

**SALT FREE COTTON REACTIVE DYEING BY PLASMA-NANO CHITOSAN TREATMENT**KARTHIKEYAN K.<sup>1\*</sup>, RAMACHANDRAN T.<sup>1</sup><sup>1</sup> DEPARATMENT OF TEXTILE ENGINEERING, KARPAGAM UNIVERSITY, INDIA*Received 11 October 2015, Accepted 25 December 2015***ABSTRACT**

The main problem in dyeing with reactive dye was low affinity caused by repulsion forces between electronegative charges in both cellulose fiber and reactive dyestuff. To overcome these forces in dyeing processes, a large amount of electrolyte was needed, thus the discharged wastewater from dye house creates unavoidable environmental threats due to very high solid concentration. Substantial remedies were being considered within the textile processing sector to reduce the effluent pollution and to fulfill the environmental regulations. In this work, plasma-nano chitosan treatment as an environmentally friendly method was employed to modify surface properties of cotton fabric to develop salt free reactive dyeing. Plasma treated cotton surface characteristics were evaluated using FTIR. The surface activation using air plasma introduces different oxygen-containing polar functional groups such as C-O, C=O, and O-C=O in cotton fabric. The plasma treatment resulted in hydrophilization of the surfaces through oxidation. Cotton surface functionalized by chitosan nanoparticles was investigated through electrokinetic (zeta  $\zeta$ ) potential measurement. The color strength (K/S) and color fastness of the dyed samples were investigated and compared with the conventional dyeing process. The results clearly demonstrated that reactive dyes were efficiently exhausted onto cellulosic fabrics under neutral conditions in absence of salt due to change of electrokinetic potential of chitosan treated cotton samples. Color strength of dyed modified cotton was markedly enhanced with the increment of chitosan concentration. The effluent showed reductions of total dissolved solids (TDS) content in salt free reactive dyeing.

**KEYWORDS**

Chitosan; colour strength; dyeing; fastness; plasma.

**1. INTRODUCTION**

The textile coloration and finishing industry is one of the major contributors to environmental pollution. This is mainly due to the discharge of non-biodegradable inorganic salts, auxiliaries and organic matter such as dyes to the effluent. The effluent treatment can play a significant role in reducing discharge pollution. However, these treatments are expensive and produce highly concentrated solid wastes. Therefore, the better approach would be required to improve the surface chemistry of textile fibers for reducing the discharge inorganic salts. The excellent washing fastness is due to the covalent bonding between the fiber polymers and the reactive dye molecules under alkaline pH conditions. The charge repulsion between dyes and cotton can be overcome by adding an electrolyte, such as sodium chloride or sulfate, which screens the surface charge of cotton (Allegre, 2006). Irrespective of the dyeing method, just about all of the salt is drained to effluent. Such effluents are characterized by high levels of dissolved solids. As an attempt to

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reduce salt usage, a number of researchers cationized cotton fibers through chemical modifications with chemicals containing cationic groups which were not environmentally safe (Wang, Ma, 2009).

The electrokinetic (zeta,  $\zeta$ ) potential is part of the total potential drop occurring in the intermediate surface layer at the boundary of the solid/liquid phases as a consequence of the ion distribution from the solid surface to the liquid mass. At the interface of electrically charged textile fibers and an aqueous solution of electrolyte, surfactants or dyes electric double layer is set up (Grancaric et al., 2005). Moving one of these two charged surfaces results in electrokinetic potential. Immersed in water (pH 6.5 - 7.0) cellulosic fibers show negative values of the zeta  $\zeta$ -potential (-10 mV to -60 mV) and negative surface charge, because chemical functional groups dissociate in water (Stana-Kleinschek, Ribitsch, 1998). There are few zeta potential measurement principles like electrophoresis, sedimentation potential, electro osmosis, streaming potential/current, potential of colloidal vibration, and the electrokinetic sound amplitude (Biscan et al., 2008).

