North American Academic Research

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Research

Potential of reactive cationization of cotton over mercerization for dyeing characteristics

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***Accepted**: 07 October , 2020; **Online**: 13 October, 2020*

***DOI** : https://doi.org/10.5281/zenodo.4087379*

***Abstract:** Many natural fibers present in nature, among all the natural fibers, cotton fiber has vital value, it is widely used because of certain desirable properties and is widely available throughout the world. Reactive dyeing of cotton produces wide range of bright colors and provides excellent color fastness because of covalent bonding between fiber polymers. For dark shades, it is essential to mercerize cotton fabric to increase dye-ability, improve luster but it has some disadvantages, mercerization needs specialized machine, high consumption of concentrated caustic soda during mercerization, need to neutralize the fabric after mercerization, use of salt during dyeing, which produces toxic effluent and have high TDS value and that is environmentally unfriendly. To overcome all these problems reactive cationic reagent is used for cotton applied through cationization process, which chemical modify cotton to produce positive dyeing sites of charges on the fabric in the place of existing hydroxyl sites that increases dye affinity, color yield, fixation percentage, produces hygroscopy, dimensional stability and increases fabric strength. For*

cationization, no need of specialized machine, neutralization and exhaust dyeing of cotton does not require salt for exhaustion. It produces less effluent and less toxicity in effluent which is environmentally friendly, all these advantages makes cationization process cheaper and superior thus cationization has great potential to be replaced with mercerization.
***Keywords**: Cotton; Mercerization, Cationization; Dyeing.*

1. Introduction

Since many fibers available in nature but only cotton fiber occupies distinctive position in textile industries owing to its natural feel, handle, look and comfort properties [1]. Prior to dyeing, cotton fabric is subjected to many pretreatment processes, one of the process is mercerization, during mercerization, cotton fabric undergoes physico-chemical, chemical, and structural modifications [2]. Chemical reaction forms alkali cellulose, and physical reaction changes the arrangement of cellulose units into the structure of fiber, to get optimum modifications in properties of cotton fabric, it is depending upon the selection of concentration of sodium hydroxide, time, temperature and tension during the treatment. But for mercerization specialized machine is required, and after mercerization neutralization and washing of cotton fabric is essential to maintain the pH of fabric which also consumes high amount of water [2]. Mostly choice of colorant for the dyeing of cotton fabric is reactive dye, because of their properties like brilliancy, verities of color and high wet fastness of fabric due to covalent bonding between fiber and dye molecule [3,4]. Reactive dyeing of cotton is being done in two stages that is exhaustion and fixation, exhaustion is achieved by the use of salt, preferably Glauber’s salt (Na2SO4) or common salt (NaCl) to overcome negative zeta potential of cotton fibers [5,6,7] and usually the amount of salt required during dyeing i.e. approximately 0.6 to 0.8 kg per kg also depending upon the depth of shade [8,9], and this is the major problem of reactive dyeing of cotton. Further, 100 % exhaustion could not be ensured and due to this, highly saline colored effluent is generated [10]. Fixation of exhausted dyes is done by using alkali such as sodium hydroxide (NaOH) or sodium carbonate (Na2CO3) [11], unfixed dyes are removed in subsequent washing, furthermore addition of amount of salt and alkali depends upon the dye structure, color depth and dyeing method [7, 12].After reactive dyeing process, hydrolyzed dye, salt and alkali are disposed as an effluent, and when the effluent is released from dyeing department it possess high total dissolve contents which is toxic and environmental unfriendly[13], treating this effluent requires an advance tertiary treatment process which increases dyeing cost [14][15]. Industries are in need of alternatives to overcome above problems, many attempts have been made, and can be summarized as modification of dyeing machine, dye chemistry, and modification of cotton using chemicals, compounds, enzymes, nanoparticles, ultrasonic waves, plasma, gamma, and ozone treatment [7]. Many attempts have been made to address this issue, among those efforts the most successful attempt is the chemical modification of cotton by using cationic cites using cationic agent, which increases substantivity and reactivity between fiber and dye molecules [11]. In this work we have compared the treatment of cotton fabric with reactive cationizer (CHPTAC) and a mercerization agent [16]. The results showed remarkable favor for the cationization process. Unlike to mercerization, treatment of CHPTAC is very simple, this process does not need any specialized machine, and can be done on simple padders [17,18]. This treatment chemical modify cotton to produce positive dyeing sites of charges on the fabric in the place of existing hydroxyl sites [11], after cationization, no need of salt during reactive dyeing unlike to mercerized cotton [19,20], this reduces the high consumption of salt, consumes less amount of water during dyeing, and produces less toxic effluent which is environmental friendly [21,22]

