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BLEACHING OF LOOM-STAT COTTON FABRIC USING ACTIVATED SODIUM CHLORITE / HEXAMETHYLENETETRAMINE SYSTEM

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Abstract

Hexamethylenetetramine (HMTA) has been investigated as novel activator for sodium chlorite to effectively bleach loom state cotton fabric in one step process and in slightly alkaline medium. This was done with a view to avoiding the troubles associated with evaluation of chlorine dioxide during chlorite bleaching. Two techniques have been separately investigated to carry out the bleaching process, namely, exhaustion and pad steam technique. The process parameters in each technique have been investigated. Results obtained show that the addition of HMT A decreased the decomposition percent of sodium chlorite during the bleaching process. It has been also found that loom state cotton fabric can be bleached in one step process with NaClO₂ / HMT A using either exhaustion or pad-steam technique. In exhaustion system maximum W.I and wettability of bleached cotton fabric was obtained when loom state cotton fabric was bleached with an aqueous solution containing 3 g/l NaClO₂ and 0.08 g/l HMT A. Whereas, in pad steam technique, maximum W.I and wettability of bleached cotton fabric was obtained when loom state cotton fabric was bleached with an aqueous solution containing 20 g/l NaClO₂ and 0.6 g/l HMT A then steamed for 60 min.

Keywords: Bleaching, Cotton Fabric, Hexamethylenetetramine, Sodium chlorite.

Introduction

Conversion of grey cotton fabrics to full bleached fabric involves three distinct steep, namely, desizing to remove the size, scouring to remove fats, waxes and pectins and bleaching to remove coloring matters. During these steps, the overall energy consummation is very high. The continuously increase in the energy cost stimulate considerable researches and development to establish a commercially viable single-stage preparatory process containing desizing scouring and bleaching⁽¹⁻¹²⁾.

Chlorite bleaching technology has undergone a rapid development and due to the several advantageous it offers, it has been adopted more and more in the existing bleaching methods. In bleaching with chlorite, an activator is usually used to control the pH and to catalyze the chlorite solution to a maximum bleaching effect⁽⁹⁻¹²⁾. Generally, these activators are either acidic in nature like phosphoric, acetic, formic, hydrochloric and oxalic acid or change to acid under the effect of chlorite and heat, like formaldehyde. Nowadays, direct addition of acid to activate the chlorite solution is avoided. This is because the acids vigorously liberate the toxic and corrosive chlorine dioxide gas⁽⁹⁻¹²⁾.

Recently, It has been reported that to avoid the troubles associated with evolution of chlorine dioxide during chlorite bleaching, the activation was carried out with formaldehyde at pH10⁽¹²⁾.

Sodium chlorite bleaching procedure depends on the machinery used, the form of the textile and the end use of the product. Woven fabrics normally desized and scoured whereas yarns and knitted fabrics simply washed off prior to bleaching to remove spinning or knitting lubricant. Because of the danger from chlorine dioxide evolution, hooded machines connected to an extraction system are usually required and even these area should also be well ventilated. After treatment with 2-5 g/l detergent plus 2 - 5 g/l soda ash at 85 - 95°C will often enhance whiteness and will usually improve absorbency of the substrate. An antichlor is only carried out for goods to be subsequently dyed. This can be accomplished by replacing the soda ash in the after treatment with sodium perborate or percarbonate but is more usually accomplished with sodium thiosulphate or oxalic acid. The reductive antichlor (hydro sulphite or bisulphite) are seldom used as they replace one problem with another.

The work presented in this paper aimed at developing a new activator for chlorite solution based on hexamethylenetetramine in neutral medium. The work also extend to investigate the technical feasibility and adaptation of one step desizing, scouring and bleaching of the loom state cotton fabrics using activated sodium chlorite/hexamethylenetetramine system.

Experimental

Materials

Starch-sized plain weave cotton fabric (3D picks, 48 ends/cm) was kindly supplied by Misr Company for Spinning and Weaving. Mehala-El-Kubra, Egypt. Sodium chlorite (NaClO_2) and hexamethylenetetramine (HMTA) were of laboratory grade chemicals. Nonidet LE* (non-ionic wetting agent) from Shell Textile Chemical Company, Cairo, Egypt.

Bleaching of loom state cotton fabric using exhaustion method:

Grey cotton fabric was impregnated in a solution containing NaClO_2 (2-5g/l), HMT A (0-0.1 g/l) and nonionic wetting agent (2g/l) at temperature 95°C for 30-150 min.

