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Pre-cationization of cotton fabrics: An effective alternative tool for activation of hydrogen peroxide bleaching process

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ABSTRACT

Hydrogen peroxide is a well-known environmentally safe bleaching agent for cotton fabric. However, bleaching of cotton based fabric with hydrogen peroxide requires alkaline medium (normally NaOH), stabilizer and either high temperatures or long dwell times. After bleaching and before dyeing, large amount of water is required for washing the residual un-decomposed hydrogen peroxide and the residual alkali. In this work, a new approach for bleaching cotton based fabric is postulated and investigated. The cotton fabric was scoured and cationized with NaOH and 3-cholor-2-hydroxy propyltrimethyl ammonium chloride (commercially known as CR-2000) either concurrent in one step process or separately in two step process. The scoured and cationized cotton fabric was then preceded for hydrogen peroxide bleaching. The cationic group on the cationized cotton fabric serves a dual function in the bleaching bath; the first is built-in catalyst for bleaching process and the second is powerful alkali site necessary for activation of hydrogen peroxide bleaching bath instead of NaOH. Three bleaching technique are utilized in bleaching of pre-scoured and cationized cotton fabric. These techniques are, exhaustion technique, pad-steam and cold pad-batch. The effects of cationization level, bleaching technique, bleaching parameters were systematically investigated. The fabric was monitored for strength properties, whiteness index and nitrogen content before and after the bleaching process. Results obtained show that, pre-cationization of cotton fabric provide comparable fabric whiteness in all technique investigated at much shorter reaction times and lower bleaching temperature in absence of NaOH. These results call for a breakthrough not only in bleaching of cotton based textiles but also in conventional detergent washing formulation.

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1. Introduction

Industrial processes in everyday life must become environmentally safer, cost-effective and must conserve energy. This is a major goal of today's life science research. Current industrial trends place an emphasis on the manufacture of diverse product ranges with high added value at relatively small lots. Consequently, increasing concern has been expressed over how industry can adapt to this trend and still achieve energy saving. For these purposes, several approaches have been postulated in the last two decades for pretreatment and bleaching of cotton fabric. One is to combine pretreatment (or preparation) processes to save energy and chemicals. Another recent aspect is the use of bleach catalyst (BoÈsinga et al., 1999; Dannacher, 2006; Suss, 2003).

Cotton cellulose has excellent properties such as higher water absorbency and moisture, being comfortable to wear and easily to dye. For these reasons, the apparel industry is predominantly cotton based, and the share of cotton in total fibre consumption is about 50% (Karmakar, 1999).

Cotton is composed almost entirely of cellulose (90–96% based on weight of fibres). The impurities in cotton fibre range from 4% to 10%. The overall composition of raw cotton fibres depends on its type, origin, fibres maturity, weathering and agricultural conditions (Brushwood, 2003; Carr, 1995; Karmakar, 1999; Segal & Wakelyn, 1988). The impurities include protein (1.0–2.1%), wax (0.4–1.7%), ash (inorganic salts) (0.7–1.8%), pectin (0.4–1.9%) and others (resins, pigments, hemi-cellulose) (1.5–2.5%) (Brushwood, 2003; Karmakar, 1999; Segal & Wakelyn, 1988). The yellowish or brown coloration of the cotton fibre is related to the protoplasmic residues of protein and the flavones pigments of cotton flowers (Karmakar, 1999; Segal & Wakelyn, 1988).

With the exception of natural coloring matters that may be removed by bleaching using certain oxidants, many other impurities are removed by alkali treatment in scouring stage. The latter in common practice involves boiling the cotton in sodium hydroxide (2–5%) for 1 h (Lewin & Sello, 1984; Segal & Wakelyn, 1988).

Hydrogen peroxide is a well-known environmentally safe bleaching agent for cotton fabrics. However, bleaching of cotton based fabrics with hydrogen peroxide requires alkaline medium

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(normally NaOH), stabilizer and either high temperatures or long dwell times. After bleaching and before dyeing, large amount of water is required for washing the residual un-decomposed hydrogen peroxide and the residual alkali (Brooks & Moore, 2000; Dannacher & Schlenker, 1996; Lewin & Sello, 1984; Schulz, 1990; Spiro & Griffith, 1997; Zeronian & Inglesby, 1995).

It is thus desirable to use bleaching systems that operate well at lower temperatures, shorter reaction times and/or using lower chemical charges than current, without causing unacceptable damage to textile fibers. This, indeed, stimulates the present work which is undertaken with a view to development a new approach for bleaching cotton based fabrics with hydrogen peroxide.