Chitosan is a natural, renewable resource; a polycationic biopolymer that possesses wide spectrum of dyeability improvement activities. Chitosan has the same backbone with cellulose except for its acetamide group instead of a hydroxyl group (Di et al., 2011). Affinity of chitosan to cotton would be by physical forces because of the similar structures of chitosan and cotton (Fras Zemljic et al., 2008). Another possibility mentioned for binding the chitosan to cellulose was cross-linking by formation of Schiff base between cellulose's reducing end and the amino group of chitosan (Strnad et al., 2008). Cellulose fibrous material has to be appropriately activated for efficient irreversible chitosan adsorption, it is essential to create appropriate binding sites such as carboxylic/aldehyde groups (Lidija et al., 2009). For this purpose, different standard chemical processes have been used, which usually modify the fibers both structurally and chemically and, in addition to frequently being ecologically less desirable, are detrimental to the fibers mechanical properties. Plasma treatments can be used as completely harmless surface activation procedures. Plasma can be described as a (partially) ionized gas and is generated by applying either high temperatures or strong magnetic or electromagnetic fields to a gas. The latter method is used for polymer functionalization (Persin et al., 2011). Plasma treatment of textiles has been investigated as an alternative wet chemical fabric treatment and pre-treatment processes. Glow discharge plasma of oxygen, nitrogen, etc. introduces functional groups like hydroxyl, peroxides, and amino polymer surface (Kang et al., 2004). The technique of plasma treatment is an effective surface modification method to save processing costs and to avoid environment pollution (Shahidi et al., 2007).

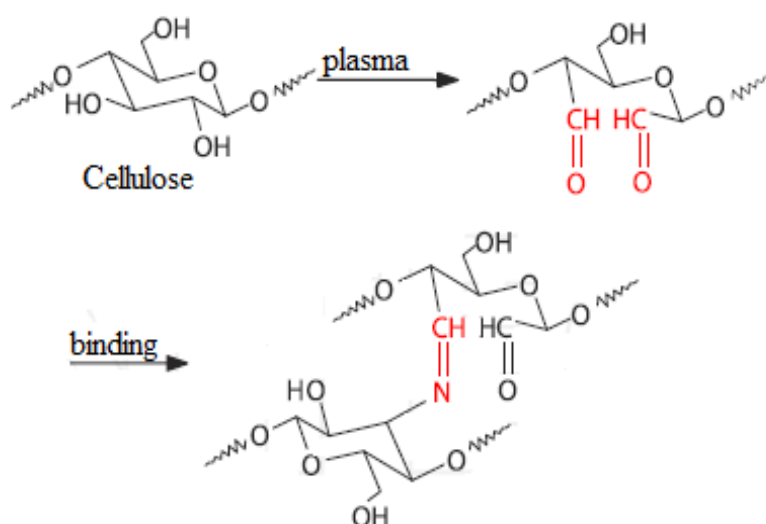


Figure 1: Scheme of plasma oxidation and chitosan binding onto cellulose

The chitosan-treated fabric showed an improvement in the fixation of reactive dyes. This result was explained by the increased exhaustion of negatively charged reactive dyes onto the cotton, whose negative potential on the fiber surface was suppressed by the cationic chitosan treatment. Consequently, when an alkali was added to the dyebath, a substantial quantity of dye was available for the reaction with cotton.

The unique character of chitosan nanoparticles for their quantum size effect promised to exhibit superior dye ability improvement (Hu et al., 2006). The main objective of this research is to explore the possibilities to reduce the dyes and chemical auxiliaries in the dyeing effluents of cotton reactive dyeing by improvement of the dye exhaustion into the plasma activated cotton fabric with use of chitosan nanoparticles.

## 2. EXPERIMENTS

### 2.1. Materials

Scoured and bleached cotton fabric for dyeing having the specification of 140 g.m<sup>-2</sup>, 34 ends.cm<sup>-1</sup>, 33 picks.cm<sup>-1</sup> and yarn count 40/1 was used. The cotton fabric is further treated with 3 g.L<sup>-1</sup> of non-ionic detergent under the boiling condition for 4 hours, after which time it is thoroughly rinsed and air dried at room temperature. Chitosan (Degree of deacetylation 92.5%, Molecular weight 1,000,000 Da) was used for preparation of nanoparticles. Two reactive dyes namely C. I. Reactive Black 5 (C.I. 20505) and C.I. Reactive Red 6 (C.I. 17965) was used for dyeing. All other reagents used were of laboratory grade.