2. Materials and methods

2.1 Materials

100 % Cotton bleached fabric, having 110 GSM, whiteness index is 67 according to CIE system, and pH of fabric is 7, is taken from Yunus Textile Mills Ltd Pakistan. Cationic reagent 3-chloro 2-hydroxypropyl trimethyl ammonium chloride (CHPTAC) 60% weight in, with molecular weight 188 H_2O is used for cationization and supplied by Dow Chemicals. Sodium hydroxide ($\geqslant$ 98.0 %) was bought from Sigma Aldrich Pakistan. Wetting agent Mercerol QWLF is used as which is given by Clariant Chemicals Pvt: Ltd. Reactive dye Red KB4L and Black 5 was used in dyeing of optimized mercerized samples and optimized cationized samples, it is provided by Archroma Pakistan. Urea is bought from a local scientific store. Sodium Hydrogen Carbonate is bought from

ICI Pakistan. Ladipur RSK was provided by Archroma Pakistan. All the chemical reagents used, were of analytical grade.

2.2 Methods

2.2.1 Mercerization of cotton

Mercerization was performed manually using a setup of nonadjustable pan & frame. Cotton fabric was fixed in the pins of steel frame in stretched condition and immersed into sodium hydroxide solution. In order to optimize parameters of mercerization, cotton fabric was treated at different, concentration of caustic soda, time of treatment, and temperature during treatment. Wetting agent Mercerol QWLF liquid 8 ml/L was used in caustic solution. Further, cotton fabric was treated with the concentration of 20°Be to 28°Be with the increase of 2°Be after each concentration as shown in Fig. 1, and treatment time was varied from 1 min to 3 min with the increase of 30 sec after each treatment time, and temperature is varied from 40°C to 60°C with the increase of 5°C after each treatment temperature. Subsequently, all the samples were washed two times with fresh water and one-time with hot distilled water and then soaping is done for 5 minutes in a detergent solution at 90 °C, which was made with 2 g/L of Ladipur RSK. And again, fabric was hot washed and neutralized with acetic acid solution of pH 4 for five minutes. All the mercerized fabrics were dyed through rapid pad dyeing. In rapid dyeing, we used Red KB4L dye with concentration of 10 g/L and urea as an auxiliary with 100 g/L concentration, and the optimization of mercerization parameters were carried on the basis of color strength (K/S) values of dyed samples.

2.2.2 Cationization of cotton

Cationization was done on horizontal padding machine, for cationization, cotton fabric was treated with CHPTAC and sodium hydroxide solution, In order to optimize parameters of cationization, cotton fabric was treated at different parameters, such as, cationizer concentration, caustic concentration temperature and treatment time. Further, fabric was treated with CHPTAC varying concentration from 2 % to 10 % o.w.f with the increase of 2 % after each concentration as shown in Fig. 2. Solution of sodium hydroxide was prepared from 1 % to 2 % o.w.f with the increase of

0.2%, and then cotton fabric is padded with cationic solution of different concentration through padding, and then padded with sodium hydroxide solution in different concentration, in last curing is done, curing temperature was varied from 50°C to 70°C with the increase of 10°C, and curing time was varied from 2 min to 10 min with the difference of 2 min as shown in Fig. 2 (d). Subsequently, the fabric was dyed through rapid dyeing procedure for optimization of parameters of cationization, in rapid dyeing red KB4L dye 4 g/l and urea 100 g/l was used. Optimization was carried based on color strength (K/S) values of dyed samples.

