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Simple and economic bleaching process for cotton fabric

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ABSTRACT

Cotton fabric was bleached in a simple and economic process using a bleaching system composed of sodium chlorite and hexamethylenetetramine. Different bleaching trials were carried out keeping fixed sodium chlorite concentration and varying other reaction conditions. The obtained results reveal that bleached cotton fabric with satisfactory whiteness index and reasonable tensile strength can be obtained by treating the fabric at 95 °C in a bleaching bath containing 5 g/l sodium chlorite, 0.02 g/l hexamethylenetetramine and 1 g/l non-ionic wetting agent using a material to liquor ratio of 1:30. These optimum conditions lead to completion of the bleaching process in a reasonable duration of 2 h with minimum evolution of harmful chlorine dioxide gas. Lower concentrations of the activator hexamethylenetetramine were found to prolong the bleaching duration without getting satisfactory whiteness index. Higher concentrations of the activator were found to cause fast sodium chlorite decomposition without imparting bleaching effect to the fabric.

1. Introduction

The natural fibers and fabrics even after scouring still contain naturally occurring coloring matter. This yellowish and brown discoloration may be related to flavones pigments present naturally in the fiber. The climate, soil, drought and frost can also cause various degrees of yellowness. Discoloration may also come from dirt, dust, and insects or from harvesting or processing equipment in the form of oils and greases. The objective of bleaching is to produce white fabrics by destroying the coloring matter with the help of bleaching agents with minimum degradation of the fiber. The bleaching agents either oxidize or reduce the coloring matter which is washed out and whiteness thus obtained is of permanent nature. Chemical bleaching of textile fibers is further aided by addition of optical brighteners.

Technology for preparation of cellulosic fiber materials aiming to improve their performance comprises several consecutive operations, i.e. desizing (Ahlawat, Dhiman, Battan, Mandhan, & Sharma, 2009; Bae et al., 2006; Peng et al., 2010), scouring (Abdel-Halim, Fahmy, & Fouda, 2008; Abdel-Halim, Konczewicz, Zimniewska, Al-Deyab, & El-Newehy, 2010; El Shafie, Fouda, & Hashem, 2009) and bleaching (Abdel-Halim, Abdel-Mohdy, Al-Deyab, & El-Newehy, 2010; Abdel-Halim & Al-Deyab, 2011; Hashem, El-Bisi, Sharaf, & Refaie, 2010; Hebeish et al., 2009; Hou, Zhang, & Zhou, 2010; Ibrahim, Sharaf, & Hashem, 2010). The whitening of textiles is achieved with different oxidizing or reducing agents, capable of destroying the natural pigments and matter present in the fibers. Nowadays hydrogen peroxide, due to its biodegradability, almost entirely replaced the conventional chlorine oxidizing chemicals (Spirro & Criffith, 1997). Hydrogen peroxide precursors, such as perborates and percarbonates are incorporated in a major part of commercially available detergent compositions. Alternatively, hydrogen peroxide could be produced enzymatically by glucose oxidase catalyzed conversion of glucose in the presence of oxygen in aqueous solutions (Aly, Moustafa, & Hebeish, 2004).

Fabrics made of cotton are very comfortable to wear and easy to dye because cellulose has excellent properties such as higher water and moisture absorbency. For these reasons, the apparel industry is predominantly cotton based, and the share of cotton in total fiber consumption is about 50%. Cotton is composed almost entirely of cellulose (90–96% based on weight of fibers). The impurities in cotton fiber range from 4% to 10%. The overall composition of raw cotton fibers depends on its type, origin, fibers maturity, weathering and agricultural conditions (Mandal & Venugopalan, 2005; Stathakos, Gemtos, Tsatsarelis, & Galanopoulou, 2006; Wolf & Hadas, 1984). 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, hemicelluloses) (1.5–2.5%) (Gallo & Almirall, 2009; Karmakar, 1999; Plant, Kerby, Zelinski, & Munk, 1998). The yellowish or brown coloration of the cotton fiber is related to the protoplasmic residues of protein and the flavones pigments of cotton flowers (Abidi, Cabrales, & Hequet, 2010; Ghule, Chen, Tzing, Lo, & Ling, 2004). With the exception of

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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 (Chung, Lee, & Choe, 2004; Degani, Gepstein, & Dosoretz, 2004; Tanapongpipat, Khamman, Pruksathorm, & Hunsom, 2008). 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 (normally NaOH), stabilizer and either high temperatures or long treatment times (Abou-Okeil, El-Shafie, & El Zawahry, 2010). After bleaching and before dying, large amount of water is required for washing the residual non decomposed hydrogen peroxide and the residual alkali (Abou-Okeil et al., 2010; Götz, Duschner, White, & Klukowska, 2007). 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.