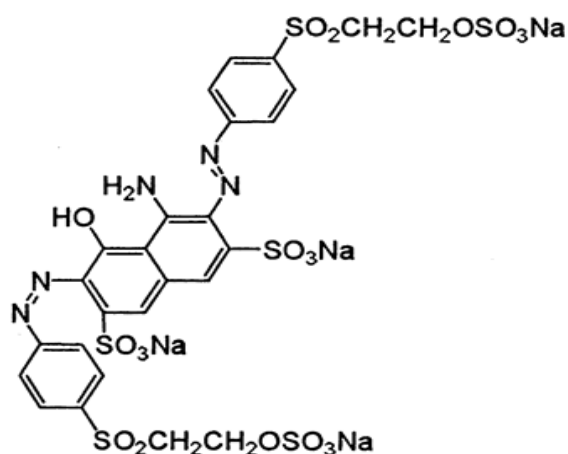


Figure 2: Chemical structure of CI Reactive Black 5

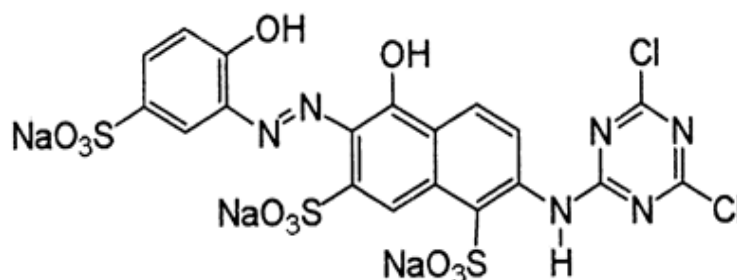


Figure 3: Chemical structure of CI Reactive Red 6

### 2.2. Plasma treatment

The cotton fabric was treated with high frequency (HF) glow discharge plasma operated at a pressure of 0.5mbar. The distance between electrodes is 0.2 cm. The samples were placed between electrodes and treated on both sides, each side for 60s (60s x 2). In all treatments, a uniform glow discharge plasma system operating with air as a processing gas. Due to interactions between air and activated surface, plasma treated fabric was conditioned for 24 h at standard atmospheric condition accordingly to ISO 139 test method.

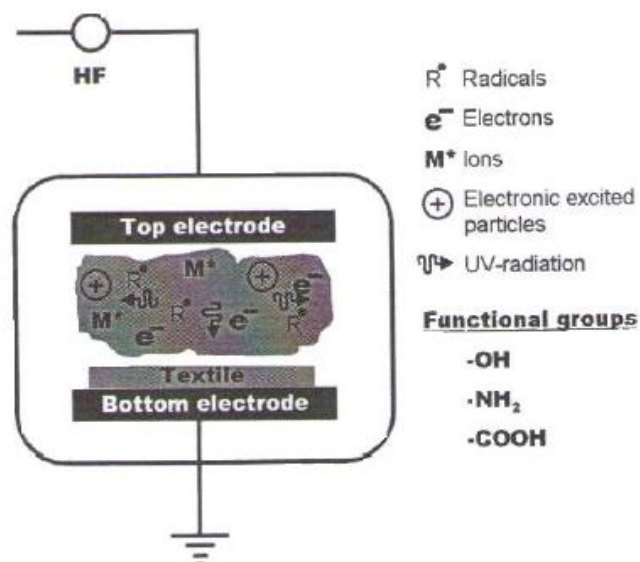


Figure 4: Schematic representation of plasma process

### 2.3. Preparation of chitosan nanoparticles

Chitosan was dissolved in a dilute aqueous acetic acid solution of 0.5% ( $\text{g.L}^{-1}$ ) under microwave irradiation. Aqueous ammonia was then dropped into the chitosan solution to precipitate the chitosan. The obtained gel-like swollen chitosan was washed to neutral with Deionized water, and was then transferred into a 25 mL volumetric flask.

The total volume of liquid was added to 25 mL with Deionized water. An ultrasound processor with a 6mm probe was used and it was put into the volumetric flask. Ultrasound treatment was conducted under an ice-water bath. Finally, a milky nano-emulsion chitosan was obtained.

