

Low-Temperature Bleaching of Cotton Cellulose using an Ultrasound-assisted Tetraacetylenediamine/Hydrogen Peroxide/Triethanolamine System

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This work investigated the efficiency of a tetraacetylenediamine/hydrogen peroxide/triethanolamine (TAED/H₂O₂/TEA) system for low-temperature bleaching of cotton fabric, with and without ultrasonic assistance. The low-temperature bleaching results were compared to those obtained from a conventional H₂O₂ bleaching system. The application of TAED/H₂O₂/TEA without ultrasonic assistance to grey cotton fabric with a whiteness index (WI) of 28 and tenacity of 40 CN/Tex resulted in full bleaching of the cotton fabric at 70 °C within 60 min. This low-temperature bleaching system produced a bleached cotton fabric with a WI of 69 and tenacity of 38 CN/Tex, while the conventional H₂O₂ bleaching system gave a bleached cotton fabric WI of 73 and tenacity of 32 CN/Tex. The application of ultrasound to a low-temperature bleaching system showed a reduction in the duration required for full bleaching to 30 min rather than 60 min and gave bleached fabrics of the same physiochemical properties.

Keywords: Cotton; Low-temperature bleaching; TAED; Hydrogen peroxide; Ultrasound

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INTRODUCTION

The main aim for the bleaching of cotton fabrics is to remove the color of naturally occurring pigments, primarily the flavonoids, from the cotton structure. These colorants make cotton fibers acquire an undesired yellowish to brown coloration; thus, their successful removal during bleaching makes cotton fabrics acquire a white appearance (Ibrahim *et al.* 2008; Abdel-Halim 2012a,b,c, 2013, 2014; Abdel-Halim and Al-Deyab 2013; Xu *et al.* 2013; Al-Hoqbani *et al.* 2014; Ali *et al.* 2014; Gonçalves *et al.* 2014; Abdel-halim *et al.* 2015; Benli and Bahtiyari 2015; Xu *et al.* 2015). The success of the overall bleaching process is directly reflected on the success of all the succeeding wet processes, such as functional finishing, printing, and dyeing (Abdel-Halim *et al.* 2010; Abdel-Halim *et al.* 2011; Long *et al.* 2012; Colleoni *et al.* 2013; Krishnaveni and Thambidurai 2013; Teli *et al.* 2013; Messaoud *et al.* 2014; Xie *et al.* 2014; Ferrero *et al.* 2015; Xu *et al.* 2015b; Qi *et al.* 2016). Because of its eco-friendly nature, the biodegradable bleaching agent hydrogen peroxide has recently begun to replace environmental polluting chlorine-based bleaching agents, which have been used traditionally for many decades. The main drawback in hydrogen peroxide bleaching is that it needs to be applied at high temperatures near the boiling point of water under strongly alkaline conditions. These processing conditions demand more energy consumption through attaining such a high temperature and moving the large volume of

wash water to remove the alkalinity after bleaching. Moreover, the radical species of hydrogen peroxide at such high temperature most likely causes severe oxidation to the cotton fibers, resulting in a decrease in the degree of polymerization. Thus, gentler processing conditions during bleaching such as milder temperatures and lower hydrogen peroxide concentration would be advantageous from both the material and energy consumption points of view.

Many researchers devoted great efforts to develop bleaching systems operating at a low temperature to replace the traditional hydrogen peroxide bleaching system to reduce the energy consumption and to preserve a reasonable amount of the bleached fabric's mechanical properties. One of the common hydrogen peroxide activators that is used for bleaching cellulosic fibers is N,N,N,N-tetraacetythylenediamine (TAED) (Scarborough and Mathews 2000; Cai *et al.* 2001). The chemical structure of TAED is shown in Fig. 1.

