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Research Article

Reactive Dyeing of Cotton using Chitosan Extracted from Fish Scales

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Abstract

Dyeing of cotton with reactive dyes requires alkali dye bath which requires more electrolytes for exhaustion leading to the environmental concerns during the release of effluent. One of the explored proposals to address this challenge is the salt free dyeing. In this paper, cotton fiber modification technique based on chitosan was studied. Chitosan used was extracted from discarded fish scales. From the study, chitosan demonstrated to be a successful in modification of cotton fabric enabling salt free dyeing with a reduction in dyeing temperature and time. Dyeing on chitosan treated cotton gave better color strength as compared to conventional dyeing method. The wash fastness was better than that of untreated cotton with values of 5 and 4/5, respectively, on a grey scale. The flexural rigidity of cationized Cotton showed lesser value than conventional indicating the treatment making cotton softer. Chitosan extracted from fish scales proved effective in supporting salt free dyeing of cotton fabric which translate to environmental protection.

Keywords: Cotton fabric; Salt free dyeing; Chitosan; Color strength; Fastness.

Introduction

Cotton fibers are extensively used in fabric production because of their exceptional characteristics of comfortability, air permeability, biodegradability, no static electricity build up and excellent hygroscopicity. Reactive dyes are usually used to dye cotton fibers owing to their brightness, diversity of colors, good wet fastness and easier applicability [1,2]. When applied to a cotton fabric in an alkaline environment, the dye reactive group forms a bond linking dye molecule and that of the fiber. Therefore, the dye form part of the fiber and will be less prone to removal by washing as compared to other dyestuffs which stick to the fabric by adsorption[3]. Conventional dyeing process of cotton fabric with reactive dyes requires the use of large amount of electrolyte as exhausting agent [4,5].

These electrolytes lead to enormous environmental problems because they are neither exhausted nor destroyed thus remaining in the discharged dye liquor [2,6,7]. To reduce the use of electrolytes in dyeing, different options have been explored. These options include alteration of dye structure to make it extra attracted to cellulose fiber, surface modification of cellulose fabrics by reacting with compounds having cationic groups or managing dye and salt addition during the exhaustion process [8]. The surface of cotton fiber can be modified to increase dye-fiber interactions by introducing cationic sites either by aminization or cationization. This will overcome the lack of affinity of reactive dye to cotton fiber, therefore providing the possibility of salt free dyeing [9].

Cotton fiber surface modification techniques has been studied by various researchers. Modification of cotton fiber with slaughter house waste which contained keratin hydrolysate for cationization has been studied [7,10]. From the study, it was found that cationized cotton allows higher dye utilization. The effect of cationic modification of cotton fabrics has also been studied with the use of commercial agent Sintegal V7 conc. [11]. The treated fabrics had more positive zeta potential improving dyeing with reactive dyes. Oxidation of cotton fiber with sodium periodate in water and grating it with amino-terminated hyper branched polymer also achieve salt free dyeing [12].

Cationic acrylic copolymer has been used to pre-treat cotton fiber [2] for better dye ability.

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Disodium salt of ethylene diamine tetra acetic (EDTA) acid has also been used in the presence of potassium sodium tartrate as the esterification catalyst to modified cotton [13]. Dye-ability of cotton fabric was also improved by grafting with dimethylaminoethyl methacrylate [14] and chemically with the use of carboxymethyl chitosan [15], acryl amide through Michael addition and Hoffman degradation reaction [16] and with betaine hydrochloride [17]. However, the cationization agents used are expensive and not environmentally friendly. Therefore, there is need for cheaper, eco-friendly and easily available cationizing agents.

In the present paper, dyeing of cotton with reactive dyes without an electrolyte for exhaustion has been studied. This was done by treating fabric with chitosan extracted from discarded fish scales. Extraction parameters were optimized in order to get a high yield extraction solvent. The dyed cotton fabric was evaluated for its color strength, dye exhaustion, perspiration fastness, wash fastness, rubbing fastness, light fastness and flexural flexibility.

Materials and Methods

Fabric

100% half bleached cotton fabric with 21sNe, 28 ends per inch, and 22 picks per inch with fabric density of 137 g/m^2 was used in this experiment. It was kindly supplied by Bahir Dar Textile Share Company, Ethiopia.