### 2.4. Pre-treatment with chitosan nanoparticles

Pre-washed cotton fabrics were soaked for 15 minutes in chitosan nano-emulsion at five different concentrations separately 0.01, 0.05, 0.1, 0.3 and 0.5% (w/v).

The padding processes were then completed with pick up weight of around 80%. All padded samples were dried at 100°C for 3 minutes, cured at 150°C for 3 minutes and finally rinsed with warm water (40°C) for 1 min. Finally fabric rinsed with running cold water and dried again.

### 2.5. Streaming Zeta Potential Measurement

Streaming zeta potential experiments were carried out with the Electro Kinetic Analyzer (Anton Paar: VA, USA) using the Cylindrical Cell developed for the measurement of fibrous samples. For each measurement a fiber plug was placed between the Ag/Gal disc electrodes of the Cylindrical Cell. Zeta potential at pH 10 was investigated with the background electrolyte of 1 mM KCl solution. Surface conductivity of the fibrous samples was not taken into account.

### 2.6. Reactive Dyeing

To emphasize the differences in dye uptake between the plasma-nano chitosan treated and untreated cotton fabrics, dyeing properties were investigated with Reactive Black 5 and Reactive Red 6. Dyeing was carried out in a dye bath containing 3% (o.w.f) of dyes with liquor ratio 1:30.

Cotton fabric was added at 35°C and kept at this temperature for 30 min. The temperature was raised to 60°C and 80°C for RB 5 and RR 6 respectively and fixation was subsequently conducted for 40 min by addition of sodium carbonate (15 g.L<sup>-1</sup>).

Dyed samples were thoroughly washed in hot water, then soaping with a solution containing 4 g.L<sup>-1</sup> soap at 90°C for 15 min. Washing carried out for one more time to ensure good washing fastness and finally rinsing with cold water then air dried. For comparison, conventional dyeing carried out in untreated cotton fabrics.

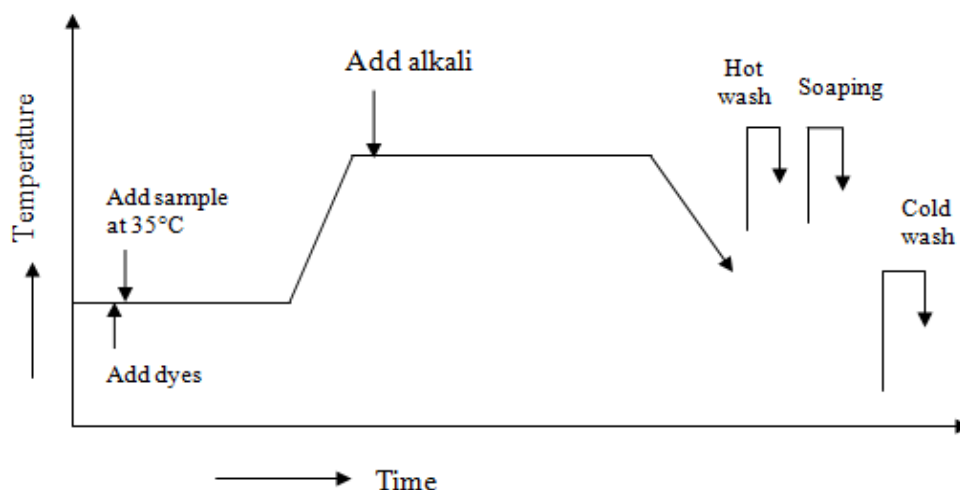


Figure 5: Scheme of salt free reactive dyeing process

## 2.7. Evaluation of Dyed samples

The reflectance of dyed samples and colour coordinates CIE L\*, a\*, b\* values were measured on X-rite spectrophotometer, colour-eye 5000 equipped with integrated sphere using illuminate D65. Colour strength (K/S) of the dyes samples were calculated using according to Kubelka- Munk equation.

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

- R: Decimal fraction of the reflectance of undyed fiber.
- K: Absorption coefficient.
- S: scattering coefficient.

## 2.8. Fourier Transform-infrared analysis

Fourier Transform-infrared measurements carried out using a Nicolet 670 instrument (Thermo scientific). An average of 20 scans was recorded in the attenuated total reflection (ATR-Smart Endurance) mode.

