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One-step bleaching process for cotton fabrics using activated hydrogen peroxide

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ABSTRACT

Cotton fabric was bleached in a simple and economic process using a bleaching system composed of hydrogen peroxide activated with thiourea. Different bleaching trials were carried out with varying hydrogen peroxide and thiourea concentrations, as well as the bleaching medium temperature. The obtained results reveal that bleached cotton fabric with satisfactory whiteness index and reasonable tensile strength can be obtained by treating the fabric at 90 °C in a bleaching bath containing 6 g/l hydrogen peroxide, 1.5 g/l thiourea and 1 g/l non-ionic wetting agent using a material to liquor ratio of 1:20. These optimum conditions lead to completion of the bleaching process in a reasonable duration of 1 h. Lower concentrations of the activator thiourea were found to prolong the bleaching duration without getting satisfactory whiteness index. Higher concentrations of the activator were found to cause early termination of the oxidizing species leading to bad whiteness index.

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 of the cotton flower. The climate, soil, drought and frost can also cause various degrees of yellowness. Apart from these, the loom-state fabric is also contaminated with size, processing lubricant, such as machine oils, tars, and greases from harvesting, ginning, spinning and weaving. Prior to the dyeing and other finishing processes, non-cellulose substances have to be removed and natural pigments discolored. Cotton cellulose has excellent properties such as high water and moisture absorbency, being comfortable to wear and easy to dye. For these reasons, the apparel industry is predominantly cotton based, and the share of cotton in total fiber consumption is about 50% (Abdel-Halim, Fahmy, & Fouda, 2008; Fahmy, Aboshosha, & Ibrahim, 2009; Hebeish et al., 2009; Hou, Wang, & Yu, 2009; Hou, Zhou, & Wang, 2009; Shafie, Fouda, & Hashem, 2009; Wei, Cheng, Hou, & Sun, 2008; Xie, Wang, & Xu, 2009). Cotton is composed almost entirely of cellulose (90–96% based on weight of fibers). The impurities in cotton fiber range from 4% to 10% (Abdel-Halim & Al-Deyab, 2011a; Hashem, El-Bisi, Sharaf, & Refaie, 2010).

Hydrogen peroxide is widely employed as an environmentally benign bleaching agent used to bleach wood pulp (Abdel-Halim, El-Rafie, & Kohler, 2008; Abdel-Halim, Konczewicz, Zimniewska, Al-Deyab, & El-Newehy, 2010; Abrantes, Amaral, Costa, Shatalov, & Duarte, 2007) and raw cotton (Tian, Branford-White, Wang, Nie, & Zhu, 2012). A variety of chromophores present on the substrates need to be whitened or removed using bleaching chemicals. As the chromophores vary in chemical nature, different types of reactivity are required. The main bleaching systems used for laundry cleaning, raw cotton bleaching and pulp-paper bleaching are chlorine-based bleaches (Abdel-Halim, 2012a, 2012b), hydrogen peroxide (Hou, Zhang, & Zhou, 2010), ozone (Shatalov & Pereira, 2010) and peracids (Abdel-Halim & Al-Deyab, 2011b). Although hydrogen peroxide is from an environmental point of view the most favorable bleaching agent, long reaction times or high temperatures are required to attain sufficient bleaching of chromophores, like 3 h at 90 °C for wood pulp (Requejo, Rodríguez, Colodette, Gomide, & Jiménez, 2012). As most consumers do not wash generally at this temperature, alternative bleaches for laundry applications were developed. They are mostly based on peracids, such as peracetic (Abdel-Halim, Abdel-Mohdy, et al., 2010; Abdel-Halim & Al-Deyab, 2011a, 2011b) which give bleaching at considerably lower temperatures, but are still not active enough below 40 °C.