The liquor ratio was adjusted at 3D. After the desired reaction time, the samples were washed thoroughly with hot water (70°C) rinsed in cold water and finally dried at ambient conditions.

Bleaching of loom state cotton fabric using pad-steam technique

Grey cotton fabric was padded in a solution containing sodium chlorite (20 g/l), HMT A (0.4 - 0.8 g/l) and non-ionic wetting agent (2 g/l), The sample was then squeezed to wet pick up of Ca. 80%(contain about 80%)and batched in thermostatic Launder ameter (type Atlas) at 100°C for different lengths of time (30-90 min). After the desired reactions time the sample was then washed thoroughly with hot water (70°C) for 5 min then rinsed with cold water and finally dried at ambient conditions.

Testing and Analysis:

The degree of whiteness of cotton fabric, expressed as whiteness index was measured by using Hunter lab. Color Difference Meter D 25 M/L-2. The wettability of the sample before and after bleaching was assessed by drop disappearance method according AATCC standard test method,⁽⁹⁾. Reported methods were used for determination of the copper number⁽¹⁰⁾ and carboxyl content⁽¹¹⁾ of the bleached cotton fabrics. The percent loss in fabric weight was calculated as follows:

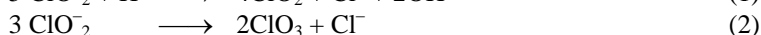
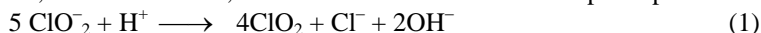
$$\% \text{ loss in fabric weight} = \frac{W_1 - W_2}{W_1} \times 100$$

Where: W_1 and W_2 are the dly weight of the cotton fabric before and after bleaching respectively.

The tensile strength and elongation at break were determined according to ASTM standard method⁽¹²⁾. The rate of sodium chlorite decomposition was monitored according to reported method⁽¹³⁾.

Results and Discussion:*Tentative Mechanism:*

The bleaching activity of sodium chlorite depends strongly on pH. The reactions occurring in solution are highly complex and have been reviewed⁽¹⁻⁹⁾. According to several authors, the chlorous acid (HClO_2) formed in solution by hydrolysis of the sodium salt undergoes the series of reactions shown by equations 1-3. In order to achieve the necessary concentration of chlorous acid, acid or latent acid generators (activators) must be added to the bleaching solution. Activators include sodium chloroacetate, chloroacetamide, triethanolamine and ammonium persulphate⁽¹²⁾.

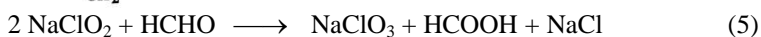
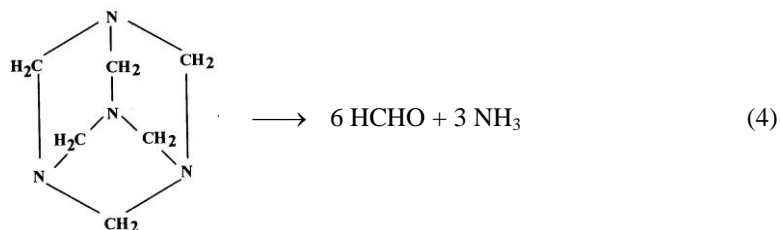


Bleaching occurs most rapidly below pH 1-2, rapidly at pH 2-3 and the rate decreases as the pH increases to 8-9, between pH 2 and 9, the rate is proportion to the concentration of chlorous acid in solution. Below pH 2 the species providing the bleaching action is chlorine dioxide, a toxic, explosive, corrosive gas. In practice, a balance is usually struck between the rate of bleaching and the evolution of chlorine dioxide by controlling the bleach liquor pH. This is conveniently done on batch machinery by adjusting the pH to 3.5-4.5 with acetic or formic acid and then buffering at this pH by addition of sodium dihydrogen phosphate. The phosphate addition not only buffers the system but also improves whiteness^(1,11,12).

In this work, a study was made to activate sodium chloride solution by using hexamethylenetetramine (HMTA) instead of traditional acids. This was undertaken with a view to establishment the most appropriate condition for bleaching the loom state cotton fabric using NaClO_2 /HMTA in pH range of 7-7.5 to ensure minimum

liberation of toxic chlorine dioxide and lower fabric tendering.