Desirable properties are imparted to cellulosic textile fabrics when treated with 3-chloro-2-hydroxypropyl trimethyl ammonium chloride, render them cationic in nature (Hashem, 2006; Hauser, 2000; Hauser & Tabba, 2001, 2002). The quaternary ammonium group-+N(CH₃)₃ has a very high positive charge and can thereby lead to the formation of ionic bonds (salt linkages) with negatively charged anionic groups, such as those found in wide array of anionic dye classes or carboxyl containing compounds. The former impart no-salt dyeing properties to cotton fabric (Hashem, 2006; Hauser, 2000; Hauser & Tabba, 2001, 2002) whereas, the latter impart ionic crosslinking for cotton fabric renders it wrinkle recovery (Hashem, Hauser, & Smith, 2003a; Hashem, Smith, & Hauser, 2003b). Moreover, the presence of cationized groups in the cellulose imparts also antimicrobial properties to cotton fabric (Seong & KO, 1998).

In this work, a new approach for bleaching of cotton based fabric is postulated and investigated. Accordingly, cotton fabric was scoured and cationized with NaOH along with 3-cholor-2-hydroxy propyltrimethyl ammonium chloride (CR-2000) either concurrent in one step process or separately in two step processes then proceed for hydrogen peroxide bleaching. The cationic groups on the cationized cotton fabric serves a dual function in the bleaching bath; the first is *built-in* catalyst for bleaching process and the second is powerful alkali site necessary for activation of hydrogen peroxide bleaching bath instead of NaOH

The effects of cationization level, bleaching technique, bleaching parameters were systematically investigated. The fabric was monitored for strength properties, whiteness index and nitrogen content before and after the bleaching process.

2. Experimental

2.1. Cotton fabric and chemicals

Greige 100% woven cotton fabric was supplied by Misr Company for Spinning and Weaving, Mehala El-Kura, Egypt. The fabric has the following specification: plain weave, warp 36 yarn/cm, weft 30 yarn/cm, fabric weight, 150 g/m². Chemical analysis carried out on the greige fabric showed that the fabric warps were sized with starch-based sizing agent.

Sodium hydroxide, sodium carbonate, sodium chloride, ammonium persulphate, acetic acid, hydrochloric acid, were of laboratory grade chemicals. 3-Chloro-2-hydroxypropyl trimethyl ammonium chloride (CHTAC) (69%) of technical grade chemicals was kindly supplied under the commercial name CR-2000 by Dow Chemical Company, USA. Egyptol® (non-ionic wetting agent based on ethylene oxide condensate) and Espycon® (anionic wetting) agent were supplied from Starch and Yeast Co., Alexandria, Egypt. Hydrogen peroxide (50 wt.%), sodium silicate solution 48° Be', and amylase enzyme were of technical grade chemicals.

2.2. Desizing

Greige cotton fabrics (100 g) were desized by padding the fabric in solution containing diastase enzyme (2 g/l), sodium chloride (1 g/l), acetic acid (1 g/l), Egyptol® (4 g/l) and Espycon® (2 g/l). The sample was then squeezed to a wet pick up of 100% and stored at room temperature for 8 h. The sample was washed several times with hot water then with cold water and dried at ambient conditions.

2.3. Scouring

Scouring of the desized cotton fabrics were carried out using impregnation technique, the experimental technique was adapted as follows: 100 g of desized cotton fabric were treated with an aqueous solution containing NaOH (6 g/l), Egyptol® (2 g/l) and Espycon® (1 g/l) using material to liquor ratio (LR) 1:30 at 95 °C for 30 min. The samples were washed several times with boiling water then washed with cold water and finally dried at ambient conditions.

2.4. Cationization of scoured cotton fabric

Scoured cotton fabrics were cationized using the cold pad-batch technique according to previous reported method (Hauser, 2000; Hauser & Tabba, 2001, 2002). Experimental procedure adopted as follows, 100 g/l, CR-2000 was mixed with 40 g/l, sodium hydroxide then the solution was completed to 1 L. Scoured cotton fabric was padded through this mix and squeezed to wet pick up of 100%, then batched in a plastic bag at room temperature overnight. The fabrics were washed with cold water and 1% acetic acid, then washed several times with cold water and finally dried at ambient conditions.