2.2.3 Dyeing of mercerized and cationized fabrics

Dyeing of both the mercerized and the cat-ionized treated fabrics done in two different dye solutions. The first one dyeing solution was prepared by mixing 10 g/l Red KB4Ldye, 100 g/L urea and 15 g/L sodium bicarbonate in di-ionized water. The second one dyeing solution was prepared by mixing 10 g/L Black 5 dye, 100 g/L urea, 15 g/L sodium bicarbonate in di-ionized water. Dyeing was done on horizontal padding machine through two dip two nip process at 70 % pick up, after dyeing of mercerized and cationized fabrics, fabrics were dried at 80 °C for 3 min and cured at 150°C for 1 min, then washed, two times with cold wash and one time hot wash and then soaping is done with 2 g/L Ladipur RSK detergent solution in hot water for 15 min and again fabrics were hot washed and dried.

2.2.4 Testing and measurement

Dyed, undyed, mercerized and cationized samples were tested, and measurements were taken as per standards

2.2.4.1 Color strength K/S

Color strength K/S value was assessed on Datacolor 600TM spectrophotometer using illuminant D65 with 10° observer. The minimum reflectance value was taken because having maximum absorption value. The K/S value was calculated by using Kubelka Munk equations. The reflectance value was calculated by equation (1) and reflectance percentage was calculated by equation (2).

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

$$\frac{K}{S} = \frac{(100-R\%)^2}{200R} \qquad (2)$$

2.2.4.2 Determination of fixation

Percentage of dye fixed on the substrate was calculated by equation (3).

$$F(\%) = \frac{b}{a} \times 100 \qquad (3)$$

Where, ‘F’ is dye fixation, ‘a’ shows K/S value of unwashed sample and ‘b’ shows K/S value of washed sample.

2.2.4.3 Tensile strength

The tensile strength of mercerized, cationized, dyed and undyed fabrics was carried out on Universal Titan machine by following ASTM D5035 standard.

2.2.4.4 Absorbency test

Absorbency test of mercerized, cationized, dyed and undyed fabrics was done by drop test and capillary rise method.

2.2.4.5 Drop test

By following AATCC 79 standard for absorbency (drop) test, the drop of water was dropped from the height of 6 cm on the fabric clamped with embroidery frame. The drop of water was assessed at 90° angle and time was noted till the absorption of water from the surface of sample.

2.2.4.6 Capillary rise test

The absorbency of cotton fabric was also assessed through capillary rise test method. In this, sample was cut into 10×4 cm size and marked till 5 cm at the regular interval of 1 cm. The 1%

solution of turquoise dye was prepared. The sample was dipped into solution by 1 cm for 1 minute and then capillary rise of dye was assessed.

2.2.4.7 Color fastness test

2.2.4.7.1 Washing fastness

Washing fastness of mercerized dyed and cationized dyed fabrics was done by following ISO-105-CO3 standard. The samples were cut into 10×4 cm size and attached with multi fiber strip. The solution of soap was prepared. The sample was dipped into soap solution in HT dyeing machine container and then placed into HT dyeing machine at 60°C for 30 minutes and then rinsed and dried. After drying the staining on multi fiber strip and change in color were assessed through gray scale.

2.2.4.7.2 Rubbing fastness

Rubbing fastness was carried out on crock meter in order to measure the fixation of the dye with the fabric. To check the staining white test cloth was put onto the grating and stag by steel wire. The sample was run ten times manually. For rubbing fastness (wet), the wet sample was taken and run ten times over the dyed sample. Degree of staining on undyed fabric was assessed through gray scale.

2.2.4.7.3 Pilling resistance

Test The pilling test of mercerized and cationized fabrics was done on Martindale Pilling Tester by following the ASTM D-4970 standard. For each fabric pilling resistance test two samples was made, one was 140 mm diameter and other was 38mm diameter, these samples then placed in machine, and revolution was kept 1000 rpm and pressure was kept 9 kPa, after completion fabrics were assessed through fabric pilling scale.

3. Results and discussion

3.1 Optimization of mercerization parameters

Three parameters of mercerization were optimized which are varied according to the commercial process, caustic concentration, time and treatment temperature. The optimization was done based on highest color strength (K/S) values of mercerized samples which were dyed through rapid dyeing. The bleached cotton fabric was mercerized at various caustic concentrations, varied from 18°Be to 28°Be. Time and temperature were kept constant i-e 60 sec and 40°C respectively. As shown in Fig.1(a) 24°Be shows highest color strength value. The immersion time was varied from 60 sec to 180 sec. The concentration of caustic and temperature were kept constant i-e 24°Be (optimized) and 40°C respectively. Fig. 1(b) shows 1 min has highest color strength value at 24°Be caustic concentration. As shown in Fig. 1(c) temperature of caustic solution was varied from 40°C to 60°C. And the concentration of caustic and the immersion time were kept constant i-e 24°Be (optimized) and 60 sec (optimized) respectively. Highest color strength value obtained at 50°C.