It has been demonstrated that one molecule of HMT A decomposes gradually in aqueous solution to yield six molecules of formaldehyde and four molecules of ammonia as shown equation 4⁽¹²⁾.



The suggested reaction scheme (equations 4-6) features that: the gradual release of formaldehyde by hydrolysis of HMT A (equation 4) acts as a catalyst for chlorite bleaching (as shown by equations 5-6) whereas the simultaneously liberating ammonia behaves as a buffering agent to prevent sudden decrease in solution pH and consequently, the liberation of undesirable ClO_2 is minimized.

Considering the above reaction scheme, the magnitude of chlorite activation would rely on HMT A concentration, bleaching temperature, bleaching time, material to liquor ratio and application technique. In this regard, two techniques, namely exhaustion techniques and pad steam technique were separately investigated to carry out the bleaching of loom state cotton fabric using NaClO_2 /HMTA system. The percent decompose of NaClO_2 during the bleaching process was monitored, whereas the bleached fabric was evaluated for tensile strength, elongation at break, total loss in fabric weight after bleaching, whiteness index and wettability. The bleached fabric was monitored for copper number and carboxyl content. Results obtained along with their appropriate discussion are given below.

Bleaching of loom state cotton fabric using exhaustion method

Effect of HMT A concentration:

Loom state cotton fabric was bleached in an aqueous solution containing (2-5 g/l) NaClO_2 along with HMT A (0-0.1g/l). The liquor ratio was adjusted at 30 and the bleaching temperature was kept at 95°C for different duration. The bleaching reaction was monitored via determining the percent decomposed chlorite throughout the bleaching time and the physico-chemical properties of the bleached fabric.

Figures 1, 2 and 3 show the effect of HMTA concentration on the decomposed percent of sodium chlorite. The results signify the following:

(a) Regardless the concentration of HMT A concentration, the percent decomposed of sodium chlorite increased as the bleaching time increased.

(b) At given bleaching time, increasing the concentration of HMT A decreases the decomposition percent of sodium chlorite solution. This was observed with all concentrations of sodium chlorite used. On using 2g/l sodium chlorite addition of 0.1 g/l HMT A decreases the decomposition percent after two hours from about 60 % to only 20 %. Similar results were obtained when using 3 and 5g/l sodium chlorite.

It could be emphasized from results of figure 1-3 that: the addition of HMT A decreased the decomposition percent of sodium chlorite during the bleaching process.

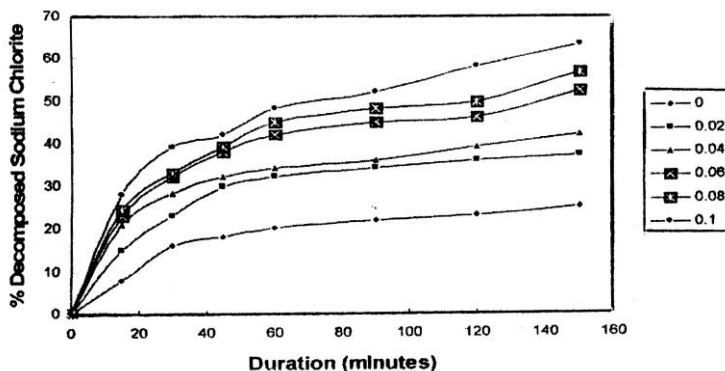


Fig. 1: Effect of [HMTA] on NaClO₂ Decomposition Reaction Conditions: Temperature 95°C, Sodium Chlorite 2g/l.

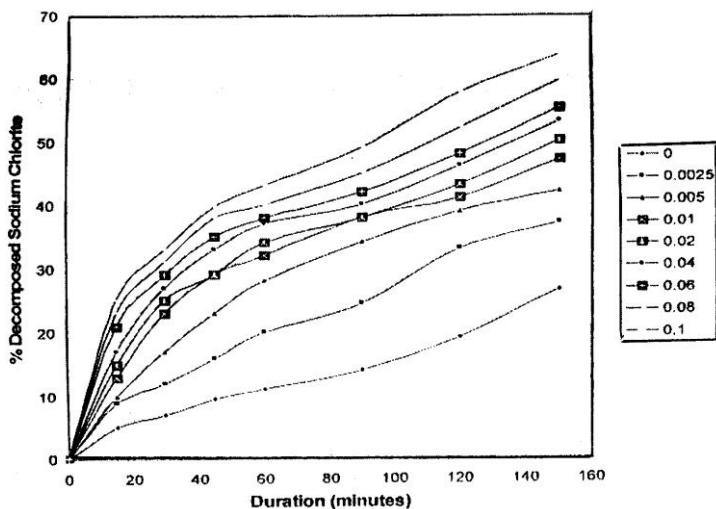


Fig. 2: Effect of [HMTA] on NaClO₂ Decomposition Reaction Conditions: Temperature 95°C, Sodium Chlorite 3g/l.