The chemistry of hydrogen peroxide bleaching is reviewed by Renders, Chauveheid, and Pottier (1995) and Ackermann (2000). The bleaching effect uses the dissociation of hydrogen peroxide in water to form a hydronium ion (H_3O^+) and a perhydroxyl ion (HO_2^-):

$$H_2O_2 + H_2O \leftrightarrow HO_2^- + H_3O^+$$

The perhydroxyl anion acts as a nucleophilic bleaching agent and increasing its concentration is necessary to achieve a high bleaching effect. This is possible by increasing the hydrogen

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peroxide concentration and by addition of sodium hydroxide. The following is activation of the hydrogen peroxide:

$$H_2O_2 + OH^- \leftrightarrow H_2O + HO_2^-$$

Some attempts have been made to investigate new compounds as activator for H_2O_2 bleaching in order to reduce bleaching temperature. Hydrogen peroxide can bleach cotton cellulose fabrics using bleach activators at the low temperature conditions. Tetraacetylethylenediamine (TAED) is well known as activator for H_2O_2 (Khristova, Tomkinson, & Jones, 2003). TAED activating hydrogen peroxide system can improve bleaching effectiveness under mild conditions. In fact, bleach activators are peracid precursors, which generate peracids in situ in an alkaline hydrogen peroxide solution. The peracids are safe and efficient bleaching agent at mild condition (Abdel-Halim & Al-Deyab, 2011a, 2011b). However, TAED is not soluble in water and its water insolubility limits its application in the textile bleaching field.

Thiourea is an important reagent in various industrial productions (Gönen, 2003; Örgül & Atalay, 2000), and also valuable reactants in fundamental scientific researches, especially in the study of nonlinear reaction dynamics. Only a few chemical systems are known to be capable of exhibiting such rich dynamics. The studies of the kinetics and mechanisms of the oxidation of thiourea have attracted a great deal of attention (Zhou, Peng, Yang, & Wang, 2008). These investigations led to a general conclusion that the oxidation of thiourea goes through S-oxygenation to form sulfenyl, then sulfinic and sulfonic acids, and finally sulfate ions. Apart from scientific interests, the oxidation kinetics of thiourea and thiourea oxides may also be critical in the understanding of the physiological effects of sulfur-containing species. The treatment of thiourea with hydrogen peroxide in neutral (pH 7.4) or alkaline media gives thiourea dioxide.

$$(NH_2)_2CS + 2H_2O_2 \rightarrow (NH)(NH_2)CSO_2H + 2H_2O$$

The present study aims at enhancing the degree of whiteness of the scoured cotton cellulose in one step using thiourea as an activator for hydrogen peroxide bleaching process. Besides searching for the proper conditions for attaining better degree of whiteness along with good tensile properties.

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 hydroxide, hydrogen peroxide, thiourea, sulfuric acid, sodium carbonate, potassium permanganate, potassium iodide, potassium bromide were all laboratory grade chemicals. Non-ionic surfactant (Marlipal O13/80) was supplied by BDH. Non-ionic wetting agent, namely Texazym T and bacterial alpha-amylase, namely Texamyl BL, were supplied by Inotex Company.

2.3. Desizing

Gray cotton fabric was treated with an aqueous solution containing 0.1% (owf) Texamyl BL, 5 g/l, Na_2CO_3; 2 g/l, Texazym T using material to liquor ratio of 1:20. The desizing bath temperature was raised gradually to 70 °C and the desizing treatment continues for 60 min. The fabrics were washed thoroughly with boiling water to kill the enzymes and remove degraded starches then with cold water till neutralization. The fabric was tested for sizing agent removal by means of simple iodine test and the test showed the complete removal of sizing agent.

2.4. Scouring

Desized cotton fabric was treated with an aqueous solution containing 5 g/l, NaOH; 3 g/l, Na_2CO_3; 2 g/l, Marlipal O13/80 using material to liquor ratio of 1:20. The bath temperature was gradually increased to 95 °C and the scouring treatment continues for 60 min. The fabric was washed thoroughly with boiling water then with cold water. The efficiency of the scouring process was evaluated by carrying out the fabric wetability test.