## 3. RESULTS AND DISCUSSIONS

### 3.1. Effect of plasma treatment on fabric properties

The FTIR spectra of the untreated and plasma treated cotton samples were taken to determine the chemical changes that could have occurred as a result of a plasma treatment. Figure 6 shows these spectra. An increase in absorbance at the 1629 cm<sup>-1</sup>(C=O) band and 1080~1300 cm<sup>-1</sup>(C-O) group was noticed after plasma treatment. These functional groups were produced on the fabric by the reaction between the active species induced by the plasma and the fabric surface atoms. There were five vibrational modes corresponding to the carboxyl group. The bands were obtained at the frequencies, 667.3, 1002.9, 1203.7, 1629.3 and finally at 3334.9 cm<sup>-1</sup>. On plasma treatment, an increase in the value of normalized peak intensity for the =C-O group was observed. The increase in =C-O groups which indicate the formation of more carboxyl (=COOH-) groups. Possibly, the presence of oxygen in the air plasma promoted the formation of carboxyl groups in cellulose substrate.

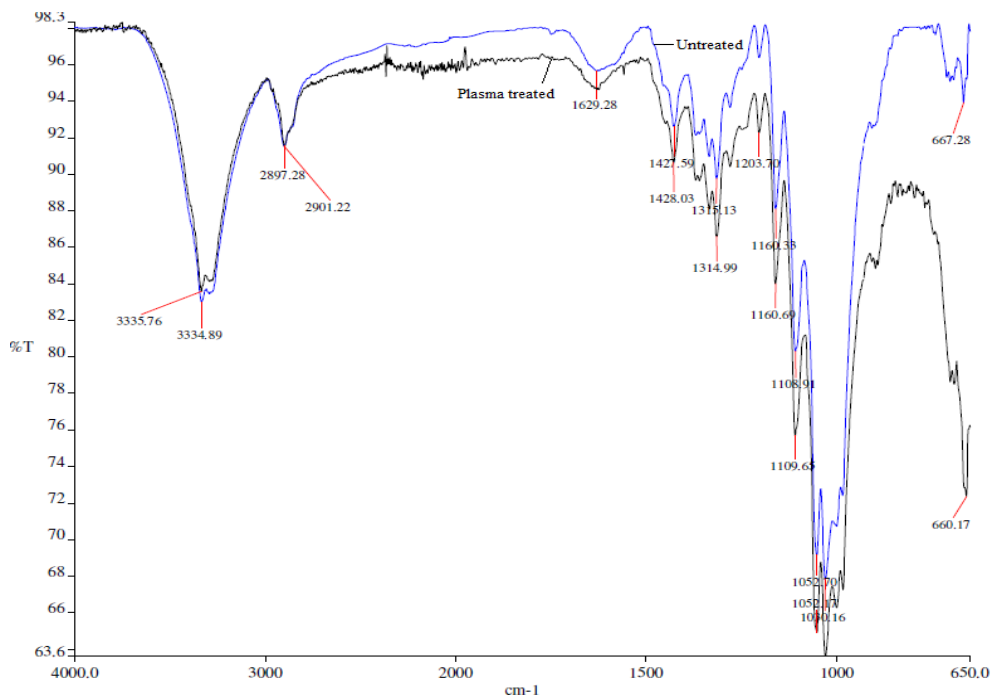


Figure 6: FTIR spectra of untreated and plasma treated samples

### 3.2. Morphology of treated sample

The chitosan nano-emulsion consisted of positive charged nanoparticles with average size of 250nm as determined by laser scan. The emulsions had zeta potentials of +25 mV and pH values around 6.7. Surface morphology of the chitosan nanoparticles was investigated through Transmission Light Microscope (x1000 magnifications). As shown in figure 7, the particles were in irregular shapes, mainly in the shape of triangles and rods.