2.5. One step process for scouring and cationization

One step process for scouring and cationization of cotton fabric was carried out as described elsewhere (Hashem, 2006). The experimental technique was adopted as follows:

Desized cotton fabric was padded in an aqueous solution containing $(50-200\,\mathrm{g/l})$ CR-2000 and $(25-75\,\mathrm{g/l})$ NaOH. The fabric was then squeezed to a wet pick up of 100%, and then batched at room temperature overnight in a plastic bag. At the end, the fabrics were washed with boiling water then acidified with 1% w/w aqueous acetic acid. Finally, washed several times with cold water and dried under ambient conditions.

2.6. Bleaching

Three techniques were investigated to bleach scoured and cationized cotton fabric, namely, exhaustion, pad-steam and cold pad-batch. In the exhaustion method, scoured cotton fabrics were treated with an aqueous solution containing H_2O_2 , (6 g/l), sodium silicate, (0–2 g/l), NaOH, (0–2 g/l). A material to liquor ratio of 1:30 was used and the bleaching process was carried out at (30–90 °C) for (15–90 min). The fabric was then washed several times with boiling water, then with cold water and finally dried at ambient conditions.

In pad-steam method, the fabric samples were padded in a solution containing H_2O_2 (15 g/l), sodium silicate (0–8 g/l) NaOH, (0–2 g/l). The fabric was then squeezed to a wet pick up of 100% and stored in a sealed stainless steel cup at 100 °C for 45 min. After bleaching, the sample was washed several times with hot water, then with cold water and finally dried at ambient conditions.

In cold pad-batch method, the sample was treated similar to that obeyed in pad-steam method except that the fabric was batched overnight at room temperature in polyethylene bag.

Scoured uncationized cotton fabrics were bleached similarly with each technique for comparison.

(9)

2.7. Testing and analysis

 Nitrogen content of the cationized samples were determined by the Kjeldhal method (Vogel, 1975). Fixation percent (F%) was calculated as per the following equation:

$$F\% = \frac{\text{amount of nitrogen fixed (detected)}}{\text{total amount of nitrogen of CHTAC applied}} \times 100$$

- Tensile strength and elongation at break was determined according to ASTM standard test method (ASTM, 1994).
- The degree of whiteness of the fabric sample expressed as whiteness index was measured according to reported method (Rowe, 1978).
- Wettability was assessed in terms of drop disappearance, measured by allowing a drop of water to fall on the sample and recording the time required for drop disappearance (AATCC, Standard Test Method).

3. Result and discussion

3.1. Chemical aspects of peroxide bleaching and mechanism of peroxide bleaching

For bleaching to occur, the stability of hydrogen peroxide must be overcome by activation. The usual activator for hydrogen peroxide in textile bleaching is sodium hydroxide. Hydrogen peroxide liberates perhydroxyl anion (HOO⁻) in aqueous medium which chemically behaves like a weak acid Eq. (1) (Brooks & Moore, 2000; Dannacher & Schlenker, 1996; Schulz, 1990; Zeronian & Inglesby, 1995). The perhydroxyl anion is highly unstable and reacts as nucleophile with oxidizable substance (colored impurities in cotton) by a displacement reaction or by addition to a double bond. The pigments responsible for the natural color of cotton contain a chromophoric system of conjugated double bonds. This will attack by a free-radical system or perhydroxyl anion as indicated by Eqs. (6) and (7), and thus bleaching action takes place (Brooks & Moore, 2000; Dannacher & Schlenker, 1996; Schulz, 1990; Zeronian & Inglesby, 1995).

Sodium hydroxide activates hydrogen peroxide because H⁺ ion is neutralized by alkali which is favourable for liberation of HO₂⁻ (Eq. (1)). However, at higher pH (above pH 11) the liberation of HOO⁻ anion is so rapid that it becomes unstable with the formation of oxygen gas which has no bleaching property. If the rate of decomposition is very high, the unutilized HOO⁻ anion may damage the fibre. A safe and optimum pH for cotton bleaching lies between 10.3 and 10.8 where the rate of evolution of perhydroxyl ion is equal to the rate of consumption (for bleaching). At higher pH, hydrogen peroxide is not stable and hence a stabilizer is frequently added in the bleaching bath (Zeronian & Inglesby, 1995).

$$H_2O_2 \leftrightarrow H^{\oplus} + 00H^{-} \xrightarrow{OH^{-}} H_2O + 00H^{-}$$
 (1)

$$H_2O_2 + M^{+2} \to M^{+3} + OH^- + OH$$
 (2)

(or)
$$H_2O_2 + OOH^- \rightarrow \cdot OOH + OH^- + \cdot OH$$
 (3)

$$H_2O_2 + \cdot OH \rightarrow \cdot OOH + H_2O \tag{4}$$

$$: 00H + M^{+3} \to M^{+2} + H^{\oplus} + O_2$$
 (5)

Dannacher and Schlenker (1996) rejected the role of the perhydroxyl anion in bleaching because of a belief that there is an optimum pH beyond which bleaching decreases. This pH dependence of $\rm H_2O_2$ bleaching suggests that the $\rm HOO^-$ anion is not the active bleaching agent. They purposed instead that the active oxygen is the superoxide radical $\rm ^{\circ}O^{-2}$, formed in an alkaline medium from the perhydroxyl radical.