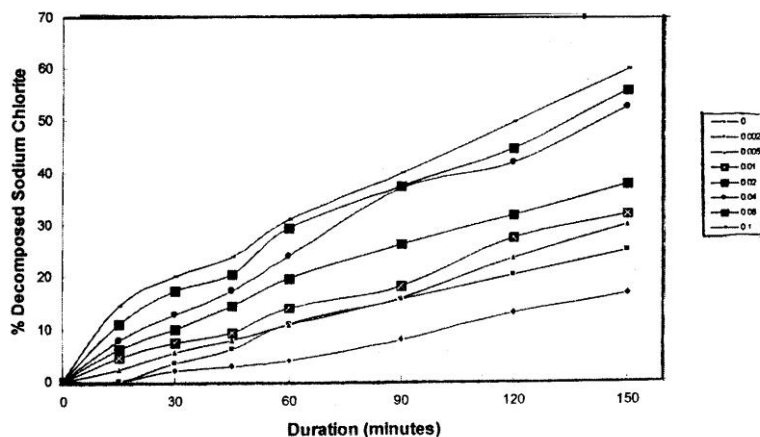


Fig. 3: Effect of [HMTA] on NaClO_2 Decomposition Reaction Conditions: Temperature 95°C , Sodium Chlorite 5g/l .

Effect of NaClO_2 and HMTA concentrations on physico-chemical properties of the exhaust bleached cotton fabric:

Loom state cotton fabric was bleaching using different concentration nom NaClO_2 and HMTA. Physico-chemical properties of the bleached fabric are determined. These properties include wettability, whiteness index, total loss in fabric weight, degree of polymerization (DP) and tensile strength. The carboxyl content and copper number for the bleached fabric were also determined. Results obtained are set out in Tables I-IV. Results of grey fabric are set out in the same table for comparison.

Table I shows the effect of NaClO_2 and HMT A concentrations on the whiteness index (WI), loss in fabric weight after bleaching and wettability of the bleached cotton fabric. Table I makes it clear that:

a) At constant HMTA concentration, increasing NaClO_2 concentration is accompanied by increasing in WI of the bleached cotton fabric.

b) At zero HMTA concentration, maximum value of W.I is obtained at 5 g/l NaClO_2 is 55, whereas approximately similar result was obtained on using 2 g/l along with 0.1 g/l HMT A. A point which indicates that, upon addition of 0.1 g/l HMT A to NaClO_2 bleaching bath the amount of NaClO_2 used in the bleaching could be reduces to less than half amount without detracting from the fabric W.I

c) At constant NaClO_2 concentration, increasing HMTA concentration from zero to 0.08 g/l increases the W.I of the bleached fabric. Further increase in HMTA concentration has little effect on W.I.

d) Similar trends were observed with the % loss in weight and wettability of the bleached cotton fabric.

A close examination of results in Table I show that, maximum W.I and wettability of bleached cotton fabric was obtained when loom state cotton fabric was bleached with exhaustion technique using an aqueous solution containing 3 g/l NaClO_2 and 0.08 g/l HMTA.