2.5. Bleaching

Desized and scoured fabric was impregnated in a bleaching solution containing different concentrations of hydrogen peroxide and thiourea, together with 2 g/l non ionic wetting agent (Marlipal O13/80) for different time intervals (30–210 min) and different initial pH's which were adjusted by adding either sulfuric acid or sodium hydroxide. The bleaching reaction was carried out at varying temperatures (50–90 °C) using material to liquor ratio of 1:20. The extent of hydrogen peroxide decomposition was monitored via determining the amount of residual H_2O_2 throughout the bleaching reaction. At the end of reaction duration, the bleached samples were washed thoroughly with hot and then cold water and finally air dried.

2.6. Analysis and measurements

2.6.1. Fabric wetability

The wetability of the desized, scoured and bleached cotton samples was measured using the drop penetration test (Arbeitsgruppe, 1987).

2.6.2. % Loss in fabric weight

The percent loss in weight of cotton fabric after bleaching treatment was calculated according to the following equation

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

where W_1 is the weight of cotton fabric sample before bleaching treatment and W_2 is the weight of the sample after the treatment.

2.6.3. Determination of percent decomposed hydrogen peroxide

The percent decomposed hydrogen peroxide was calculated via quantitative estimation according to a reported method (Vogel, 1961).

2.6.4. Determination of degree of whiteness

The degree of whiteness of hydrogen peroxide bleached cotton fabric samples, expressed as whiteness index was measured using a Hunterlab Reflectometer (Model D25 M/L-2). The W_1 was calculated in terms of CIE Y (green) and (blue) reflectance components using the equation (ASTM Method E31373).

$$\text{W.I.} = \frac{4Z}{1.18} - 3Y$$

where Y and Z are the readings of the device.

2.6.5. Mechanical properties of the bleached fabrics

The tensile strength of bleached cotton fabrics was measured according to DIN 53857.

Table 1
Effect of pH of the bleaching medium on the physical and chemical properties of the bleached fabric.

pH	—C=O (m equiv./100 g)	—COOH (m equiv./100 g)	Tenacity (kg.f)
5	7	10	62
7	10.3	18.6	57
9	11.5	18.7	55
11	15.2	24.5	52
Gray fabric	5	4	67

[Hydrogen peroxide, 6 g/l; [thiourea], 1.5 g/l; bleaching temperature, 90 °C; material to liquor ratio, 1:20; duration, 210 min.

2.6.6. Determination of carboxyl content and carbonyl content

Reported methods were used for determination of carboxyl content (Mattisson & Legendre, 1952) and carbonyl content (Tihlérik & Pasteka, 1992).

3. Results and discussions

3.1. Activation of hydrogen peroxide by thiourea

Controlled decomposition of hydrogen peroxide in highly alkaline medium as well as reduction of bleaching time and temperature with the aid of certain amides have been extensively reported (Ibrahim, Sharaf, & Hashem, 2010; Hou et al., 2010). The hydrogen peroxide activation by thiourea is suggested to proceed via free radical mechanism that the first step is the formation of the intermediate hydrogen peroxide–thiourea complex which simultaneously decomposes to give hydroxyl free radicals (Eq. (1)).

$$(NH_2)_2CS + H_2O_2 \rightarrow (NH_2)_2CS \cdot H_2O_2 \rightarrow (NH_2)_2CS + 2HO^{\bullet} \quad (1)$$

These hydroxyl free radicals react with hydrogen peroxide to give perhydroxyl free radicals (Eq. (2)), which in turn react with hydrogen peroxide to give hydroxyl free radicals and nascent oxygen (Eq. (3)).

$$HO^{\bullet} + H_2O_2 \rightarrow H_2O + HO_2{}^{\bullet} \quad (2)$$

$$HO_2{}^{\bullet} + H_2O_2 \rightarrow H_2O + HO^{\bullet} + 2O \quad (3)$$

3.2. Bleaching

Different factors affecting the bleaching process of cotton fabric were systematically studied in details. These factors include pH of the bleaching reaction medium, concentrations of hydrogen peroxide and thiourea and temperature of the bleaching medium.