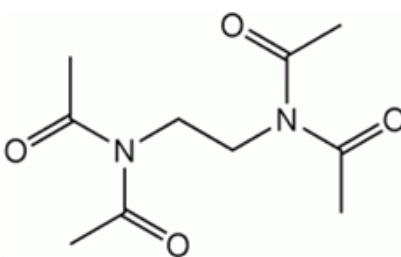


Fig. 1. Chemical structure of N,N,N,N-tetraacetythylenediamine (TAED)

In the area of textile wet processing, ultrasonic radiation is considered to be one of the most promising candidates to verify the concept of material and energy consumption. Technically, the frequency of the ultrasonic radiation is higher than the frequency of the audible range for human beings. The idea of utilizing ultrasound in textile wet processing is to apply ultrasonic energy coming from an ultrasonic source during the treatment bath process. This ultrasonic energy assists the textile wet processing by significantly accelerating the chemical and physical actions involved in the process through a cavitation phenomenon. When the generated sound waves transfer into the liquid bath, a local pressure wave is generated by the sonic vibration, and together with the hydrostatic pressure, leads to successive waves of rarefaction and compression. Microscopic bubbles form during the rarefaction wave and collapse during the following compression wave. The collapse of such a huge number of bubbles generates very hot spots (Liu *et al.* 2014; Macedo *et al.* 2014). These shocking waves, having high temperature and pressure, when localized, cause high shear force, which is enough to induce the breaking of chemical bonds. Ultrasonic treatment requires vigorous agitation, good dispersion and miscibility of chemicals, temporary emulsification of non-wetting auxiliaries, degassing, and effective generation of free radicals (Suslick 1988). The action of cavitation in heterogeneous system operations, like wet processing of textiles, is much higher than the action in processes involving homogeneous systems (Adewuyi 2001; Fakin *et al.* 2006; Munteanu *et al.* 2007). An ultrasonic-assisted bleaching technique could be successfully adopted into the traditional bleaching techniques of many papers and with these ultrasonic-assisted methods, researches could obtain a much greater whiteness index for the treated fabrics as compared to conventional bleaching processes with the absence of ultrasonic energy (Gonçalves *et al.* 2014a,b; Mistik and Yükselgöçlü 2005).

The present work aimed to develop a TAED/H₂O₂/TEA bleaching system for cotton fabrics, with and without ultrasonic assistance, and to compare its bleaching efficiency to that of the traditional hydrogen peroxide bleaching system. The performance of the suggested ultrasonic-assisted bleaching system was evaluated through measuring the percent loss in fabric weight, percent decomposition of hydrogen peroxide, carboxyl content, carbonyl content, degree of whiteness, and tenacity of the cotton fabrics before and after bleaching.

EXPERIMENTAL

Materials and Chemicals

Plain weave 100% cotton fabric was supplied by Misr Company for Spinning and Weaving, Mehalla El-Kobra, Egypt. The fabric specifications were as follows: fabric weight 150 g/m², weft 30 yarn/cm, and warp 36 yarn/cm. The fabric was tested for the presence/absence of any sizing agent and the test showed the presence of a starch-based sizing agent.

N,N,N,N-tetraacetylenediamine was supplied by Sigma-Aldrich, Bahrain. Hydrogen peroxide, sodium hydroxide, sodium carbonate, sulfuric acid, potassium bromide, potassium permanganate, potassium iodide, and triethanolamine were all laboratory grade chemicals. A non-ionic wetting agent (Marlipal O13/80) was supplied by BDH (Czech Republic). Bacterial α -amylase Texamyl BL and non-ionic surfactant Texazym T were supplied by Inotex Company, Czech Republic.

Methods

Fabric desizing

The raw cotton fabric was pretreated to remove the sizing agent prior to bleaching. This was done by immersing the cotton fabric in a desizing bath consisting of the α -amylase Texamyl BL, 0.1% (owf), Na₂CO₃, 5 g/L, and the non-ionic wetting agent Texazym T, 2 g/L, such that the ratio of cotton fabric to desizing liquor was 1:20. The temperature of the desizing liquor was raised to 70 °C, and the desizing bath was kept at this temperature for 60 min. At the end of the process, the fabrics were immersed in boiling water for two minutes to kill the enzyme activity as well as to remove the degraded fragments of the sizing agent. The hot washing was followed by cold washing until neutrality, and then the fabrics were air-dried. The treated fabrics were tested to confirm the removal of the sizing agent by a simple iodine test, which showed complete desizing.