Sodium chlorite on decomposition produces a strong oxidizing gas known as chlorine dioxide. The lower the pH values and the higher the temperature (above 70 °C) of bleach bath, the greater is the speed of decomposition (Hubbell & Ragauskas, 2010). The rate of chlorine dioxide formation is proportional to the concentration of sodium chlorite in the solution. Chlorous acid ($HClO_2$) that is formed is unstable and decomposes to chlorine dioxide (ClO_2) which is the active species so far as bleaching is concerned. Maximum chlorine dioxide is formed between pH 2.5 and 3.0. In the absence of metal ions and textiles, the rate of formation of chlorine dioxide approximately doubles for each 0.4-pH drop, in the 3.0–5.0 range, at 85 °C. Addition of an acid such as formic, acetic or phosphoric acid is recommended so that pH lies between 3 and 5. If the pH drops below 3.0, cellulosic fiber is severely damaged because weak acid ($HClO_2$) and strong acid (HC1) are formed which hydrolyze the cellulose. On the other hand, the loss of oxygen evolution from hot chlorite solution when cellulosic fiber is added also suggests that chlorite ion rather than chlorous acid is responsible for bleaching (Tamura, Hirota, Saito, & Isogai, 2010).

$$2NaClO_2 + H_2SO_4 \rightarrow Na_2SO_4 + 2HClO_2$$

$$HClO_2 \rightarrow ClO_2 + H^+$$

$$HClO_2 \rightarrow HCl + 2O$$

Aqueous sodium chlorite solutions, which are stable under alkaline conditions, are to be activated by acidification. Activation with strong acid produces toxic corrosive chlorine dioxide gas. It is thus necessary to control its rate of evolution. Technical developments have been linked mainly to control the chlorine dioxide evolution (Hirota, Tamura, Saito, & Isogai, 2009). In practice this is generally achieved by controlling the temperature and pH by addition of buffers in the bleach bath. This can be avoided by addition of weak acid (which forms a buffer with the alkali in the chlorite) or a mixture of weak acid and its salt with a strong base. Some auxiliary products function by giving a slow development of acidity on heating or long storage at ambient temperatures. Activation with persulphates above pH 7 and aldehyde at pH 3–7 (López et al., 2001) have been proposed and also activation by organic acid esters such as ethyl lactate (1–3 g/l) with addition of sodium nitrate (1–3 g/l) to inhibit corrosion (Bäck & Singh, 2004) is possible. Sodium hydrogen orthophosphate (NaH_2PO_4) and sodium formate which produce acid when the liquor is heated are also suggested as buffers. Organic esters such as diethyl tartarate which hydrolyses to give tartaric acid, or sodium chloroacetate which liberates hydrochloric acid and glycolic acid during steaming, have also been suggested as buffers. Other alternatives are the use of either special auxiliary products which limit chlorine dioxide evolution, for example, foam formation on the bleach liquor surface or other chemicals capable of trapping chlorine dioxide chemically.

The present work aims at finding out the most appropriate conditions to bleach cotton in one step process keeping in mind the quality of the bleached fabric, the cost of the bleaching process and its environmental impact.

2. Experimental

2.1. Materials

Plain weave 100% cotton fabric was supplied by Misr Company for Spinning and Weaving, Mehalla El-Kobra, Egypt. The fabric specifications were as follow: fabric weight, 150 g/m^2, weft 30 yarn/cm, warp 36 yarn/cm. The fabric warps were tested for sizing agent and the test showed starch-based sizing agent.

2.2. Chemicals

Sodium chlorite, sodium hydroxide, sodium carbonate, sodium bicarbonate, sodium thiosulphate, hexamethylenetetramine (HMTA), potassium iodide, potassium permanganate and sulphuric acid were of laboratory reagent grade. Non-ionic wetting agent, Nonidet LE was supplied by Shell Textile Chemicals, Cairo, Egypt

2.3. Bleaching

Cotton samples (5 g each) were impregnated in a solution containing $NaClO_2$ (5 g/l), HMTA (0–0.5 g/l), non-ionic wetting agent (1 g/l), using a material to liquor ratio of 1:30. The bleaching was carried out at different temperatures (30–95 °C). The bleaching reaction was monitored by following the medium pH and percent decomposed $NaClO_2$ at different reaction time intervals (Vogel, 1961). After bleaching, the samples were removed from the bleaching liquor, washed thoroughly with hot and then cold water, and finally air-dried.