3.2.1. pH of the reaction medium

Fig. 1 shows the effect of pH of the reaction medium on the decomposition rate of hydrogen peroxide (Fig. 1A), the percent loss in fabric weight due to bleaching at different pHs (Fig. 1B) and the whiteness index (W.I.) of the resultant bleached fabrics (Fig. 1C). The reaction was carried out using 6 g/l of hydrogen peroxide and 1.5 g/l thiourea at 90 °C using a material to liquor ratio of 1:20 for 210 min. Table 1 shows the mechanical properties and chemical properties (carbonyl content and carboxyl content) of the bleached fabrics at different pHs. The given data indicate that the rate of decomposition of H_2O_2 increases significantly by raising the pH from 5 to 11 and that the pH has great effect on the time required for complete hydrogen peroxide decomposition, that at pH 5, it is necessary to prolong the bleaching duration up to 210 min to get 95% hydrogen peroxide decomposition. As the pH gets higher the bleaching duration required for complete hydrogen peroxide decomposition decreases to be 180 min, 150 min and 60 min for the pH values of 7, 9 and 11, respectively. It is clear from Fig. 1B that

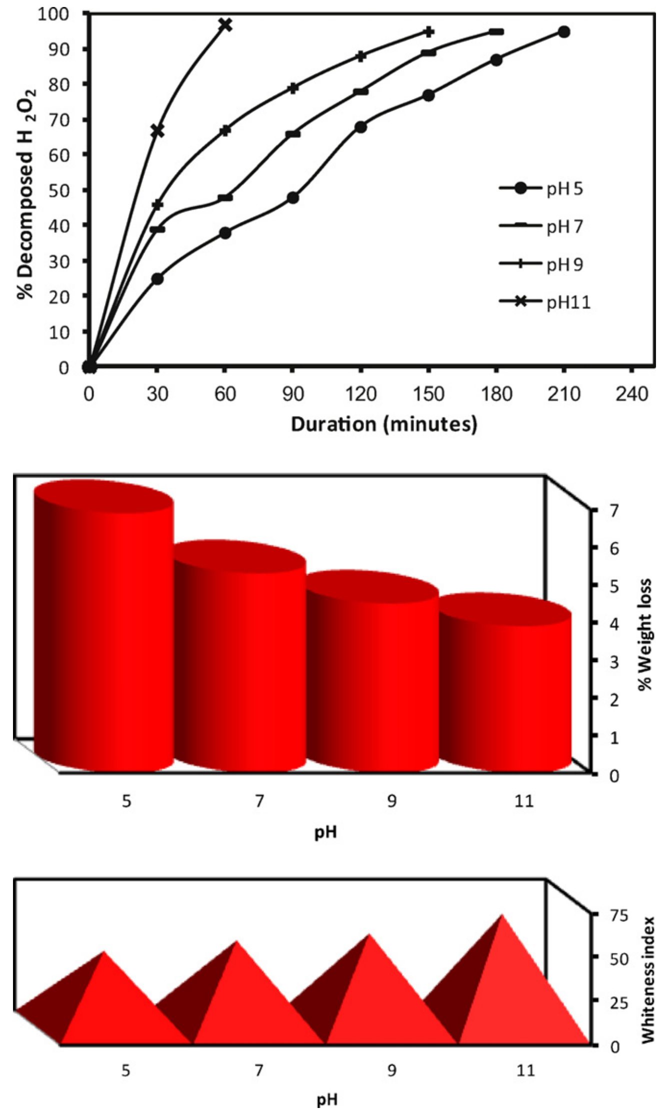

Fig. 1. (A) Effect of pH of the bleaching medium on the decomposition rate of hydrogen peroxide. (B) Effect of pH of the bleaching medium on the total percent loss in weight. (C) Effect of pH of the bleaching medium on the whiteness index.