However, it has been proved recently that, there is no any oxidizing species other than HOO^- and H_2O_2 . The perhydroxyl anion formation is the key in textile bleaching and that the degree of whiteness obtained is directly related to the concentration of perhydroxyl anion in the bleaching solutions. There are two competing reactions of H_2O_2 in the bleaching bath: first, there is the reaction with colored bodies on the fabric to give bleached fabric and second, decomposition to form oxygen (Spiro & Griffith, 1997).

In this work, we postulated the cationic groups on the cationized cotton fabric can serves a dual function in the bleaching bath; the first is *built-in* catalyst for bleaching process and the second is powerful alkali site necessary for activation of hydrogen peroxide bleaching bath instead of NaOH as explained by Eqs. (8)–(10). The effects of cationization level, bleaching technique and bleaching parameters were systematically investigated. The fabric was monitored for strength properties, whiteness index, and nitrogen content before and after the bleaching process.

3.2. Exhaust bleaching of cationized cotton fabric

3.2.1. Effect of NaOH concentration

Scoured and cationized cotton fabrics were bleached with $\rm H_2O_2$ in presence of different concentration from NaOH. The bleached fabrics were monitored for W.I, T.S and elongation at break as well as nitrogen content before and after bleaching. Results obtained are set out in Table 1.

Results of Table 1 which are self explanatory, depict that, increasing NaOH concentration from 0 g/l to 2 g/l is accompanied by decreasing in W.I of the bleached cotton fabric from 73.5 to 68.79. The results show also that, the retained tensile strength after bleaching are marginally decreased as the concentration of NaOH increased whereas the results of elongation at break show no spe-

Table 1Effect of NaOH concentration on the properties of cationized cotton fabric after bleaching with hydrogen peroxide using exhaustion technique.

NaOH conc. (g/l)	W.I	R.T (%)	Elongation (%)	N%	
				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
0.0	73.50	83	19	0.23	0.22
0.5	73.10	83	19	0.23	0.23
1.0	71.02	83	20	0.23	0.21
1.5	70.20	82	20	0.23	0.21
2.0	68.79	82	19	0.23	0.22

Blank mean scoured and cationized sample before bleaching.

R.T = Retained tensile strength.

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 6 g/l; sodium silicate, 2 g/l; LR, 1:30; temperature, 90 °C; time, 45 min

cific trend. It is further observed that, the nitrogen content of cationized cotton fabric remain practically unaltered after bleaching. A point which indicates that, the pre-scoured and cationized cotton fabric pertain its cationic nature after peroxide bleaching. A silent feature observed in result of Table 1 is, the higher W.I and retained tensile strength observed at zero NaOH concentration. Currently, bleaching of cotton based fabric with hydrogen peroxide requires alkaline medium (normally NaOH) as indicated by Eq. (1). But in our case (bleaching of pre-scoured and cationized cotton fabric) addition of NaOH decreases the extent of the bleaching. This feature is due to that the quaternary ammonium chloride group (cationic groups) is converted to quaternary ammonium hydroxide after its reaction with cotton hydroxyls and therefore, offers the necessary alkaline conditions for the bleaching reaction (see Eqs. (8)–(10)). Stated in other words, the cationic groups in the pre-scoured and cationized cotton fabric form an alkali site that might stimulate in situ the peroxide bleaching process. At the same time the alkaline cationic sites furnish a safe and optimum pH for cotton peroxide bleaching where the rate of evolution of perhydroxyl anion is equal to the rate of consumption for bleaching.

Although, alkali medium activates hydrogen peroxide because H^{\star} ion is neutralized by alkali which is favourable for liberation of $\mathrm{HO_2}^-$ (Eq. (1)). However, at higher pH (above pH 11) the liberation of HOO^- anion is so rapid that it becomes unstable with the formation of oxygen gas which has no bleaching property. If the rate of decomposition is very high, the unutilized HOO^- anion react with themselves and produces unbleachable species which leading to damage the fibre and lower W.I.