2.4. Testing and analysis

2.4.1. Weight loss

Fabric weight loss was calculated on the basis of dry weight using the following equation:

$$\text{Weight lost}\,(\%) = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 and W_2 are the dry weights of the fiber before and after bleaching treatment, respectively.

2.4.2. Carboxyl and carbonyl contents

Carbonyl content and carboxyl content of bleached cotton fabric were measured according to the following reported methods, respectively (Mattisson & Legendre, 1952; Nelson & Tripp, 1953).

2.4.3. Degree of whiteness

The degree of whiteness of bleached cotton samples, expressed as whiteness index was measured using a Hunterlab Reflectometer (Model D25/L-2). The WI was calculated in terms of CIE Y (green) and (blue) reflectance components using the equation (ASTM Method E31373).

$$WI = \frac{4Z}{1.18} - 3Y$$

where Y and Z are the readings of the device.

2.4.4. *Tensile strength*

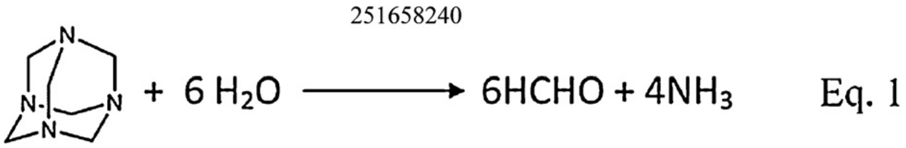
The tensile strength of cotton fabrics was measured according to the ASTM strip test (ASTM Test Method).

3. Results and discussion

3.1. *Mechanism*

It is well established that acidified $NaClO_2$ librates chlorine dioxide rather than chlorine and the rate of chlorine dioxide libration determines the efficiency of the bleaching bath.

Hexamethylenetetramine is a heterocyclic organic compound with the formula $(CH_2)_6N_4$. This white crystalline compound is highly soluble in water and polar organic solvents and has a cage-like structure. Activation of $NaClO_2$ by hexamethylenetetramine may be explained as follows: Hexamethylenetetramine decomposes under the bleaching conditions to give six molecules of formaldehyde and four molecules of ammonia (Eq. (1)).



(1)

The liberation of HCHO accelerates the decomposition of $NaClO_2$ according to the redox reaction suggested by Eq. (2) to form formic acid and sodium hypochlorite.

$$NaClO_2 + HCHO \rightarrow NaClO + HCOOH \tag{2}$$

Once the hypochlorite is formed it is readily hydrolyzed to hypochlorous acid, which is thermodynamically unstable and may be decomposed to its components, HCl and oxygen (Eq. (3)). The hypochlorite ion can also decompose to librate oxygen (Eq. (4)) or react with HOCl to form active free radical species (Eqs. (5) and (6)):

$$2HOCl \leftrightarrow 2HCl + 2[O] \tag{3}$$

$$2ClO^- \rightarrow 2Cl^- + O_2 \tag{4}$$

$$ClO^- + HOCl \rightarrow ClO_2 + Cl^- + HO \tag{5}$$

$$ClO^- + ClO + HO^- \rightarrow 2Cl^- + HO + O_2 \tag{6}$$

In the reaction scheme suggested by Eqs. (5) and (6), the formation of the highly reactive hydroxyl radical can react further with hypochlorite and/or chlorite ions, thereby, perpetuating a chain reaction (Eqs. (7) and (8)).

$$ClO^- + HO \rightarrow ClO + HO^- \tag{7}$$

$$ClO_2{}^- + HO \rightarrow ClO_2 + HO^- \tag{8}$$

The librated NH_3 molecules (Eq. (1)) may render the bleaching medium alkaline, in particular, at the initial stage of the treatment. On the other hand, as the bleaching reaction proceeds, the medium changes to acidic due to the formation of formic acid (Eq. (2)).

3.2. *Effect of HMTA concentration on $NaClO_2$ decomposition*

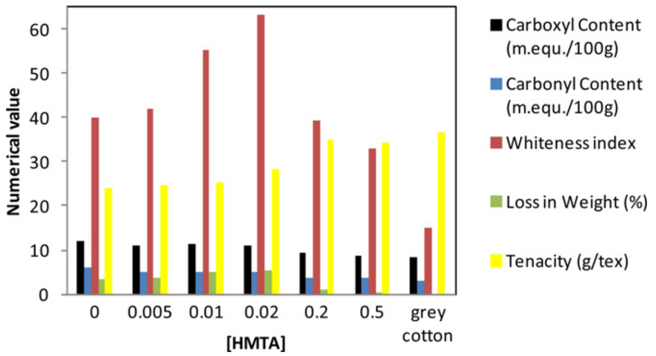


Fig. 1. Effect of [HMTA] on physicochemical properties of bleached cotton fabrics.