Chemicals

Chitosan was extracted from fish scales collected from Ethiopia’s Bahir Dar Lake Tana fisheries waste disposal. Auxiliary chemicals sodium hydroxide (NaOH) and Hydrochloric acid (HCl) used were of commercial grade and used without any modification. A standard commercial detergent was used for cotton fabric rinsing purposes. Reactive Remazol Brilliant blue /vinyl sulphone/ type reactive dyes were employed as shown in Figure. 1.

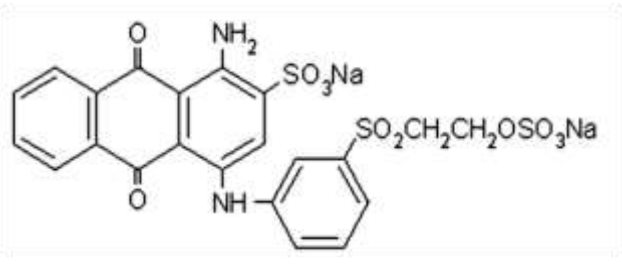

Figure 1. Reactive Remazol Brilliant blue Dye

Preparation of Fish scales

The collected fresh fish scales were washed to remove impurities on the surface. It was done using tap water followed by sunlight drying for 3 days.

Extraction of chitosan from fish scales

Chitosan was extracted from the dried fish scales using the following procedures as shown in Figure 2 [19].

Demineralization step

With the use hydrochloric acid solution (1.0 M) at room temperature, the fish scales were demineralized for 3 hours. Hydrochloric acid was used to get rid of calcium carbonate and to avoid the hydrolysis of chitin. Different ratios used were 1% to 10% insteps of 1% of solid to acid solution. This was done to find the optimal extraction solution. The mixture was stirred until the fish scales became quite spongy after which it was rinsed with distilled water to neutrality eliminating acids and salts. The washed material was placed on a glass tray, and dried for 12 hours at 60°C in an oven.

Deproteinisation step

This step was done with sodium hydroxide solution (1.0 M). The demineralized fish scales from previous step were added slowly to the alkaline solution. Different ratios used were 1% to 10% insteps of 1% of solid to alkaline solution. This was done to find the optimal extraction solution. The temperature of the mixture was raised to 60°C and maintained while stirring continuously for 6 hours. The remaining solid was washed with distilled water until a neutral pH obtaining the final product as chitin.

Preparation of chitosan from chitin

The solid chitin was mixed with a solution of (40%, w/v) sodium hydroxide at a temperature of 100°C maintained for 8 hours. Acetic acid solution (5%, w/v) was used to dissolve the achieved chitosan to a ratio of 1:10 (w/v) solid to acidic solution. The solution was kept for 12 hours and centrifuged to get a clear chitosan solution. Sodium hydroxide solution (5%, w/v) was added little by little into the acidic chitosan solution until a precipitation of pure chitosan formed which was then washed until a neutral pH [20,22].

Percentage dissolution (yield) was calculated using eq. (1) by drying the residual after treatment.

$$Yield\ (\%) = \frac{(W1-W2)}{W1} \times 100 \ldots\ldots\ldots \quad (1)$$

where, W_1 is the original weight of the fish scales while, W_2 is the residual weight of the fish scales after treatment.

Figure 2. Extraction of chitosan from fish scales

Cationization of cotton fabric

The cotton fabric was cationized with the extracted chitosan by exhaustion method. Cationization was done for 5 min at 55°C temperature using different concentration of pure chitosan. The concentration used were 1, 2, 3, 4, 5, 6 and 7%. The presence of chitosan in the cationized cotton fabric was confirmed with the use of FTIR technique. This was done by checking presence of additional functional groups and by measuring the dye uptake phenomena using reflectance spectrophotometer color Eye 3100. Cationization parameters were optimized for the exhaust technique regarding the dye evenness on the fabric and the obtain K/S values.

Conventional and cationized cotton fabric dyeing

Four dyed samples were prepared for this experiment. The first sample was dyed without salt while the second sample was dyed conventional with the use of salt. The third sample was dyed after treating the fabric with chitosan without addition of salt while the fourth sample was dyed with half salt and half treatment with chitosan. The salt dyeing and chitosan treated fabric dyeing were carried out as follows: Dyeing of Cotton Fabric by Conventional Method with Reactive Dye. The bleached cotton fabric was dyed with reactive Remazol Brilight blue 1% shade. Beaker dyeing with a liquor ratio of 1:10 was used. The fabric was immersed at 30ºC into the dye bath and 25 g/L pre-dissolved Glauber's salt was added after 10 min. Dyeing continued while the temperature was increased to 60ºC in 30 min and the remaining 25 g/L pre-dissolved Glauber's salt was added. Dyeing continued for 20 min at 60ºC after which 5 g/L caustic soda was added into the solution and dyeing continued for another 60 min. At the end, the fabric was rinsed at 50ºC two times in 20 min and hot soaped at boiling temperature for 20 min using 5 g/L standard soap [18].