Fabric scouring

Prior to bleaching, the desized cotton fabric was scoured with the aim of improving its wettability. This was done by immersing the cotton fabric in a scouring bath consisting of 5 g/L sodium hydroxide, 3 g/L sodium carbonate, and the non-ionic wetting agent of 2 g/L Marlipal O13/80, such that the ratio of desized cotton fabric to scouring liquor was 1:20. The temperature of the scouring liquor was raised to 95 °C, and the scouring bath was kept at this temperature for 60 min. At the end of the process, the fabrics were washed several times with boiling water until neutrality followed by washing with cold water. The scouring efficiency was evaluated by the wettability test (Arbeitsgruppe 1987) and the test showed a wettability time of less than one second.

Fabric bleaching

The scoured cotton fabrics were immersed in bleaching liquors containing different concentrations of H₂O₂ and TAED, along with TEA and the non-ionic wetting agent (Marlipal O13/80 at 2 g/L). The bleaching reaction was run in an ultrasonic water bath Model FS110 Fisher Scientific Ultrasonic Cleaner (Fisher Scientific UK Ltd., UK) with a frequency of 42 kHz and maximum power of 495 W. The bleaching temperature was adjusted to 70 °C. Bleaching was carried out at various temperatures (50 to 90 °C). The pH of the bleaching liquor was varied in the range of 5 to 12, keeping the ratio of cotton fabric to bleaching liquor constant at 1:20. The progress of the bleaching reaction was determined by measuring the hydrogen peroxide percent decomposition, through determining the concentration of residual hydrogen peroxide throughout the course of the reaction (Vogel 1961). At the end of the bleaching reaction, the treated samples were washed several times with hot water until neutrality, followed by a cold wash and air-drying. A sample of the same cotton fabric was bleached by the traditional hydrogen peroxide bleaching process just for the purpose of comparison. The traditional bleaching was carried out in a bleaching liquor containing 5 g/L H₂O₂ together with 2.5 g/L NaOH, a 1 g/L chelating agent, and the 2 g/L non-ionic wetting agent. The ratio of cotton fabric to bleaching liquor was fixed at 1:20, the bleaching system temperature was raised to 95 °C, and the system was kept at this temperature for 45 min. At the end of the traditional bleaching treatment, the sample was washed several times with hot water until neutrality, followed by a cold wash and air-drying.

Tests, Analysis, and Measurements

The percent weight loss of the bleached cotton fabric was calculated according to Eq. 1:

$$\% \text{weight loss} = (W_1 - W_2) \times 100 / W_1 \quad (1)$$

where W_1 is the weight of the cotton fabric before the treatment and W_2 is the weight of the cotton fabric after treatment. The whiteness index (WI) of the scoured cotton fabrics as well as the hydrogen peroxide-bleached cotton fabrics was measured using a Hunterlab Reflectometer (Model D25 M/L-2). The WI was calculated in terms of CIE Y (green) and reflectance components (blue) using Eq. 2 (ASTM Method E31373 (1993)):

$$\text{Whiteness Index} = (4Z/1.18) - 3Y \quad (2)$$

where Y is the relative luminance (brightness) of green color and Z is the value quasi-equal to the blue color stimulation. The tensile strength of the scoured cotton fabrics as well as the hydrogen peroxide-bleached cotton fabrics was measured according to DIN 53857 (1979). The following reported analysis methods were used for measuring the carboxyl content (Mattisson and Legendre 1952) and the carbonyl content (Tihlířik and Pateka 1992) of the cotton fabrics.

RESULTS AND DISCUSSION

Mechanistic Aspects of TAED/H₂O₂/TEA Bleaching System

TAED reacts with hydrogen peroxide in a perhydrolysis reaction as illustrated in Eq. 3 (Davies and Deary 1991). The mode of action of a TAED/H₂O₂ bleaching system is that one mole of TAED reacts with two moles of H₂O₂ and the net result is the formation

of one mole of diacetylenediamine (DAED) and two moles of active peracetic acid (PAA) (Davies and Deary 1991). The key to the perhydrolysis reaction is attributed to the perhydroxyl anion (HOO^-); hydrogen peroxide liberates these perhydroxyl anions, which undergo the perhydrolysis reaction with TAED to form peracetic acid molecules in situ.