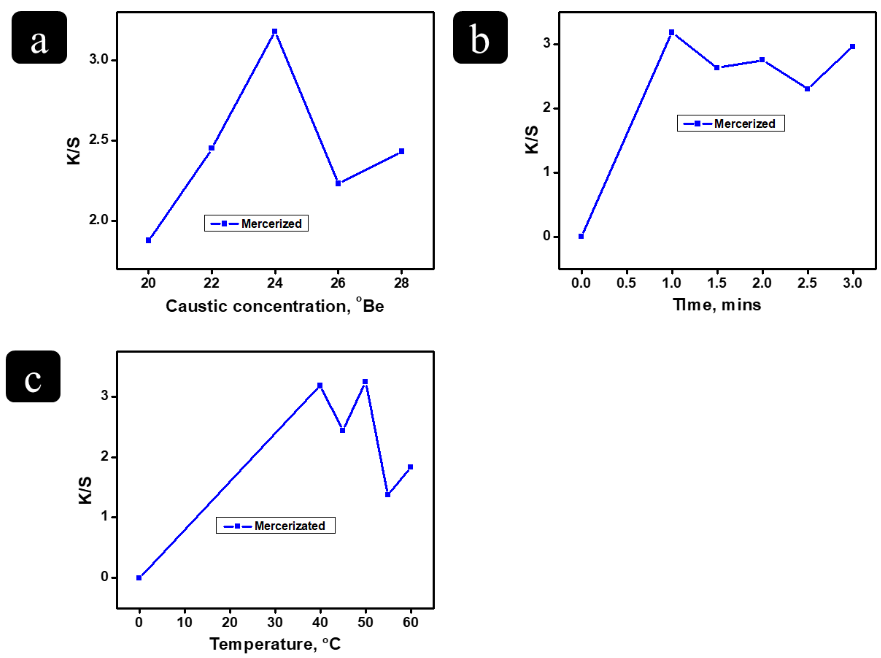

Fig. 1 Mercerization of cotton fabric at different parameters (a) effect of time (b) effect of temperature & (c) effect of caustic concentration

3.2 Optimization of cationization parameters

The optimization of cationization process was also carried out based on color strength (K/S) value of dyed cationized samples. Four parameters were varied. The parameters varied are, amount of cationic reagent, concentration of caustic, curing temperature and time. The amount of cationic reagent was varied from 2% to 10% (o.w.f). The other parameters like, amount of caustic, temperature and time were kept constant i-e 1.8% (o.w.f), 60°C and 6 minutes respectively. As shown in fig. 2(a), 8% cationic amount (o.w.f) shows highest color strength. The amount of caustic was varied from 1.2% to 2% (o.w.f). The other parameters, amount of cationic reagent, temperature and time were kept constant i-e 8% (optimized), 60°C and 6 min respectively. In fig. 2(b) caustic amount of 1.4% (o.w.f) shows highest color strength value**.** The temperature was varied from 50°C to 70°C, other parameters like, amount of cationic reagent, amount of caustic soda and curing time were kept constant i-e 8% (optimized), 1.4% (optimized) and 6 min respectively. Fig. 2(c) shows highest color strength value at 70°C. The time was varied from 2 min to 10 min and other parameters like amount of cationic reagent amount of caustic and temperature were kept constant i-e 8% (optimized), 1.4% (optimized) and 70°C C (optimized) respectively. Fig. 2(d) shows optimum curing time for cationization is 6 min.