Dyeing of Chitosan Treated (Cationized) **Cotton Fabric**

The fabric was first cationized by immersing it in to a solution containing chitosan for about 5 min at a temperature of 55°C. It was followed by dyeing with reactive Remazol Brilight blue dye for 1% shade. Exhaust dyeing with beakers with a liquor ratio of 1:10 was used. The fabric was immersed at 30 °C to dye bath solution and the temperature was raised to 55°C in 30 min. Then 5 g/L caustic soda was added after 30 min and dyeing continued for another 45 min. The fabric was rinsed at 50 °C for 20 min and hot soaped at boiling temperature for 20 min using 5 g/L standard soap [18].

Evaluation Parameters

Color strength

Color intensities (K/S) were determined at maximum dye absorption wave length of 600 nm using Kubelka-Munk eq. (2) [16,21].

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

where, *K* is the light absorption coefficient, *S* is the light scattering coefficient while *R* is the D65/10 light reflection. K/S values were calculated based on reflection values of untreated and treated cotton fabrics. This value symbolizes the reduction ratio of light owing to absorption and scattering achieved based on reflectance.

Using color eye 3100 reflectance spectrophotometer result in L x a x b D-65 and 10° observer the color intensities (K/S) of Cationized samples at different concentrations was determined. The color strength also of five samples was also determined. The five samples were half bleached cotton, dyed fabric with no salt no chitosan, conventionally dyed fabric, half salt half chitosan treated fabric and fully cationized cotton fabric.

Determination of dye exhaustion

The absorbance of the dye solution was measured before and after the dyeing processes using a Lambda 25 PerkinElmer UV/VIS Spectrometer. The percentage of dye bath exhaustion (E) was calculated using Eq. (iii), where A0 and A1 were the absorbance of the dye solution before and after the dyeing process, respectively. (E) was calculated using eq. (3), where A_0 and A_1 were the absorbance of the dye solution before and after the dyeing process, respectively.

$$E = \frac{A_0 - A_1}{A_0} \times 100\% \quad \text{……..(3)}$$

Perspiration fastness

Perspiration fastness was tested according to ISO 105F10 standard. Specimens of 40×100 mm were attached to same size of multi-fiber to form a composite fabric. The composite fabric was wetted in acidic and basic solutions and placed between acrylic-resin plates under a pressure of 12.5 kPa separately. The test devices containing the composite specimens were placed in the oven for 4 h at 37°C ± 2 °C. The change in color of each specimen and the staining of the adjacent fabrics were assessed and compared with on the grey scales.

Testing of Washing Fastness, rubbing fastness and light fastness

Wash fastness was tested according to ISO 105 C06 2010 standard. Rubbing fastness and light fastness was also tested. AATCC Test Method 8-2007 was used to measure both dry and wet rubbing fastness and AATCC Test Method 16 2007 was used to measure the light fastness.

Testing of Flexural Rigidity

Rectangular sample of 2.5 cm x 20 cm (template size) in warp and weft direction, respectively, was prepared. The sample was placed on the stiffness tester and moved gently until the sample coincided with inclined indicator. The length to which the specimen moved was measured. The flexural rigidity was calculated using eq. (4)

$$G = \frac{1}{8} \times W \times L^3 \quad \text{……(4)}$$

where G is flexural rigidity, W is the GSM of the fabric while L is the bending length

Results and discussion

Concentration optimization for chitosan extraction

Hydrochloric Acid (HCl)

Table 1 shows that as percentage of HCl increased from 1 to 5%, the yield increased significantly as shown in Figure 3. But from 5 to 10%, the increase in yield was low. For this reason, an efficient way of demineralization with HCl is to use 5% v/v solution. A higher concentration will be expensive on the HCl side and at same time higher concentration in the effluent released.

