, an increase in the concentration of the triple bonds leads to improved dyeability of the material. The sample irradiated by 600 eV ion shower at 15-minute processing time showed good performance in terms of hydrophobicity and dye adhesion. This treatment caused the fabric to become both dye-adhering (K/S value  $\sim 20$ ) and hydrophobic ( $\theta_A \sim 90^\circ$ ) due to the parallel effects of strengthened carbon triple bonds ( $C\equiv C$ ) and roughened fiber surface. In contrast the sample irradiated by 600 eV at 60-minute processing time (*i.e.*, longest processing time) showed good moisture absorption (low contact angle at  $\theta_A \sim 11^\circ$ ) and best color yield (deepest hue at K/S value  $\sim 27$ ). This is about a tenfold increase in coloration compared to the control. Improved coloration is attributed to enhancements in  $C\equiv C$  bonds while increased moisture uptake is due to stronger O-H polar groups.

There are telling advantages in treating textile solely with ion showers. This study has shown that the wettability and dyeability properties of textiles can be tailored-fit simply by varying the duration and dosage of ion irradiation. This type of textile modification is potentially useful when high value-added garment finishing is required. Apparels with high-end functional properties find importance in specialized areas of medicine, the

military, space technology, biotechnology, water filtration etc. Numerous researches and investigations have shown that plasma treatment of textiles is a fitting green technology that can provide improvements in surface and bulk properties of fibrous materials. This takes the form of enhanced dyeing rates, better color fastness, wash and shrink resistance resulting in advanced, far superior fabrics.

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