Table 1 demonstrates the effect of HMTA concentration on the percent of $NaClO_2$ decomposition during bleaching cotton fabric at 95 °C. The obtained results indicate the following facts: (i) the higher the concentration of HMTA incorporated to the bleaching medium, the higher is the percent decomposed $NaClO_2$ at a given bleaching time, (ii) at a given HMTA concentration, the higher the bleaching time, the higher is the percent decomposed $NaClO_2$, (iii) 100% $NaClO_2$ needs either long time at low HMTA concentration or shorter time at higher HMTA concentration. However, whiteness index and tensile strength measurements (Fig. 1) showed that 0.02 g/l is the optimum HMTA concentration, that at this concentration, $NaClO_2$ decomposes gradually within reasonable bleaching duration (120 min) giving rise to acceptable whiteness index and good tensile strength. Using lower HMTA leads to prolonging the bleaching duration up to 6 hours without further improvement in the whiteness index or the tensile strength. On the other hand, using higher concentration of HMTA leads to instant 100% decomposition of $NaClO_2$ without imparting any bleaching effect to the cotton fabrics. Moreover, it was noticed that the amount of ClO_2 evolved during bleaching is negligible irrespective of the reaction duration or the amount of HMTA incorporated to the bleaching medium. This indicates that using HMTA to activate $NaClO_2$ decomposition, the active species released is oxygen rather than ClO_2.

3.3. *Effect of HMTA concentration on pH of the bleaching bath*

Table 2 presents the change in bleaching bath pH with time, when bleaching reaction was carried out at 95 °C, using different initial concentrations of HMTA. The date shows that for a given initial hexamethylenetetramine concentration, the pH value decreases by prolonging the bleaching duration. For a given bleaching time (except 0 time), the same trend is also found, that the pH decreases as the initial hexamethylenetetramine concentration increases. These two remarks could be explained in terms of the effective role of both ammonia and formic acid, librated as a result of hexamethylenetetramine decomposition in the bleaching liquor (Eqs. (1) and (2)). At the early stage of the bleaching reaction, HMTA decomposes giving rise to the formation of ammonia and formaldehyde (Eq. (1)), that is why the medium is alkaline. As the reaction proceeds, more formaldehyde is oxidized to formic acid (Eq. (2)), which neutralized the alkaline effect of ammonia and then the excess of formic acid makes the medium turns acidic with time. However, the continuous production of ammonia due to the gradual decomposition of HMTA function as a buffer by neutralizing any excess acid produced, thus does not allowing the pH to fall and make the medium strongly acidic, and accordingly regulate sodium chlorite decomposition during the bleaching course. At hexamethylenetetramine concentrations higher than 0.02 g/l, large amounts of formic acids are formed, causing sudden decrease in the pH which leads to uncontrolled fast decomposition of sodium chlorite at a very early stage of the bleaching process without being used for actual decolorization of cotton fabric.

Table 1
Effect of [HMTA] on % decomposed $NaClO_2$.

Duration (min)	%Decomposed $NaClO_2$ at different HMTA concentrations (g/l)					
	0	0.005	0.01	0.02	0.2	0.5
30	6	18	25	34	98	100
60	21	38	43	69	100	
90	41	51	60	85		
120	62	75	83	100		
150	81	84	96			
180	89	98	100			
210	97	100				
240	100					

[$NaClO_2$], 5 g/l; non-ionic wetting agent, 1 g/l; M/L ratio, 1:30; bleaching bath temperature 95 °C.