the percent loss in fabric weight decreases as the pH of the bleaching medium got higher, that it was 6.9% at pH 5 and decreased to reach 3.9% at pH 11. Also, it is clear from Fig. 1C that the whiteness index of the bleached fabric increases as the pH of the bleaching medium got higher, that it was 44 at pH 5 and increased to reach 65 at pH 11. For the chemical properties carbonyl content and carboxyl content, it is noticed that they get higher as the pH gets higher and for the mechanical properties, they were found to improve, as the pH of the bleaching medium gets higher. It could be concluded from the data that maximum effect for bleaching and removal of the impurities is obtained at pH 11 and that the loss in tenacity is due to the removal of the noncellulosic materials from the cotton fiber structure. Also it could be concluded from the data that at pH 5 the low decomposition rate of hydrogen peroxide led to decrease in the mechanical properties without efficient bleaching action to the cellulosic fibers, which was reflected in poor whiteness index. As the pH gets higher, the decomposition rate increased and the bleaching system efficiency becomes better, which is reflected in better mechanical properties and improved whiteness index.

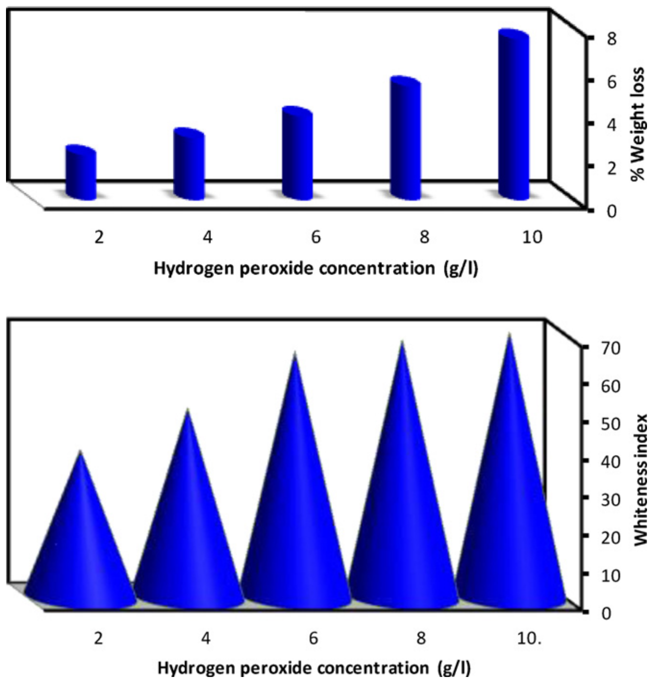

Fig. 2. (D) Effect of hydrogen peroxide concentration on the total percent loss in weight. (E) Effect of hydrogen peroxide concentration on the whiteness index.

3.2.2. Effect of hydrogen peroxide concentration

Fig. 2 shows the effect of the amount of hydrogen peroxide incorporated in the reaction medium on the percent loss in fabric weight (Fig. 2D) and whiteness index (Fig. 2E), while Table 2 presents the physical and chemical properties of the bleached cotton fabric. The bleaching process was carried out at 90 °C for total duration of 120 min, using fixed thiourea concentration of 6 g/l and the initial pH of the reaction medium was adjusted at pH 11. The obtained results reveal that increasing the amount of hydrogen peroxide incorporated in the reaction medium is accompanied by significant and continuous increase in the percent loss in fabric weight, W.I., carboxyl content and carbonyl content compared with the gray fabric. On the other hand, increasing hydrogen peroxide concentration leads to gradual decrement in the tenacity of the treated samples. These behaviors reflect the extent of removal of the noncellulosic materials from the cotton fiber structure as the concentration of hydrogen peroxide incorporated to the bleaching medium is increased. These noncellulosic materials are very important in the structure of cotton fibers and certain content of noncellulosic materials must remain together with the cellulose fibers to bind them and keep reasonable tensile properties. As is clear from the results, low concentrations of hydrogen peroxide incorporated to the bleaching medium (2 g/l and 4 g/l) leads to little removal of noncellulosics and accordingly good mechanical properties but the whiteness index is not satisfactory. On the other hand, high concentrations of hydrogen peroxide incorporated to the bleaching medium (8 g/l and 10 g/l) leads to high removal of noncellulosics and accordingly very bad mechanical properties and high whiteness index. It could be concluded from the obtained data that the optimum concentration of hydrogen peroxide which leads to acceptable whiteness index and retains good deal of the fabric's tensile strength is 6 g/l.