Table 1. Demineralization step using Hydrochloric acid

Con. of HCl, % (v/v)	1	2	3	4	5	6	7	8	9	10
Yield, %	38	39	65	87	91	91	91	91	92	92

Sodium Hydroxide (NaOH)

Table 2 shows the yield of deprotinized chitosan increased with the increment in concentration of NaOH. As concentration increased from 1 to 4%, the yield increased significantly as shown in Figure 3. But from 4 to 10 % increase in concentration, the increase in yield was low. Therefore the optimal concentration for deprotinization step with NaOH was 4% (w/v).

Table 2. Deprotinization step using NaOH

Con. of NaOH, % (w/v)	1	2	3	4	5	6	7	8	9	10
Yield, %	24	67	92	94	94	94	94	95	95	95

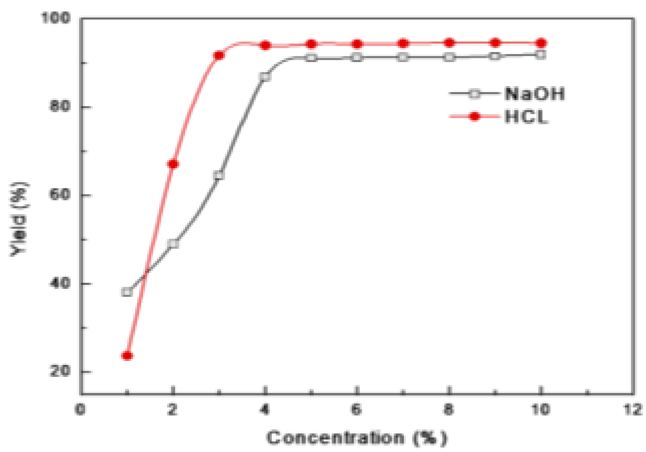

Figure 3. Concentration versus yield percentage

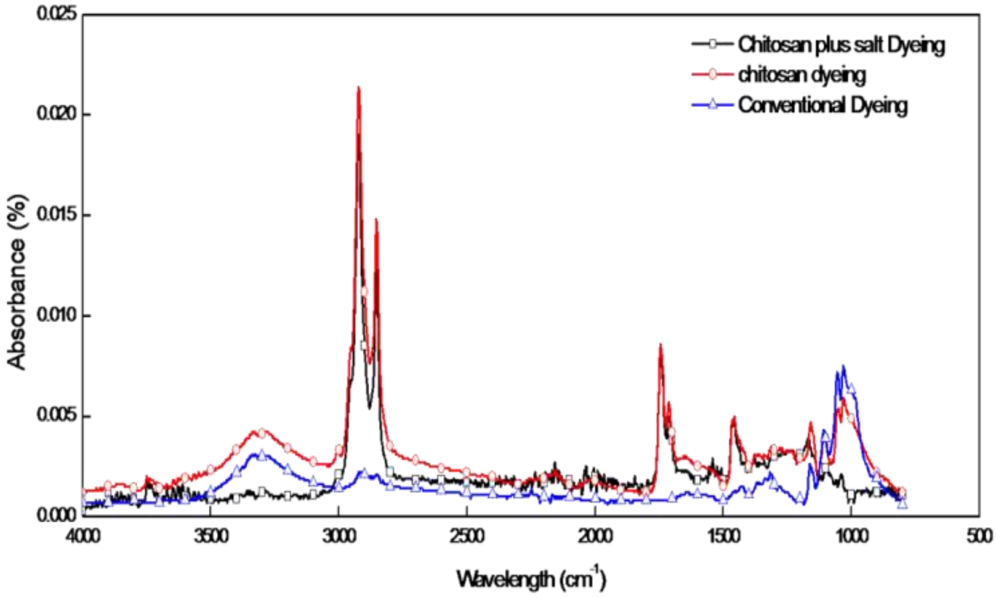

Figure 4. FTIR results

Confirmation of Chitosan Cationization

The peaks in FT- IR curve (Figure 4) showed that there was a change in chemical composition after being cationized by exhaust method, in that, the chitosan was fixed on the fabric. This is recognized by additional amine functional groups. The FTIR spectrum of untreated cotton and treated cotton are shown in Figure 4. Compared with untreated cotton, the absorption peak at about 1660 cm^{-1} is visible in the spectrum of the treated cotton, which is assigned to the bending vibration of C-N. This observation shows the presence of $-CH_2-N^+R_3$ type nitrogen (quaternary ammonium groups). It fully confirmed that the amine group from chitosan was absorbed on the treated cotton fabrics. It designated that the reaction involving cotton fabrics and chitosan groups occurred.