3.4. Effect of HMTA concentration on physicochemical properties of bleached cotton fabric

The physicochemical properties of bleached cotton fabric are shown in Fig. 1. Results in Fig. 1 indicate that whiteness index increases by increasing [HMTA] up to 0.02 mg/l. Above this limit the whiteness index showed lower values but in general the whiteness index of the bleached fabric improved compared to the grey fabric. This could be explained in terms of very fast decomposition of $NaClO_2$ at theses high concentrations in the very early stage of bleaching duration without causing any bleaching effect to the fabric. Regarding the % loss in fabric weight, it increases continually as the concentration of HMTA increases, but after 0.02 g/l HMTA the percent weight loss decreases by increasing [HMTA]. Regarding the tensile strengths, the data show that regardless of [HMTA] used, the tenacity of the bleached cotton fabric is lower than that of the unbleached cotton fabric. The tensile strength decreases gradually on increasing [HMTA] up to 0.02 g/l but using amounts of HMTA higher than 0.02 g/l for bleaching cotton fabric leads to bleached samples with higher tenacity. The increase in weight loss which is of course accompanied by decrease in tensile strength of cotton fabric on increasing [HMTA] up to 0.02 g/l could be ascribed to removal of cementing and binding materials such as hemicelluloses and lignin. The decrease in weight loss (increment in tensile strength) and also the decrease in carbonyl and carboxyl contents on increasing [HMTA] over 0.02 g/l again indicate that these high concentrations of HMTA lead to useless fast decomposition of $NaClO_2$ without affecting cotton fabric

3.5. Effect of bleaching temperature on sodium chlorite decomposition

Table 3 shows the dependence of sodium chlorite decomposition, at fixed hexamethylenetetramine concentration (0.02 g/l), on the bleaching bath temperature. The results show that at the early stages of the bleaching reaction (the first 2 h), and at a given duration the percent decomposed sodium chlorite is proportionally related to the bleaching medium temperature. On carrying out the bleaching reaction at 95 °C, sodium chlorite is fully decomposed within 2 h, while on carrying the bleaching at lower temperature, namely 70 °C, 100% sodium chlorite decomposition needs 8 h to attain. On carrying out the bleaching reaction at lower temperatures, namely, 50 °C and 30 °C, 100% sodium chlorite decomposition was not attained even after 24 h. The enhancement in sodium chlorite activation at high bleaching temperatures (95 °C) is due to the favorable effect of temperature on decomposition of HMTA to give formaldehyde, which in turn activates sodium chlorite decomposition, plus giving the energy required for decomposition of sodium chlorite itself to the bleaching ingredients shown in Eqs. (1)–(8). Also one of HMTA decomposition products is ammonia which acts as buffer keeping the bleaching medium pH almost fixed during the course of reaction. In addition to the above, high temperature makes cotton fabric more swollen and increases its accessibility, giving rise to faster diffusion of the bleaching agent to the bulk of the fabric. The enhancement in sodium chlorite decomposition at lower temperatures (50 °C and 30 °C), by prolonging the reaction duration could be understood in terms of allowing enough opportunity for the said reaction to proceed.

3.6. Effect of reaction temperature on physicochemical properties of bleached cotton fabric

Fig. 2 represents physicochemical properties of cotton fabric bleached at different temperatures, keeping sodium chlorite and hexamethylenetetramine concentrations constant in all cases (5 g/l and 0.02 g/l, respectively). The data in Fig. 2 indicate that the higher the bleaching temperature, the better is the whiteness index of the bleached fabric. This could be explained in terms of the above mentioned dependence of sodium chlorite and hexamethylenetetramine decomposition on the bleaching medium temperature. Raising the bleaching temperature from 30 °C up to 95 °C increases the rate of hexamethylenetetramine decomposition

Table 2
Effect of [HMTA] on bleaching bath pH.

Time (min)	pH change on using different HMTA concentrations (g/l)				
	0.005	0.01	0.02	0.2	0.5
0	7.4	8	8.2	9.9	10.8
30	7.1	6.5	6.4	6	4
60	6.3	6.3	5.6	4	
90	6	6	5		
120	5.6	5.4	4.2		
150	5.3	5			
180	5.3	5			
210	5				
240	5				

[$NaClO_2$], 5 g/l; non-ionic wetting agent, 1 g/l; M/L ratio, 1:30; bleaching bath temperature 95 °C.

Table 3
Effect of bleaching bath temperature on % decomposed $NaClO_2$.

Time (h)	Bleaching temperature			
	30	50	70	95
0.5	3	3	15	34
1	6	9	20	69
1.5	8	16	25	85
2	15	25	47	100
4	22	49	80	
6	29	54	93	
8	33	67	100	
10	41	71		
12	46	77		
24	51	80		

[$NaClO_2$], 5 g/l; [HMTA], 0.02 g/l; non-ionic wetting agent, 1 g/l; M/L ratio, 1:30.