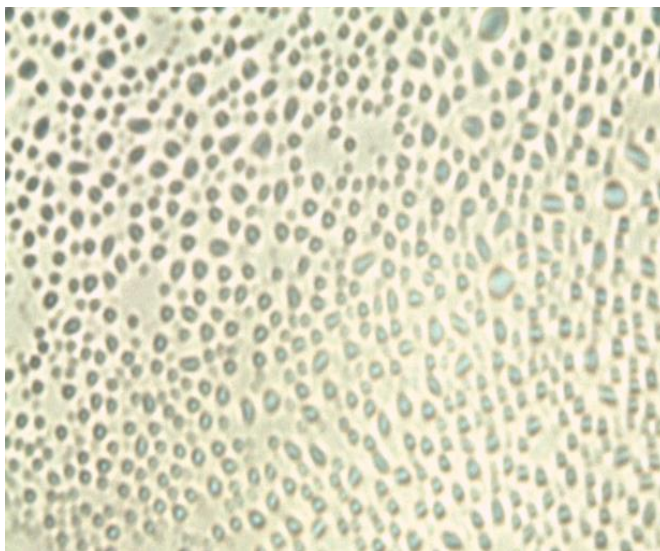


Figure 7: TLM image of chitosan nanoparticles

The Chitosan nanoparticles in the emulsion accumulated onto the surface of cotton aggregated together during drying and finally formed a rough film, promising a huge surface area that could be useful in the dyeing process. Chitosan has the same backbone with cellulose except for its acetamide group instead of a hydroxyl group. The pH of the emulsion of the nanoparticles is more favorable for the carboxyl groups to confer negative charge, which attracted more positive charged chitosan nanoparticles. These nanoparticles

were believed to adhere on the individual fiber surface by electrostatic and other physical forces. Chitosan content on the treated cotton fabric was investigated through UV-vis absorption method. It has been observed that the chitosan content was  $96 \text{ mg.g}^{-1}$  in the plasma- nano emulsion treated fabrics.

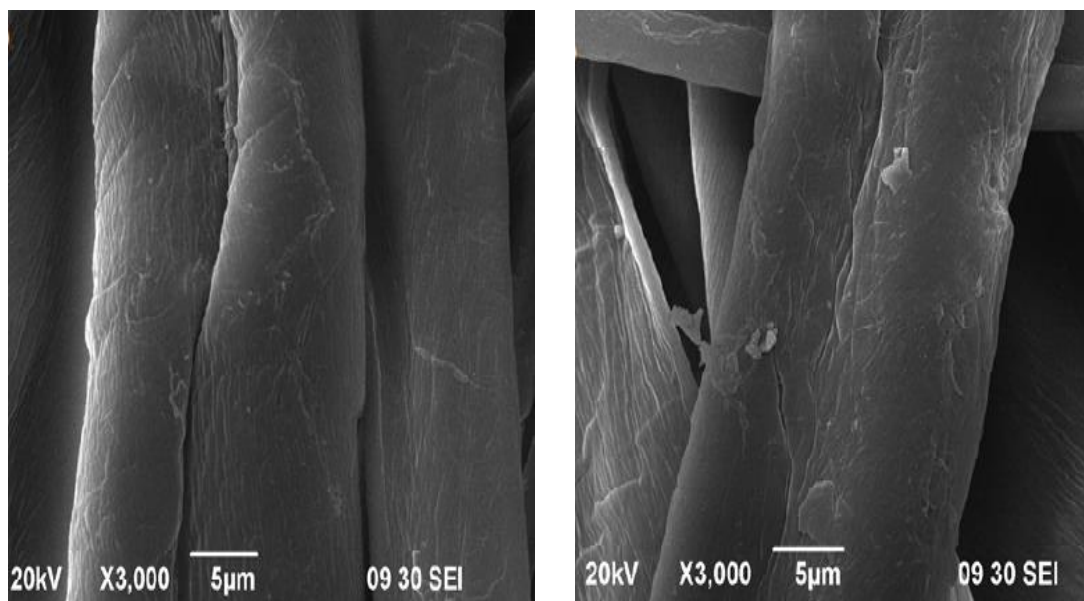


Figure 8: SEM image of the cotton (Left) & chitosan treated cotton (Right)

### 3.3. Zeta Potential Measurements

Zeta potential was investigated in streaming current method at pH 10. It was observed in table 1 that untreated cotton has low zeta potential which due to dissociation of surface acidic functional groups of cellulose. Zeta potential results imply that cotton electric charge was noticeably changed by nano chitosan treatment due to available amino groups.