Effect of Cationized Dyeing

Color strength

The color strength of dyed fabric was evaluated by K/S value. This value statistically shows the characteristics of the dye coloring matter on the dyed fabric and a simple way to establish a color as a concentration. The color concentration diminishes as the value for reflectance increases, and vice versa. Using color eye 3100 reflectance spectrophotometer result in L x a x b D-65 and 10° observer the color intensities (K/S) of Cationized samples at different concentrations was determined and it is as shown in Figure 5. From the Figure 5, it can be seen that 4% concentration gave a high K/S value therefore it was used for comparison with conventionally dyed fabric. The values of dyed fabric with Remazol Brilight blue reactive dye with different dyeing techniques but similar conditions are shown in Table 3. It shows that a higher K/S values was obtained with cationized cotton fabric. The increased in K/S value in the fabric treated with chitosan point towards the existence of higher amount of the dye absorbed in the treated fabric.

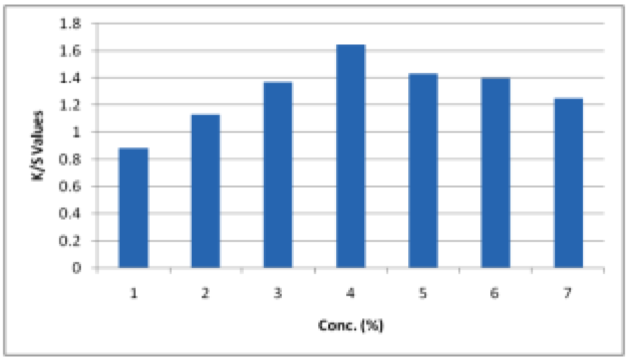

Figure 5. K/S values from different concentration of cationizing agent on cotton fabric

This point to the fact that the improvement of dye absorption took place due to creation of supplementary hydroxyl groups which increases dye attachment sites. This proves the success of chitosan treatment in enabling cotton fabric to be dyed in absence of salt.

Dye exhaustion

The exhaustion of Reactive dye on the samples was calculated based on the results in Table 4 and eq. (3). From Table 4, it can be seen that cationized fabric had high dye exhaustion showing that there was better utilization of dyes.

Table 3. Color eye 3100 reflectance spectro photometer result in L × a × b × D-65 and 10° observer

Color factors \ Samples	Half bleached	No salt no chitosan	Conventional	Half salt half chitosan	Cationized
L*	89.15	62.47	61.06	56.81	58.65
a*	0.35	0.54	-2.12	-5.30	-1.13
b*	8.08	-21.90	-25.46	-29.26	-25.27
X	70.92	29.55	27.32	22.32	25.05
Y	74.49	30.96	29.32	24.37	26.65
Z	69.89	52.23	53.37	49.88	49.09
x	0.329	0.262	0.2484	0.230	0.248
y	0.346	0.275	0.2665	0.255	0.264
Reflect-ance (%)	77.75	25.04	21.39	17.55	19.61
K/S	0.032	1.122	1.4445	1.938	1.648

Table 4. Dye exhaustion at maximum wave length in the dye bath

Samples	Original dye absorption, A_0	Residual dye absorption, A_1	Exhaustion (%)
No salt, no chitosan	1.1243	0.8657	23.00
Conventional	1.4701	0.70013	52.37
Cationized	2.2312	0.6359	71.49
Half salt and half chitosan	1.5861	0.59709	62.35

Perspiration, Wash, Rubbing and Light fastness

Color fastness to perspiration of the cationized fabric was compared with conventionally dyed cotton fabric. It was assessed in respect of color change and staining on multifiber fabric (acetate, cotton, nylon, polyester, acrylic, wool) as shown in Table 5. It can be seen that cationized treated fabric had similar color change with conventionally dyed one but better perspiration fastness on the staining with different fabrics as shown in Table 5. Table 6 shows that the wash fastness of the cationized cotton fabric was good as compared to conventional dyed cotton fabric and there was no staining in all cases. This also confirms the effectiveness of chitosan treatment on cotton fabric for salt free dyeing.

For the case of rub fastness, dry rub fastness of the dyes on cationized cotton fabric was outstanding, which was higher than grade 4; whereas wet rub fastness on the treated fabrics was all half grade lower than that on the untreated ones as shown in Table 7. The wet rubbing of chitosan treated fabrics showed a lower rating than untreated fabric. The chitosan found in the fabric increases the dye sites giving a deeper shade. For this reason, the result of having a deeper shade, the fabric treated with chitosan showed somewhat lesser fastness score as compare to lighter untreated fabric.