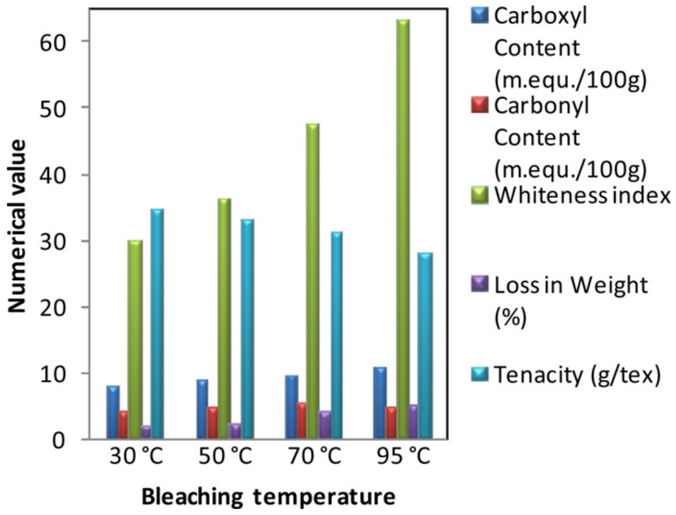


Fig. 2. Effect of bleaching temperature on physicochemical properties of bleached cotton fabrics.

to its components formaldehyde and ammonia. As already mentioned in the above scheme (Eq. (2)), sodium chlorite is reduced by formaldehyde to give the active bleaching species (Eqs. (3)–(8)).

The data also show that the percent loss in fabric weight increases by raising the bleaching temperature. This is related to the improvement in whiteness index by raising the bleaching temperature. The noncellulosic materials present naturally in the structure of cotton fabric are responsible for imparting dark color to the fabric. The higher the bleaching temperature, the more effective is the oxidative reaction in removing these noncellulosic materials (higher percent loss in fabric weight) and this of course will be reflected on improved whiteness index. Moreover, this higher percent loss in fabric weight accompanying raising bleaching temperature will also appear as loss in tensile strength of the bleached fabric. These noncellulosic materials are responsible for good tensile strength of the fabric but also impart some disadvantages to the fabrics like, dark color and hydrophobicity. Partial removal of such non-cellulosic materials from the fabric is necessary to improve its water absorbency, but this will be of course on count of fabric tensile strength.

Fig. 2 shows enhancements in the magnitude of carboxyl and carbonyl contents upon raising the bleaching temperature from 30 °C to 95 °C. This is due to acceleration in the bleaching redox reaction, which is responsible for introducing carboxyl and carbonyl function groups to the cellulosic backbone of cotton fabric.

4. Conclusion

Sodium chlorite/hexamethylenetetramine bleaching system was tried to bleach Cotton fabric in one-step process. Different bleaching trials were carried out keeping fixed sodium chlorite concentration and varying other reaction conditions like hexamethylenetetramine concentration (0.005, 0.01, 0.02, 0.2 and 0.5 g/l) and bleaching temperature (30, 50, 70 and 95 °C). The bleaching reaction was monitored by following the bleaching liquor pH and percent decomposed sodium chlorite at different time intervals. The obtained results indicate that the higher the concentration of HMTA incorporated to the bleaching medium, the higher is the percent decomposed $NaClO_2$ at a given bleaching time. Also at a given HMTA concentration, the higher the bleaching time, the higher is the percent decomposed $NaClO_2$. Also pH measurements showed that for a given initial hexamethylenetetramine concentration, the pH value decreases by prolonging the bleaching duration. For a given bleaching time (except 0 time), the same trend was also found, that the pH decreases as the initial hexamethylenetetramine concentration increases. The bleached fabrics were fully characterized by measuring their whiteness, carboxyl and carbonyl contents, percent loss in fabric weight and tensile strength. The obtained results reveal that bleached cotton fabric with satisfactory whiteness index and reasonable tensile strength can be obtained by treating the fabric at 95 °C in a bleaching bath containing 5 g/l sodium chlorite, 0.02 g/l hexamethylenetetramine and 1 g/l non-ionic wetting agent using a material to liquor ratio of 1:30. These optimum conditions lead to completion of the bleaching process in reasonable duration of 2 h with minimum evolution of harmful chlorine dioxide gas. Lower concentrations of the activator hexamethylenetetramine were found to prolong the bleaching duration without getting satisfactory whiteness index due to incomplete decomposition of sodium chlorite, while on the other hand, higher concentrations of the activator hexamethylenetetramine were found to cause very fast sodium chlorite decomposition at very early stage of the reaction without imparting any bleaching effect to the cotton fabric.

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