Table1: Zeta Potential Value

Sample	ZP ( $\zeta$ ), mV at pH 10
Untreated	-19.3
Chitosan Treated (0.01%)	-16.3
Chitosan Treated (0.05%)	-16.0
Chitosan Treated (0.1%)	-15.5
Chitosan Treated (0.3%)	-15.2
Chitosan Treated (0.5%)	-14.9

### 3.4. Colour Strength

The colour strength of untreated and plasma-nano chitosan treated dyed samples were reported in table 2. The plasma-nano chitosan treated sample had higher colour strength and so absorbed more dye than the untreated samples. It was due to the formation of a greater number of new functional groups on the surface of the plasma treated sample, which attached more chitosan in cotton fabric (combination of electrostatic and electro-dynamic interactions). As the chitosan concentration increases, the dye uptake also increases. The pre-treatment of cotton fabric with chitosan nanoparticles introduced new functional amino groups which increased the electrostatic attraction of dyes and also the substantivity towards cotton. The cationic charged amino groups could be involved in the absorption of anionic auxochromes of reactive dyes. Increasing the number of active functional groups due to plasma activation in the cotton surface enabled the absorption of higher amounts of chitosan and, consequently, a higher amount of



amino groups responsible for dyeing. It has to be pointed out that several past researches showed that, in some cases, when chitosan interacts with non-activated cellulose, absorption could also be irreversible (Cakara et al., 2010). That irreversible absorption of chitosan onto weakly acidic cotton fabric was predominately driven by a non-electrostatic attraction. Myllitye et al. (2009) also reported that a non-electrostatic interaction between chitosan and cellulose. This could be attributed to specific structural interaction between chitosan and cellulose (H-bonds and hydrophobic interactions). The higher amount of amino groups in plasma activated nano chitosan treated samples increased the probability that a protonated amino group met electrostatic bond with reactive dye anions under neutral conditions. This result further affirms that chitosan increases the amount of dye uptake in the treated cotton fabrics without salt. K/S values of chitosan treated cotton fabrics revealed that the chitosan has an incremental effect in dyeing processes.

Table 2: K/S values of dyed sample

Dyes	Chitosan concentration (%)	$\Delta E$	K/S
Reactive Red 6	0	-	8.246
	0.01	1.213	7.869
	0.05	1.006	7.994
	0.1	0.652	8.253
	0.3	0.754	8.489
	0.5	0.934	8.502
Reactive Black 5	0	-	5.985
	0.01	1.134	5.653
	0.05	1.066	5.785
	0.1	0.733	5.881
	0.3	0.433	6.052
	0.5	0.496	6.073

### 3.5. Colour Fastness Properties

The durability of the finish applied on the textile material is extremely important in washing, Crocking and light conditions, it has been assessed and was given in Table 3 for both reactive dyes respectively. There was no considerable difference between the colour fastnesses to washing, crocking and light of the samples. The attachment of the dye molecules onto the partially-modified cellulosic substrate is by covalent bonding since no dyes strips out from the dyed sample.

Table 3: Colour fastness properties

Dyes	Chitosan concentration (%)	Wash Fastness		Wet Crocking Fastness	Light Fastness
		Colour change	Staining on cotton		
Reactive Red 6	0	4	4-5	3-4	4
	0.01	4	4-5	3-4	4
	0.05	4	4-5	3-4	4
	0.1	4	4-5	3-4	4
	0.3	4	4-5	3-4	4
	0.5	4	4-5	3-4	4
Reactive Black 5	0	4	3-4	2-3	4
	0.01	4	3-4	2-3	4
	0.05	4	3-4	2-3	4
	0.1	4	3-4	2-3	4
	0.3	4	3-4	2-3	4
	0.5	4	3-4	2-3	4



Light fastness is directly linked with dye concentration in the substrate. Increase in depth of the shade has positive effect on light fastness on treated sample. The fastness values of all such dyed samples are quite satisfactory and comparable with those of conventional dyed samples.

### 3.6. Physical properties

Cotton dyed samples were tested for physical properties such as air permeability, vertical wicking and tensile strength. Plasma-nano chitosan treated dyed fabric performance was compared with untreated dyed fabric. Also plasma effect on cotton fabric physical characteristics evaluated.