Table 5. Perspiration Fastness

Fabric sample	Color change		Staining											
			Acetate		cotton		Nylon 66		Polyester		Acrylic		wool	
	alkaline	acidic	alkaline	acidic	alkaline	acidic	alkaline	acidic	alkaline	acidic	alkaline	acidic	alkaline	acidic
No salt, no chitosan	4/5	4/5	4	4	4/5	4/5	4	4	4/5	4/5	4	4	4/5	4/5
Conven-tional	4/5	4/5	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Cationi-zed	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5	5	4/5	4/5
Half salt and half chitosan	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4

Table 6. Wash fastness

Samples	Color change	Staining	
		Cotton	Polyester
No salt, no chitosan	4/5	4/5	5
Conventional	4/5	4	5
Cationized	4/5	4/5	5
Half salt and half chitosan	4/5	4	5

As given in Table 8, the light fastness was still reasonable. This was most likely due to the fact that the dye penetrability was excellent in the cationized fibers with the intended dye fixation method, which assisted in obtaining high light fastness.

Table 7. Rubbing fastness (ISO 105F09)

Samples	Staining	
	Dry	Wet
No salt, no chitosan	5	4/5
Conventional	5	4/5
Cationized	5	4
Half salt and half chitosan	5	4/5

Table 8. Light fastness (ISO 105B2)

Samples	Color change
No salt, no chitosan	7
Conventional	7
Cationized	6
Half salt half chitosan	7

Effect on Flexural Rigidity

Table 9 shows that treatment of cotton with chitosan reduced the flexural rigidity of the fabric as a result tearing strength was expected to improve. These observations were both in the warp and weft direction.

Table 9. Effect of cationization on flexural rigidity

Sample	**Conventional**			**Cationized**			**Remark**
Direction	Weft	Warp	Overall	Weft	Warp	Overall	Average
Bending Length (cm)	2.1	2.2	2.26	2.0	2.1	2.1	Average
Flexural rigidity	161	193	175	137	183	163	Average

Effect of Different Dyeing Methods

Dyeing of cationized cotton fabric and convention dyed cotton fabric with Remazol Brilight blue reactive dye was carried out as per the procedures stated early. Washing and soaping was done for both samples. The visual observation confirmed that color yield was higher in the cationized sample as shown in Figure 6. For the untreated samples dyed with no salt, the lowest color intensity was obtained but with salt addition, higher color intensity was obtained. This indicates the significance of using salt in conventional dyeing of cotton with reactive dyes. But the presence of chitosan in the fabric improves the sites for dye absorption therefore giving a deeper shade and higher color intensity**.**

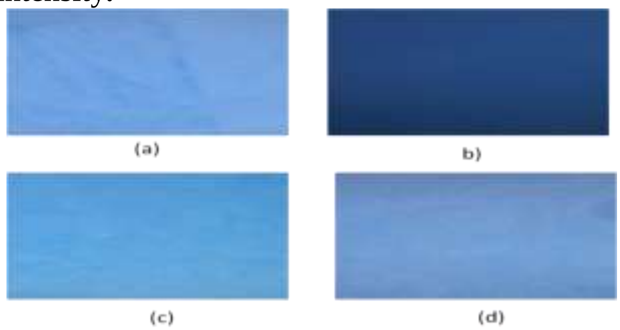

Figure 6. Dyed samples result with different dyeing methods, (a) Conventional dyeing, (b) Chitosan, (c) Salt and Chitosan dyeing (d) No salt and Chitosan

Conclusion

In the present study, chitosan provided cationic site on cotton fabric which was dyed with reactive dyes without electrolytes giving excellent results. When the cotton fabric was treated with chitosan, the primary hydroxyl groups of cellulose were partially modified into amide groups. This enabled cotton to be dyed with reactive dyes in the absence of an electrolyte. The treated cotton fabric gave better color strength when compared with conventionally dyed one had 1.6478 and 1.4445, respectively K/S values at wave length of 600nm respectively. The color fastness on wash, rubbing, light and perspiration was better than that of untreated cotton. The flexural rigidity for cationized and conventional dyeing was 163.22 and 175.10 respectively. It shows that chitosan was effective in improving dye ability of cotton fabric in the absence of electrolyte.

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Conflicts of Interest

Authors declare no conflict of interest.

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