3.2.2. Effect of sodium silicate concentration

At higher pH, hydrogen peroxide is not stable and hence a stabilizer is frequently added in the bleaching bath (Brooks & Moore, 2000; Dannacher & Schlenker, 1996; Schulz, 1990; Spiro & Griffith, 1997; Zeronian & Inglesby, 1995).

The process of regulation or control of perhydroxyl ion to prevent rapid decomposition of peroxide bleach and to minimize fibre degradation is described as stabilization. The stabilizer control and regulate the bleaching by a multiplicity of functions. They control the formation of free radicals. They also act as buffers, sequestrants and dispersants. The sequestering action inactivates metallic impurities which cause catalytic decomposition of hydrogen peroxide or precipitation of hydroxides or carbonates (Brooks & Moore, 2000; Dannacher & Schlenker, 1996; Schulz, 1990; Spiro & Griffith, 1997; Zeronian & Inglesby, 1995). The stabilizers first used in bleaching of cellulosic fibres with peroxide were the sodium polysilicates, often called water glass or simply sodium silicate which is highly effective and low cost. Sodium silicate is

Table 2Effect of sodium silicate concentration on the properties of cationized cotton fabric after bleaching with hydrogen peroxide using exhaustion technique.

Sod. silicate conc. (g/l)	W.I	R.T (%)	Elongation (%)	N%	
conc. (g/1)				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
0.0	68.50	86	18	0.23	0.22
0.5	71.10	85	17	0.23	0.23
1.0	74.30	83	19	0.23	0.21
1.5	74.10	83	19	0.23	0.21
2.0	73.50	83	19	0.23	0.22

Blank mean scoured and cationized sample before bleaching.

R.T = Retained tensile strength

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 6 g/l; LR, 1:30; temperature, 90 °C; time, 45 min.

mildly alkaline in nature and the commercial grade has a Na₂O:-SiO₂ ratio equal 1:3.3 (Nettles, 1983).

Table 2 shows the effect of sodium silicate concentration in the peroxide bleaching bath on the properties of the pre-scoured and cationized cotton fabric after bleaching. It is seen from Table 2 that, increasing sodium silicate concentration from zero to 1 g/l is accompanied by increased W.I of the bleached fabric. Further increase in sodium silicate concentration is accompanied by decreased in W.I. It is further noted that, the retained tensile strength and the nitrogen content after bleaching remain almost constant upon increasing sodium silicate concentration. Obviously, 1 g/l sodium silicate represents the optimum concentration when the bleaching process is carried out using the exhaustion technique.

3.2.3. Effect of bleaching temperature

Table 3 shows the effect of bleaching temperature on the major properties of pre-cationized and scoured cotton fabric when the bleaching process was carried out using the exhaustion technique. It is seen from Table 3 that raising the bleaching temperature from 30 °C to 50 °C is accompanied by increasing in W.I whereas both retained tensile strength and elongation at break gradually decreased. Further increasing in beaching temperature kept the W.I practically intact whereas both retained tensile strength and elongation at break gradually decreased. A salient feature observed in Table 3 is the higher W.I at lower bleaching temperature (50 °C). Indeed this ability to bleach the pre-scoured and cationized cotton fabric with hydrogen peroxide in such short times at low temperature form a new route for low temperature bleaching of cotton fabrics.

3.2.4. Duration of the exhaust bleaching

Table 4 shows the effect of bleaching time on the major properties of pre-scoured and cationized cotton fabric when the bleaching

Table 3Effect of bleaching temperature on the properties of cationized cotton fabric after bleaching with exhaustion technique.

Temp. (°C)	W.I	R.T (%)	Elongation (%)	N%	
				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
30	74.50	87	21	0.23	0.22
50	74.50	87	21	0.23	0.23
60	74.30	86	21	0.23	0.21
80	74.30	83	19	0.23	0.21
90	74.30	83	19	0.23	0.22

Blank mean scoured and cationized sample before bleaching.

R.T = Retained tensile strength.

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 6 g/l; sodium silicate, 1 g/l; LR, 1:30; time, 45 min.

Table 4 Duration of the exhaust bleaching.

Time (min)	W.I	R.T (%)	Elongation (%)	N%	
				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
15	55.70	87	22	0.23	0.22
30	69.50	87	21	0.23	0.23
45	74.50	87	21	0.23	0.21
60	77.80	83	19	0.23	0.21
75	77.90	82	19	0.23	0.22
90	78.10	82	18	0.23	0.21

Blank mean scoured and cationized sample before bleaching.