The plasma treated sample has high water wicking than the untreated sample. It indicates that increase in hydrophilicity after plasma activation in cotton fabric due to the formation of new functional groups.

It was inferred from the table 4 that there was a change in air permeability of the plasma- nano chitosan treated cotton fabric as compared to the untreated one. There are some factors affecting the air permeability of the fabric, e.g., the fabric structure, thickness, and surface characteristics, etc. The fabric thickness has a significant effect on the air permeability values of the fabric, as the air permeability tends to decrease as the thickness increased. It is postulated that the plasma treatment induces a certain degree of roughness on the fiber surface which increases the fabric thickness and changes the fabric surface characteristics. Plasma has a permanent effect on air permeability as air is kept inside the plasma-treated fabric and cannot escape easily. Also reduction of air permeability was speculated from a slight thickening of the fibers due to a layer of nanoparticles. All these factors contribute towards the lower air permeability. But however, nanoparticles of chitosan incorporated on the individual fiber surface by electrostatic and other physical forces not the inter-fiber voids in the fibrous network. The slight significant losses of air permeability in the plasma-nano chitosan treated fabrics have not affected intact breathability of the cotton fabrics. It is also obvious from Table 4 that tensile strength loss slightly significant after the process. The slight loss of strength is mainly due to the oxidation and stiffening of the molecular backbone after cross-link formation.

Table 4: Physical properties of dyed sample

Materials	Chitosan concentration (%)	Tensile Strength-Warp (N)	Vertical Wicking (cm in 5 min)	Air Permeability (l/m <sup>2</sup> /s)
Plasma treated fabric	-	330.5	6.6	442.6
Reactive Red 6 dyed fabrics	0	351.5	4.5	480.5
	0.01	333.5	6.5	425.5
	0.05	330.6	6.9	420.7
	0.1	328.9	7.5	416.1
	0.3	325.5	8.3	413.0
	0.5	322.9	8.4	410.7
Reactive Black 5 dyed fabrics	0	350.7	4.5	478.5
	0.01	329.9	6.4	428.5
	0.05	327.2	6.8	426.3
	0.1	325.4	7.4	421.0
	0.3	323.3	8.2	418.2
	0.5	320.6	8.3	414.0

### 3.7. Comparative Effluent Analysis

Table 5 shows that new method of innovative environmental friendly dyeing provided around 75% TDS reduction in the effluent of both CI Reactive Red 6 and CI Reactive Black 5. This result indicates minimum effluent load as the maximum fixation of dye was assured through plasma- nano chitosan treatment and without addition of salt for exhaustion which has reduced total dissolved salt significantly. However perceived minimum TDS content in the effluent was due to some unfixed dyes and alkali used for fixation of reactive dyes.

Table 5: TDS of dyeing effluents

Dye (3%)	Sample (Dyebath recipes diluted 100 times)	TDS (mg.L <sup>-1</sup> )
CI Reactive Red 6	Untreated Sample	1360
	Chitosan Treated Sample	350
CI Reactive Black 5	Untreated Sample	1390
	Chitosan Treated Sample	365

## 4. CONCLUSION

In this work, the environment friendly salt free reactive dyeing of cotton fabrics through air plasma treatment and use of chitosan nanoparticles had investigated. For this purpose, the cotton fabrics were treated with air plasma and different amounts of chitosan nanoparticles. The plasma treated sample has high water wicking than the untreated sample which indicates that increase in hydrophilicity in cotton fabric due to the formation of new functional groups. It was proved that electrokinetic potential of cotton fabric changed after nano-chitosan treatment ( $\zeta$  -14.9). Then treated samples were dyed using reactive dyes without addition of electrolyte. It was found that for both reactive dyes by increasing in chitosan concentration to 0.5% (w/v), there was significant improvement in color strength (K/S). Results show that reactive dyes efficiently exhausted onto modified cellulosic fabrics under neutral conditions in absence of salt due to change of electrokinetic potential of chitosan treated samples. Colorfastness properties to washing, crocking and light of the dyed samples were quite satisfactory and comparable with those of conventional dyed samples. Dyeing effluent showed enormous reduction of total dissolved solid content (TDS) which is significance requirement for textile dyeing industry.

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