Table I: Effect of sodium chlorite and HMT A concentrations on whiteness index, loss in weight and wettability of the bleached cotton fabric:

[HMTA] conc. (g/l)	[NaClO_2] concentration (g/l)								
	1			2			3		
	W.I	Loss in weight (%)	Wet. (sec) ^a	W.I	Loss in weight (%)	Wet. (sec) ^a	W.I	Loss in weight (%)	Wet. (sec) ^a
Blank	15.0	----	>3	15.0	-----	>3	15.00	-----	>3
0.00	16.6	3.5	4	30.0	4.0	2	55.40	5.84	2
0.0025	17.0	3.0	2	37.31	4.0	2	63.60	5.45	<1
0.005	19.0	3.0	2	54.55	4.0	1	63.40	5.90	<1
0.01	20.0	3.7	2	60.21	4.8	1	66.0	6.00	<1
0.02	27.15	3.7	2	61.96	4.8	<1	60.43	5.50	<1
0.04	39.38	3.4	1	61.97	4.9	<1	67.34	5.80	<1
0.06	54.81	3.4	1	65.43	5.2	<1	67.64	5.97	<1
0.08	54.67	3.4	<1	67.27	5.3	<1	67.00	5.96	<1
0.1	55.69	3.4	<1	67.5	5.3	<1	67.70	5.98	<1

(a) Wet. = wettability

Reaction Conditions: Exhaustion technique was used, liquor ratio, 30; bleaching temperature, 95°C; bleaching time 150 min.

Table II shows the effect of different concentration NaClO_2 /HMTA bleaching system on the chemical degradation of cotton fabric after bleaching. Chemical degradation was assessed via determining the copper number and carboxyl content of cotton fabric. It is clear from Table II that the, at given NaClO_2 concentration, the copper number and carboxyl content of the bleached cotton fabric decreased as the concentration of HMT A increased then approximately level off. The initial decrement in copper number and carboxyl content compared with grey untreated cotton fabric (blank) is attributed to the removal of noncellulosic materials like starch size, natural impurities like fats, waxes, pectins motes and coloring matter.

Results of table II make it clear that the addition of HMTA to chlorite bleaching solution generally does not degrade cotton cellulose even if the chlorite is in large excess.

Table II: Effect of sodium chlorite and HMT A concentrations on the carboxyl content and copper number of bleached cotton fabric

HMTA Cone. (g/l)	NaClO ₂ Concentration (g/l)					
	2		3		5	
	Carboxyl content (meq/100e)	Copper number	Carboxyl content (meq/100e)	Copper number	Carboxyl content (meq/100e)	Copper number
Blank	12.00	0.5821	12.00	0.5821	12.00	0.5821
0.00	11.00	0.212	9.521	0.201	7.532	0.199
0.0025	10.823	0.200	8.621	0.199	7.821	0.189
0.005	8.832	0.181	7.821	0.180	8.221	0.179
0.010	8.621	0.172	7.531	0.170	8.5	0.172
0.020	8.13	0.170	7.321	0.169	8.662	0.166
0.040	7.932	0.162	7.831	0.165	8.811	0.160
0.060	7.832	0.160	7.921	0.159	8.882	0.158
0.080	7.530	0.153	8.123	0.150	8.912	0.148
0.100	7.553	0.151	8.521	0.149	8.999	0.147

Reaction Conditions: Exhaustion technique was used. liquor ratio, 30; bleaching temperature. 95°C: bleaching time 150 min.

Table III: Effect of sodium chlorite and HMT A concentrations on the tensile strength and degree of polymerization of bleached cotton fabric

HMTA Cone. (g/l)	NaClO ₂ Concentration (g/l)					
	2		3		5	
	DP ^a	T.S (kg.f) ^b	DP	T.S (kg.f)	DP	T.S (kg.f)
Blank	2500	67	2500	67	2500	67
0.00	2300	65	2200	62	2100	60
0.0025	2100	62	2099	60	2098	58
0.005	2090	60	2085	59	2080	55
0.010	2060	56	2049	57	2042	50
0.020	2050	55	2041	55	2038	48
0.040	2020	52	2013	52	2035	45
0.060	2001	50	2000	49	1999	42
0.080	2000	49	1983	42	1980	42
0.100	1992	48	1980	42	1975	40

^(a) DP = Degree of polymerization, ^(b) T.S = tensile strength

Reaction Conditions: Exhaustion technique was used, liquor ratio, 30; bleaching temperature, 95°C; bleaching time 150 min.

Effect of exhaustion time

Table IV shows the effect of exhaustion time on W.I. loss in fabric weight and wettability of the cotton fabric after bleaching with NaClO₂ and HMT A using exhaustion method. The bleaching process was carried out using two different concentrations from NaClO₂ / HMTA, namely 2/0.08 and 3/0.08. It is seen from

Table IV that: the maximum value of W.I is obtained after 120 min duration and the fabric shows wettability time less than 1 second. This was observed irrespective to the concentration of bleaching agent used but with certainty that the values of W.I are higher at higher chlorite concentrations.