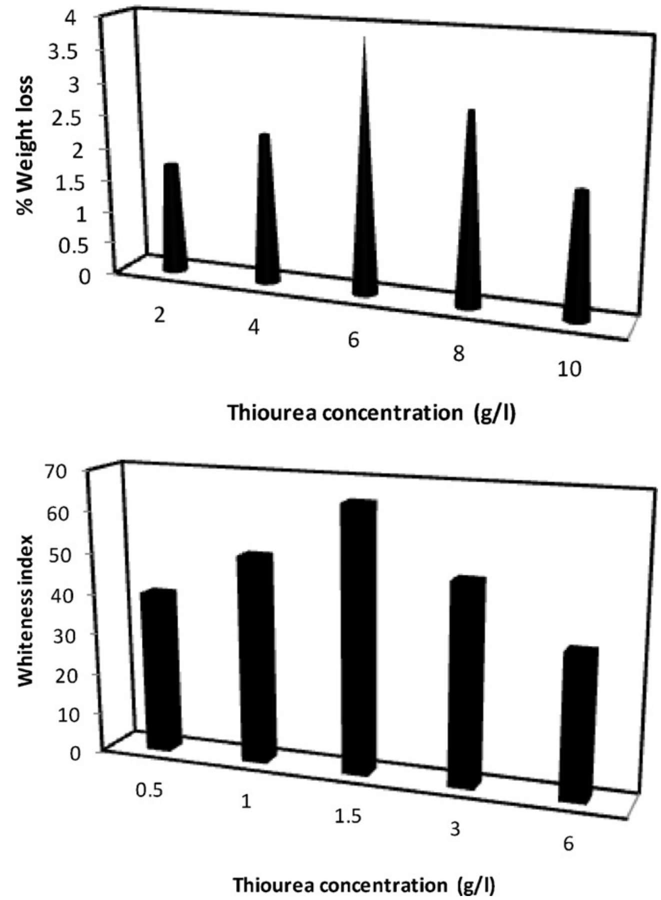

Fig. 3. (F) Effect of thiourea concentration on the total percent loss in weight. (G) Effect of thiourea concentration on the whiteness index.

3.2.3. Effect of thiourea concentration

Fig. 3 shows the effect of the amount of thiourea incorporated in the reaction medium on the percent loss in fabric weight (Fig. 3F) and whiteness index (Fig. 3G), while Table 3 presents the physical and chemical properties of the bleached cotton fabric. The bleaching process was carried out at 90 °C for total duration of 120 min, using fixed hydrogen peroxide concentration of 6 g/l and

Table 2
Effect of Hydrogen peroxide concentration on the physical and chemical properties of the bleached fabric.

[H_2O_2] (g/l)	—C=O (m equiv./100 g)	—COOH (m equiv./100 g)	Tenacity (kg.f)
2	8	11.2	60.4
4	10.3	18.5	56
6	15.2	24.5	52
8	17.3	30.2	33.3
10	22.7	39.9	24.3
Gray fabric	5	4	67

[Thiourea], 6 g/l; bleaching temperature, 90 °C; material to liquor ratio, 1:20; duration, 120 min; pH, 11.