R.T = Retained tensile strength.

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 6 g/l; sodium silicate, 1 g/l; LR, 1:30; temperature, 50 °C.

process was carried out using the exhaustion technique. The results indicated that, 60 min is enough to give the pre-scoured and cationized a maximum W.I. Prolonging the bleaching time exert practically no influence on the fabric W.I. Longer time provides better opportunity for better contact among reactants and, therefore, higher extent of bleaching.

It could be emphasized from result presented in Tables 1–4 that pre-scoured and cationized cotton fabric can be full bleached with exhaustion technique using a bleaching formulation containing 1 g/l sodium silicate and 6 g/l $\rm H_2O_2$ at 50 °C for 60 min and there is no need to add NaOH or any other alkali medium in the peroxide bleaching recipes. These results call for a breakthrough not only in bleaching of cotton based textiles but also in conventional detergent washing formulation.

3.3. Bleaching of cationized cotton fabric using pad-steam technique

3.3.1. Effect of process parameters

Similar study was carried out to bleach pre-scoured and cationized cotton fabric on using pad-steam technique instead of exhaustion technique. In pad-steam technique, three parameters play an important rule in determining the efficiency of bleaching processes. These parameters are, NaOH and sodium silicate concentrations and steaming time. The effects of these parameters are systematically investigated and the results are set out in Tables 5–7, respectively, and elucidated as follows.

Table 5 shows the effect of NaOH concentration in peroxide bleaching bath on the properties of pre-scoured and cationized cotton fabric after bleaching with pad-steam technique. Results of Table 5 are similar to those obtained previously with exhaustion technique and could be explained on similar basis. Here too, higher W.I is obtained at zero NaOH concentration, this indicate also that

Table 5Effect of NaOH concentration in peroxide bleaching bath on the properties of cationized cotton fabric after bleaching with pad-steam technique.

NaOH conc. (g/l)	W.I	R.T (%)	Elongation (%)	N%	
				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
0.0	77.50	81	17	0.23	0.22
0.5	75.30	81	17	0.23	0.23
1.0	73.40	80	16	0.23	0.21
1.5	73.27	80	16	0.23	0.21
2.0	68.79	79	16	0.23	0.22

Blank mean scoured and cationized sample before bleaching.

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 15 g/l; sodium silicate, 2 g/l; wet pick up, 100%; steaming time, 45 min

Table 6Effect of sodium silicate concentration on the properties of cationized cotton fabric after bleaching with hydrogen peroxide using pad-steam technique.

Sod. silicate conc. (g/l)	W.I	R.T (%)	Elongation (%)	N%	
conc. (g/1)				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
0.0	69.60	78	20	0.23	0.22
1.0	71.30	80	17	0.23	0.23
2.0	77.50	81	17	0.23	0.22
4.0	79.20	81	18	0.23	0.21
6.0	81.30	82	17	0.23	0.22
8.0	81.50	81	16	0.23	0.22

Blank mean scoured and cationized sample before bleaching.

R.T = Retained tensile strength.

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 15 g/l; wet pick up, 100%; steaming time, 45 min.

Table 7Effect of steaming time on the properties of cationized cotton fabric after bleaching with hydrogen peroxide using pad-steam technique.

Steaming time (min)	W.I	R.T (%)	Elongation (%)	N%	
(11111)				Before bleaching	After bleaching
Blank	15.60	100	22	0.23	0.22
15	65.40	85	19	0.23	0.22
30	75.60	83	17	0.23	0.23
45	81.30	82	17	0.23	0.22
60	81.40	80	15	0.23	0.21

Blank mean scoured and cationized sample before bleaching.

R.T = Retained tensile strength.

Bleaching conditions used: scoured and cationized cotton fabric contains 0.23% nitrogen; H_2O_2 , 15 g/l; sodium silicate, 6 g/l; wet pick up, 100%.

the *built-in* cationic groups in the pre-scoured and cationized cotton fabric furnish a sufficient alkalinity that required for H_2O_2 activation. It is further noted that, the nitrogen content remain practically intact after bleaching whatever the concentration of NaOH.

Table 6 shows the effect sodium silicate concentration on the major properties of pre-scoured and cationized cotton fabric after being bleached using pad-steam technique. Results obtain are similar to those obtained previously with exhaustion technique (Table 2) and cold be explained on similar way. The most important feature observed in Table 6 is higher concentration of sodium silicate (6 g/l) required to achieve the higher fabric whiteness (i.e., higher stabilization to perhydroxyl anion) without detracting from the other fabric properties after bleaching using pad-steam technique.