Table V shows the effect of exhaustion time on carboxyl content and copper number of NaClO₂ / HMTA bleached cotton fabric. It is clear from Table V that the value of carboxyl content and copper number decreases by increasing the bleaching time from 60-120 min. Further increase in the bleaching time is attributed by increasing in carboxyl content and copper number of bleached cotton fabric. The initial decrement in carboxyl content and copper number is attributed to the removal of sizing agent and other cotton impurities. Prolonged bleaching time oxidize cotton cellulose with formation of new carboxyl and/or aldehyde groups which enhance the carboxyl content and copper number of cotton fabric.

Table IV: Effect of exhaustion time on whiteness index, wettability and percent loss in fabric weight

Duration (min)	[NaClO ₂] concentration (g/l)					
	2/0.08			3/0.08		
	WI	% loss in weight	Wettability (sec)	WI	% loss in weight	Wettability (sec)
Blank	15.0	--	> 180	15.0	---	> 180
60	19.64	2.7	1.8	49.7	2.7	1.3
90	48.92	3.5	1	57.91	3.2	1
120	59.24	3.8	<1	63.44	3.9	<1
150	59.79	4.0	<1	61.24	4.0	<1

Reaction Conditions: Exhaustion technique was used, liquor ratio, 30; temp., 95°C.

Table V: Effect of exhaustion time on carboxyl content and copper number of bleached cotton fabric

Duration (min)	[NaClO ₂] concentration (g/l)			
	2/0.08		3/0.08	
	Carboxyl content (meq/100g)	Copper number	Carboxyl content (meq/100g)	Copper number
Blank	12.00	0.5821	12.00	0.5821
60	9.532	0.188	8.233	0.180
90	8.533	0.178	8.532	0.178
120	7.310	0.165	8.432	0.165
150	7.530	0.151	8.721	0.150

Reaction Conditions: Exhaustion technique was used, liquor ratio, 30; temp., 95°C.

Table VI shows the effect of exhaustion time on tensile strength (TS) and degree

of polymerization (DP) of NaClO₂ / HMTA bleached cotton fabric. It is clear from Table VI that increasing the bleaching time decreases the tensile strength and DP values of bleached cotton fabrics. This was observed irrespective to the concentration of sodium chlorite but with certainty that the decrement is higher at higher chlorite concentration. A salient feature of results in table VI reveals that after 120 min duration the fabric has about 90 % retained tensile strength and 80% retained DP value (calculated with respect to the corresponding blank values).

It could be emphasized from results in tables IV-VI that optimum bleaching time for grey cotton fabric using NaClO₂ I HMT A depends on the concentration of NaClO₂ used. On using 2 g/l NaClO₂, 120 min duration is required to obtain a complete bleaching without fabric deterioration. On the other hand, if 3 g/l NaClO₂ is used only 90 min duration is required to obtain the same effect.

Table VI: Effect of bleaching time on tensile strength (TS) and degree of polymerization (DP) of bleached cotton fabric

Duration (min)	[NaClO ₂] concentration (g/l)			
	2/0.08		3/0.08	
	DP ^a	T.S. ^b (kg.f)	DP ^a	T.S. ^b (kg.f)
Blank	2500	67	2500	67
60	2260	55	2110	48
90	2101	50	1988	47
120	2000	51	1895	45
150	1992	48	1737	42

^(a) DP = Degree of polymerization. ^(b)T.S = tensile strength

Reaction Conditions: Exhaustion technique was used. liquor ratio, 30; temp., 950176C.

Bleaching of loom state cotton fabric using pad-steam method

Loom state cotton fabric was subjected to bleaching with HMTA activated NaClO₂ solution using pad-steam techniques and under a variety of conditions. The latter were including HMTA concentration and steaming time. Physico-chemical properties of the bleached fabric are determined. These properties include wettability, whiteness index, total loss in fabric weight, degree of polymerization (DP) and tensile strength. Results obtained are set out in Tables VII, VIII. Results of grey fabric are set out in the same tables for comparison.

It is seen from Table VII that, at certain HMT A concentration, increasing the steaming time is accompanied by increasing in whiteness index and wettability of

the bleached fabric whereas the total loss in the fabric weight is approximately constant. Obviously, 60 min steaming is enough to make complete bleaching. Further increase in the steaming time exerts approximately no effects. It is further observed from Table VII that at constant steaming time, increasing HMTA concentration is accompanied by slightly increase in whiteness index and % loss in the fabric weight whereas fabric wettability remain improved with all HMT A concentrations.