Table 3
Effect of thiourea concentration on the physical and chemical properties of the bleached fabric.

[Thiourea] (g/l)	—C=O (m equiv./100 g)	—COOH (m equiv./100 g)	Tenacity (kg.f)
0.5	6.3	13.2	62.4
1	9.7	22.5	58
1.5	15.2	24.5	52
3	8.5	18.7	54.3
6	6.9	17.5	60.2
Gray fabric	5	4	67

[Hydrogen peroxide], 6 g/l; bleaching temperature, 90 °C; material to liquor ratio, 1:20; duration, 120 minutes; pH, 11.

Table 4
Effect of bleaching temperature on the mechanical Properties, carbonyl content and carboxyl content of the bleached fabrics.

Duration (min)	—C=O (m equiv./100 g)			—COOH (m equiv./100 g)			Tenacity (kg.f)		
	50 °C	70 °C	90 °C	50 °C	70 °C	90 °C	50 °C	70 °C	90 °C
30	5.5	9	11.5	10.3	15	20	65	60	55
60	7.3	11	15.2	12.6	17	24.5	63	58	52
90	9	12	15.2	14	18.5	24.5	62.1	58	52
120	10.3	13	15.2	16	19	24.5	61	56	52
Gray fabric		5			4			67	

[Hydrogen peroxide], 6 g/l; [thiourea], 1.5 g/l, 90 °C; material to liquor ratio, 1:20; duration, 120 min; pH, 11.

the initial pH of the reaction medium was adjusted at pH 11. The obtained results reveal that increasing the amount of thiourea incorporated in the reaction medium is accompanied by an increase in the percent loss in fabric weight, W.I., carboxyl content and carbonyl content compared with the gray fabric, up to thiourea concentration of 1.5 g/l. On the other hand, increasing thiourea concentration up to 1.5 g/l leads to gradual decrement in the tenacity of the treated samples. The reverse holds true when increasing the thiourea concentration over 1.5 g/l (3 g/l and 6 g/l). These behaviors reflect the extent of removal of the noncellulosic materials from the cotton fiber structure, that as the concentration of thiourea increases, more hydroxyl free radicals are formed due to the formation of hydrogen peroxide–thiourea complex and its simultaneous decomposition to give the hydroxyl free radicals (Eq. (1)). These hydroxyl free radicals, as illustrated by Eqs. (2) and (3) will react with hydrogen peroxide to give perhydroxyl free radicals (Eq. (2)), which in turn react with hydrogen peroxide to give hydroxyl free radicals and nascent oxygen (Eq. (3)). These active radical species are responsible for improvement of the bleaching effect as thiourea concentration increases up to 1.5 g/l. Upon increasing the concentration of thiourea over 1.5 g/l, large amounts of free radicals will be produced according to Eqs. (1)–(3) and this will lead to termination of these free radicals activity by recombination with each other (Eq. (4)):

$$HO^{\bullet} + HO^{\bullet} \rightarrow H_2O_2 \quad (4)$$

3.2.4. Effect of reaction temperature

The bleaching process was carried out at different temperatures 50 °C, 70 °C, and 90 °C using 6 g/l of hydrogen peroxide and 1.5 g/l thiourea, using material to liquor ratio of 1:20. The starting pH was adjusted to 11 and the bleaching continued for 120 min. Fig. 4 shows the rates of H_2O_2 decomposition (Fig. 4K), percent loss in fabric weight (Fig. 4L) and W.I. of the bleached fabrics (Fig. 4M). Table 4 shows the physical and chemical properties of the untreated and bleached fabrics. It is clear from the data that increasing the reaction temperature is accompanied by enhancement in the rate of H_2O_2 decomposition, marginal increase in the percent loss in fabric weight, improvement in the W.I. of the bleached samples, increment in the carboxyl content and carbonyl content and acceptable decrement in the tenacity.