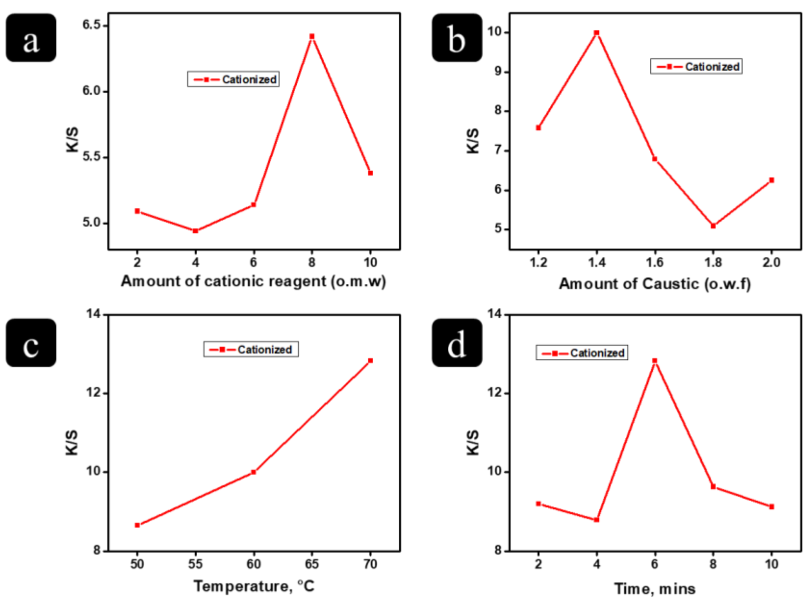

Fig. 2 Cationization of cotton fabric at different parameters (a) amount of cationic reagent (b) amount of caustic, (c) effect of temperature & (d) effect of time

3.3 Effect of treatment on dyeing and other characteristics

3.3.1 Dyeing characteristics

3.3.1.1 Color strength (K/S)

Table. 1 Dye fixation and color yield of mercerized and cationized samples

S.NO	Process	Dye Used	Color yield before washing	Color yield after washing	Fixation percentage
1	Mercerization	Red KB4L	17.0	14.23	83.7 %
2	Cationization	Red KB4L	10.3	9.1	88.3 %
3	Mercerization	Black 5	24.0	18.47	77.0 %
4	Cationization	Black 5	18.2	17.8	97.8 %

Table 1 shows that mercerized samples which are unwashed have higher color strength than cationized samples but during washing process mercerized samples bleed out, it may be because the dye molecules were not properly adhered to the fabric when the fabric gets wet and dye leaches out into the water which produces more dirty effluent and increases cost of dyeing, and after washing the color strength (K/S) values are almost same for mercerized and cationized samples. So it is good to use cationization instead of mercerization because it shows that cationized samples have good dye fixation property than mercerized samples, produces less effluent and reduces overall processing cost.

3.3.1.2 Dye fixation

Table 1 revealed that fixation percentage of cationized fabric is more than the mercerized fabric, this is because mercerized fabric bleed more than cationized fabric, which causes reduction in fixation percentage, cationized fabric need less dye as compare to mercerized fabric for the same shade, cationization seems to be economical and green process.

3.3.2 Other characteristics

3.3.2.1 Tensile strength

As shown in fig. 3 (a,b) cationization increases tensile strength, from both warp and weft side. And to check the effect of dyeing on tensile strength, mercerized and cationized fabrics were dyed with two type of dyes, Black 5 and Red KB4L, after dyeing both fabrics were subjected to tensile