Table VII: Effect of HMT A concentration and steaming time on whiteness index, loss in weight and wettability of the bleached cotton fabric:

[HMTA] conc. (g/l)	[HMTA] conc. (g/l)								
	0.4			0.6			0.8		
	W.I	Loss in weight (%)	Wet. (sec) ^a	W.I	Loss in weight (%)	Wet. (sec) ^a	W.I	Loss in weight (%)	Wet. (sec) ^a
Blank	15.0	>180	----	15.0	> 180	----	15.0	> 180	----
30	50.89	1	2.80	31.73	1	2.97	48.23	1	3.1
45	53.52	1	2.76	50.27	1	3.30	50.55	1	3.1
60	58.79	1	2.80	61.85	1	3.10	61.70	1	3.4
90	59.60	1	2.82	61.46	1	3.00	61.16	1	3.5

^(a) Wet. = wettability

Reaction Conditions: Grey cotton fabric was padded in aqueous solution containing HMT A and 20 g/l NaClO₂, squeezed to ca., 80 % wet pick up then steaming for specified time

Table VIII shows the effect of HMT A concentrations and bleaching time on tensile strength and degree of polymerization of bleached cotton fabric. Results of Table VIII which are self explanatory make it clear that optimum concentration of HMTA is 0.6 g/l along with 20 g/l NaClO₂ and optimum steaming time is 60 min.

Table VIII: Effect of HMT A concentrations and bleaching time on tensile strength and degree of polymerization of bleached cotton fabric

HMTA Conc. (g/l)	NaClO ₂ Concentration (g/l)					
	2		3		5	
	DP ^a	T.S (kg.f) ^b	DP	T.S (kg.f)	DP	T.S (kg.f)
Blank	67	2500	67	2500	67	2500
30	58	2300	56	2280	54	2260
45	57	2290	55	2270	52	2248
60	55	2210	53	2208	53	2200
90	54	2190	52	2168	49	2130

^(a) Wet. = wettability

Reaction Conditions: Grey cotton fabric was padded in aqueous solution containing HMT A and 20 g/l NaClO₂, squeezed to ca., 80 % wet pick up then stemming for specified time.

Conclusions:

Loom state cotton fabric was bleached in one step process using a novel chlorite bleaching activator. The latter involve the addition of hexamethylenetetramine to sodium chlorite bleaching solution. Results obtained show that the addition of HMT A decreased the decomposition percent of sodium chlorite during the bleaching process. It has been also found that loom state cotton fabric can be bleached in one step process with NaClO₂ / HMT A using either exhaustion or pad-steam technique. In exhaustion system maximum W.I and wettability of bleached cotton fabric was obtained when loom state cotton fabric was bleached with an aqueous solution containing 3 g/l NaClO₂ and 0.08 g/l HMTA. Whereas, in pad steam technique, maximum W.I and wettability of bleached cotton fabric was obtained when loom state cotton fabric was bleached with an aqueous solution containing 20 g/l NaClO₂ and 0.6 g/l HMT A then steamed for 60 min.

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الملخص العربي

تم تبيض الأقمشة القطنية الخام بواسطة كلوريت الصوديوم المنشط سداسي الميثيلين رباعي الأمين في خطوة واحدة وذلك لتوفير الوقت والجهد والطاقة والكماويات . تم دراسة الخواص الميكانيكية والفيزيائية والكيميائية للأقمشة القطنية المبيضة فكانت أفضل النتائج كالآتي:

تركيز كلوريت الصوديوم 2جم/لتر-تركيز سداسي الميثيلين رباعي الأمين 0.1جم/لتر عند درجة حرارة 95 درجة مئوية وزمن التفاعل 150 دقيقة مع استخدام عامل تنديية بتركيز 2جم/لتر وكانت نسبة الصلب الي السائل 30 : 1 . هذه الظروف بالنسبة لطريقة الغمر . أما أفضل الظروف بالنسبة لطريقة العصر فهي كالآتي : تركيز كلوريد الصوديوم 20جم/لتر - تركيز سداسي الميثيلين رباعي الأمين 0.6جم/لتر وذلك لمدة ساعة