For the enhancement in H_2O_2 decomposition, it is clear that the highest temperature 90 °C leads to fast and complete H_2O_2 decomposition within 60 min and this is reasonable duration for bleaching. The enhancement in hydrogen peroxide decomposition at high temperature is due to the favorable effect of temperature in increasing the kinetic energy of the bleaching species. At lower bleaching temperatures, 70 °C and 50 °C, the recorded decomposition percent after 120 min was 81.5% and 73%, respectively and this means that these low temperature need about 3 h to achieve complete hydrogen peroxide decomposition. For the enhancement in the physiochemical properties, it is due to the favorable effect of temperature in increasing the swelling of cotton fibers, giving rise to more accessibility and accordingly, the oxidizing species can penetrate easily to the bulk of the fiber and remove the noncellulosics and coloring matter, which leads to improved whiteness index and carboxyl and carbonyl contents

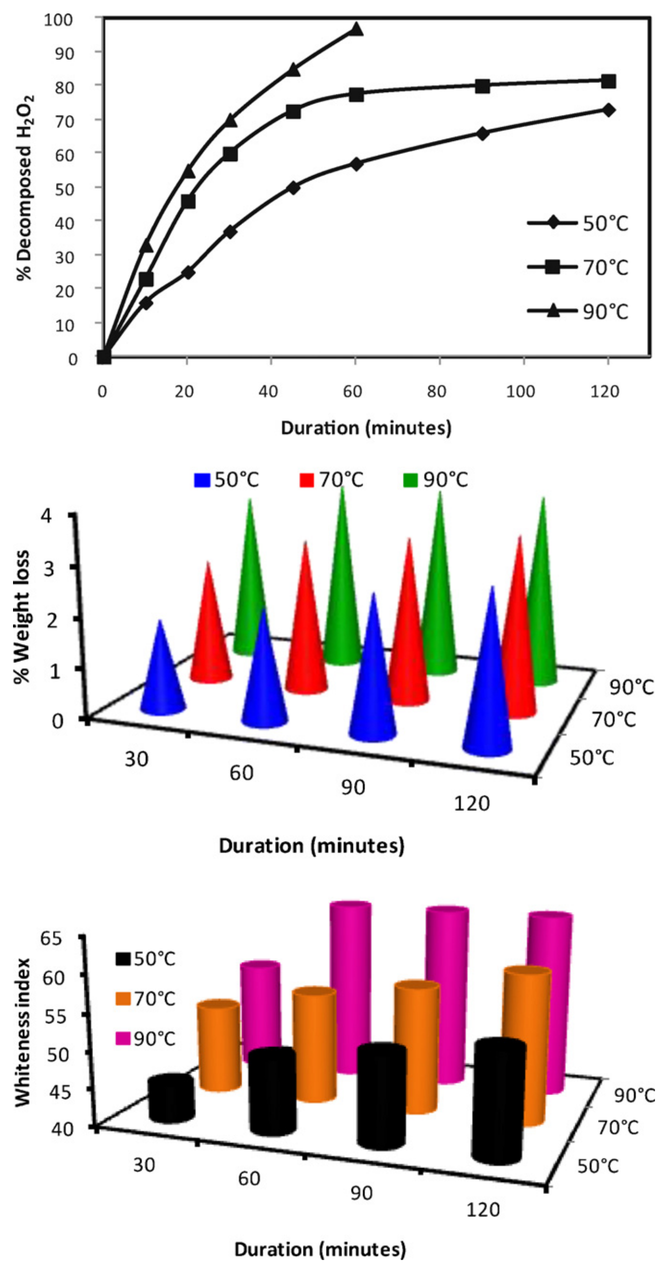

Fig. 4. (K) Effect of bleaching medium temperature on hydrogen peroxide decomposition rate. (L) Effect of bleaching medium temperature on the total percent loss in weight. (M) Effect of bleaching medium temperature on the whiteness index.

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