Table 7 summarizes the effect of steaming time on the properties of pre-scoured and cationized cotton fabric after being bleaching with $\rm H_2O_2$ using pad-steam technique. It seen from Table 7 that, increasing the bleaching time from 15 min to 45 min is accompanied by gradual increased in the fabric whiteness, whereas values of retained tensile strength and elongation at break slightly decreased from 85% and 19% to 82% and 17% respectively. Further prolonging time has practically no effect on the whiteness of the fabrics whereas both retained tensile strength and elongation at break decreased. Obviously, 45 min steaming time represent the optimum bleaching time of pre-scoured and cationized cotton fabric with $\rm H_2O_2$ using pad-steam technique.

It could be emphasized from result presented in Tables 5–7 that, pre-scoured and cationized cotton fabric can be full bleached with pad-steam technique using a bleaching formulation containing $15 \, \mathrm{g/l}$, H_2O_2 and $6 \, \mathrm{g/l}$, sodium silicate. The fabric was then

R.T = Retained tensile strength.

squeezed to a wet pick up of 100% and steamed for 45 min. Here also; there is no need to add NaOH or any other alkali medium in the peroxide bleaching recipes.

3.4. Effect of cationization extent

In order to investigate the effect of cationization extent, the desized cotton fabrics were pre-scoured and cationization in one step process using different concentration from CR-2000 and NaOH as explained in the experimental part. After scouring and cationization, the fabrics pertained different amount of cationic groups as indicated by its different nitrogen content. These fabrics were separately subjected to peroxide bleaching using three different techniques, namely, exhaustion, pad-steam and cold pad-batch. The bleached fabrics were monitored for W.I, retained tensile strength and elongation at break. Results obtained are presented in Figs. 1–3. Values obtained with scoured and uncationized (blank) sample are also set out in the same figures for comparison.

Results of Fig. 1 show that: at the same bleaching technique, increasing the nitrogen content (i.e., the cationic sites) is accompanied by increasing in W.I of the bleached fabric. Higher W.I was obtained when the cationized cotton fabric have nitrogen content equal to 0.23%. Increasing the cationization extent beyond this level gradually decreased the W.I of the fabrics. It should be mentioned here that, this nitrogen content can be obtained when the scoured cotton fabric was cationized using 100 g/l, CR-2000 and 40 g/l, NaOH using cold pad-batch technique or the desized cotton fabric was concurrent scoured and cationized using an aqueous solution containing 100 g/l, CR-2000 and 50 g/l, NaOH with the same technique.

It is further noted from Fig. 1 that, uncationized cotton fabric (zero nitrogen content) shows lower values of W.I compared with the cationized one and whatever the cationization level.

Meanwhile, at the same cationization level the W.I of the bleached fabrics depends on the technique used and shows the following order: pad-steam > exhaustion > cold pad-batch.

Fig. 2 shows the dependence of retained tensile strength of the bleached cotton fabric on the cationization extent (expressed as nitrogen content). It is seen from Fig. 2 that, higher retained tensile strength is observed at nitrogen content equal to 0.23%. This was observed irrespective to the bleaching technique employed. Fig. 2 highlights also that, at the same nitrogen content, the retained tensile strength of bleached fabric show the following order: cold padbatch > exhaustion > pad-steam.

Fig. 3 shows the relation between fabric nitrogen content and the elongation at break of scoured and cationized cotton fabric after bleaching with $\rm H_2O_2$. The results indicate that, the elongation at break of bleached cotton fabrics shows no specific trend with its nitrogen contents. This was observed with all bleaching techniques used. However, at the same fabric nitrogen content the elongation at break shows the following order: Exhaustion = cold padbatch > pad-steam.