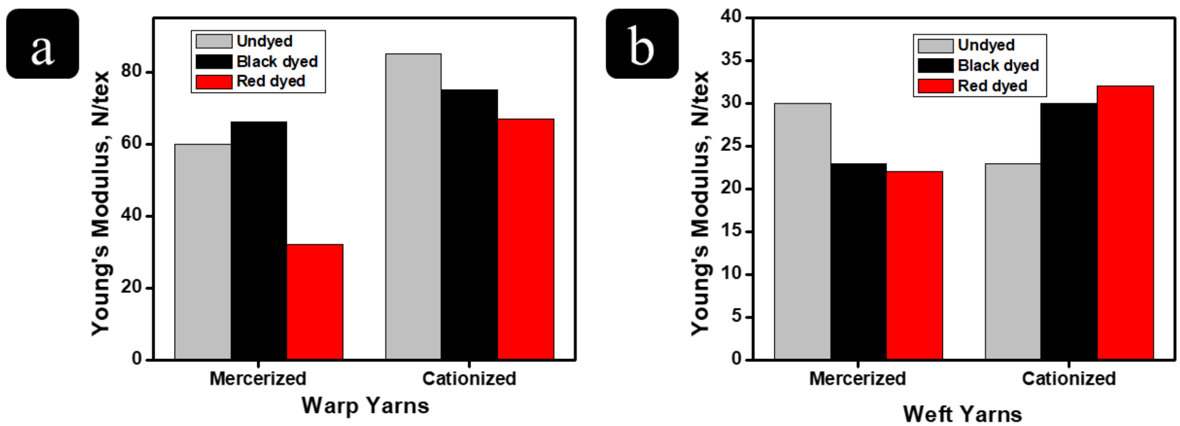

Fig. 3 Mechanical strength of mercerized and cat-ionized warp and weft yarns

strength warp side and weft side, and it is revealed from fig.4 (a, b) that, after dyeing cationized fabric has more tensile strength. It is because cationization occurs mainly at methyl hydroxyl groups of cotton, and does not break the intermolecular H-bonds, thus does not affect the tensile properties.

3.3.2.2 Absorbency

Absorbency of both fabrics was test by drop test and capillary rise test before dyeing and after dyeing, as per Table. 2, the results of both tests it shows big difference between mercerized and cationized fabrics, cationized fabric uses less time to absorb water and has high capillary raise after dyeing of both fabrics. Cationized fabric has more absorbency which helps cationization to be superior on mercerization.

3.3.2.3 Rubbing fastness

Table. 2 shows rubbing fastness (dry & wet) rating for mercerized dyed & cationized dyed fabric. And it shows almost same fastness properties between mercerized dyed and cat-ionized dyed fabric, it may be because of the formation of strong ionic bond between fiber & dye. The dried samples showed excellent rubbing fastness in mercerized dyed & cationized dyed.

3.3.2.4 Resistance to pilling

In Table. 2 cationized sample shows less pilling propensity or has higher resistance to pilling than mercerized sample.

Table. 2 Comparison of characteristics of mercerized and cationized samples.

S.NO	Sample	Dyeing	Drop test time (Sec)	Capillary rise (cm)	Rubbing fastness (Dry)	Rubbing fastness (Wet)	Pilling rating
1	Mercerized	Undyed	6	1	---	----	3
2	Cationized		3	3.5	---	----	4
3	Mercerized	Red KB4L	4	4.5	5	3/4	3
4	Cationized		1.2	6	5	3	4
5	Mercerized	Black 5	5	4	5	3/4	3
6	Cationized		1.5	4	5	3	4

4. Conclusion

Mercerization is expensive process in all pretreatment process, it needs specific machine for their application and consume high amount of caustic soda and need to be neutralize after treatment and dyeing of mercerized fabric needs salt for the exhaustion of dyes and, dye fixation percentage of mercerized fabric is not very high, and during washing of dyed fabric color bleed occurs and because of these issues toxic effluent is generated from the dyeing department which have high B.O.D, C.O.D & TDS value, In case of developing countries, where scare in natural resources such as water, thermal energy, and high capital investment/ processing cost in effluent treatment cationization can solve the issues. In this work cationization of cotton is done by a cationic reagent CHPTAC, and then dyed with reactive dyes, Red KB4L and Black 5, and in the results cationization shows huge potential over mercerization for dyeing characteristics. Color strength of mercerized fabric and cationized fabric after washing is almost same, and cationized fabric has high dye fixation percentage, tensile strength of cationized fabric is more than mercerized fabric before and after dyeing, rubbing fastness of both fabrics are almost same, cationized fabric has higher resistance to pilling ,reactive dyeing of cationized fabric produces less toxic effluent, and have low C.O.D, B.O.D & TDS value than reactive dyeing of mercerized fabric because reactive dyeing of cationized fabric does not need salt. Results shows that, Mercerization process can be replaced by cationization process with improved characteristics. Cationization is environmental friendly process. In cationization there is no need of salt and specialized machine which is required for mercerization process. In cationization there were chances of poor rubbing fastness because of surface dyeing but results showed excellent rubbing fastness (dry) and good rubbing fastness (wet).

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Dedication

All this work is dedicated to my sister N K Teacher, and my parents, whose support and prayers are always with me