4. Conclusion

Pre-scoured and cationized cotton fabric can be full bleached with exhaustion technique using a bleaching formulation containing 1 g/l sodium silicate and 6 g/l $\rm H_2O_2$ at 50 °C for 60 min and there is no need to add NaOH or any other alkali medium in the peroxide bleaching recipes. On using pad-steam technique the optimum bleaching formulation involve the utilization of bleaching bath containing 15 g/l $\rm H_2O_2$ and 6 g/l sodium silicate then steamed for 45 min. Here also, there is no need to add NaOH or any other alkali medium in the peroxide bleaching recipes. Higher W.I is obtained when the cationized cotton fabric have nitrogen content equal to 0.23%. Increasing the cationization extent beyond this level gradually decreased the W.I of the fabric. This amount of

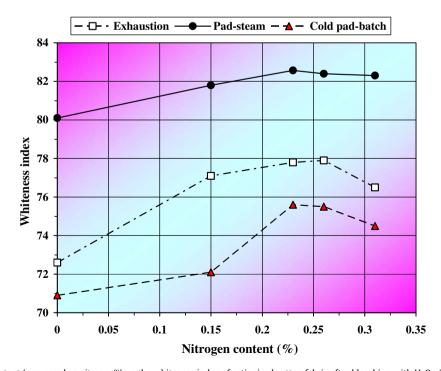


Fig. 1. Effect of cationization extent (expressed as nitrogen%) on the whiteness index of cationized cotton fabric after bleaching with H_2O_2 . Zero nitrogen content represent the values obtained with blank sample, i.e., scoured cotton fabric without cationization. *Conditions used.* Exhaustion: $[H_2O_2]$, 6 g/l; [sodium silicate], 1 g/l; LR, 1:30, temperature, 50 °C, time, 60 min. Pad-steam: $[H_2O_2]$, 15 g/l; [sodium silicate], 6 g/l; time, 45 min. Cold pad-batch: $[H_2O_2]$, 15 g/l; [sodium silicate], 6 g/l; time, 24 h, room temperature.

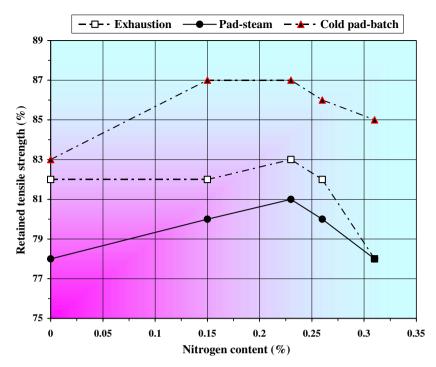


Fig. 2. Effect of cationization extent (expressed as nitrogen%) on the retained tensile strength of cationized cotton fabric after bleaching with H_2O_2 . Zero nitrogen content represent the values obtained with blank sample, i.e., scoured cotton fabric without cationization. *Conditions used*: Exhaustion: $[H_2O_2]$, 6 g/l; [sodium silicate], 1 g/l; LR, 1:30, temperature, 50 °C, time, 60 min. Pad-steam: $[H_2O_2]$, 15 g/l; [sodium silicate], 6 g/l; time, 45 min. Cold pad-batch: $[H_2O_2]$, 15 g/l; [sodium silicate], 6 g/l; time, 24 h, room temperature.

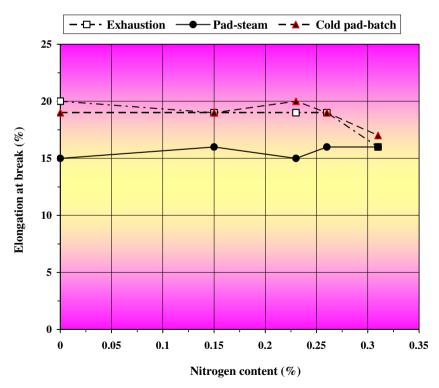


Fig. 3. Effect of cationization extent (expressed as nitrogen%) on the elongation at break of cationized cotton fabric after bleaching with H_2O_2 . Zero nitrogen content represent the values obtained with blank sample, i.e., scoured cotton fabric without cationization. *Conditions used*: Exhaustion: $[H_2O_2]$, 6 g/l; [sodium silicate], 1 g/l; LR, 1:30, temperature, 50 °C, time, 60 min. Pad-steam: $[H_2O_2]$, 15 g/l; [sodium silicate], 6 g/l; time, 45 min. Cold pad-batch: $[H_2O_2]$, 15 g/l; [sodium silicate], 6 g/l; time, 24 h, room temperature.

nitrogen was obtained when scoured cotton fabric was cationized using 100 g/l, CR-2000 and 40 g/l, NaOH using cold pad-batch technique or the desized cotton fabric was concurrent scoured and

cationized using an aqueous solution containing 100 g/l, CR-2000 and 50 g/l, NaOH using the same technique. Uncationized cotton fabric shows lower values of W.I and retained tensile strength

compared with the cationized one and whatever the cationization level when the bleaching was carried out using exhaustion or cold pad-batch technique whereas the results are comparable on using pad-steam technique.

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