From a kinetic point of view, the reactivity of peracid is much higher than that of hydrogen peroxide due to the low stability of peracids. This allows the bleaching of cotton fibers with peracids to be achieved at temperatures much lower than the temperature needed to bleach the same cotton fabrics using hydrogen peroxide (Hou *et al.* 2010).



The net result of the perhydrolysis reaction is the production of peracetic acid, which decomposes in the bleaching medium according to Eq. 4 to give acetic acid and nascent oxygen. The continuous release of acetic acid due to peracetic acid decomposition leads to a decrease in pH of the bleaching medium. This decrease in bleaching medium pH results in a decrease in the rate of the TAED perhydrolysis reaction (Eq. 3), which requires a faint alkaline to near neutral medium to proceed and produce peracetic acid. In the present work, to avoid such an undesired drop in bleaching medium pH, triethanolamine (TEA) was added as a buffering agent in order to neutralize the produced acetic acid and drive the perhydrolysis reaction to completion for achieving maximum performance of the H_2O_2 /TAED cotton fabric bleaching system.

Effect of Reaction Parameters on Bleaching Efficiency

pH of the bleaching medium

Table 1 demonstrates the effect of varying the bleaching medium pH on the physiochemical properties, percent loss in fabric weight, whiteness index, tenacity, carboxyl content, and carbonyl content of the bleached cotton fabrics, in the absence of ultrasonic energy. For comparison, the same physiochemical properties for the conventionally bleached cotton fabric as well as the raw cotton fabric were measured and are listed in Table 1. The TAED/ H_2O_2 bleaching system clearly showed optimum performance and gave the best physiochemical properties at a pH of 8. The conventional bleaching system resulted in the cotton fabrics having a higher WI (73), but this was found to be on the count of greater loss in fabric tenacity (32 CN/Tex).

The high loss in fabric weight under acidic conditions (pH 3 to 4) is most likely due to severe hydrolysis of the cotton fibers rather than oxidative bleaching of the fibers. This assumption is strongly supported by the high loss in fabric tenacity (15.5 to 18.5 CN/Tex), which is accompanied by a very low WI (28 to 30).

The conventionally bleached fabric showed a high level of loss in fabric weight and tenacity, which indicates the negative effect of such aggressive bleaching conditions on the fabric's mechanical properties, although it resulted in a higher WI of 73. Results showed no increase in the carboxyl nor the carbonyl content of the cotton fabric in the acidic range. This again enhances the conclusion that the losses in fabric weight and tenacity in the acidic range are due to fiber hydrolysis rather than oxidative bleaching. On the other hand, a clear improvement in the carboxyl and carbonyl contents was observed in the near neutral/faint alkaline pH range of 6 to 8. Little increase in the carboxyl and carbonyl contents was observed in the alkaline pH range of 11 to 12. These findings

suggest that the maximum performance of the perhydrolysis reaction of TAED is attained under near neutral/faint alkaline conditions. The lack of performance of TAED/H₂O₂ in the alkaline pH range (pH 10 to pH 12) is most likely attributable to the hydrolysis of TAED in alkaline to acetic acid rather than its perhydrolysis to peracetic acid (Shao *et al.* 2010).

Table 1. Effect of Bleaching Medium pH on the Physiochemical Properties of the Cotton Fabric

pH	3.1	4.1	5.1	6.2	7.4	8.0	9.2	10.1	11.0	12.3	Conven- tionally Bleached Cotton	Raw Cotton
Loss in Weight (%)	6	5.2	4.8	4.3	4.1	4	2.6	1.7	0.9	0.4	6.5	↓
WI	28	30	45	60	65	70	62	50	30	30	73	28
Tenacity (CN/Tex)	15	18	22	35	37	35	39	39	39	39	32	40
COOH (m.eq/100 g)	6	6	7	11	14	16	14	10	8	6	24	6
C=O (m.eq/100 g)	3	3	3	5	7	9	7	4	4	3	15	3

*[TAED], 3.5 g/L; [H₂O₂], 5 g/L; temperature, 70 °C; duration, 60 min

Figure 2 shows the effect of the bleaching medium pH on the rate of hydrogen peroxide decomposition during the bleaching treatment. The data presented in Fig. 2 show that the rate of hydrogen peroxide decomposition was very low in the strongly acidic medium (pH 3), almost 6% after 60 min (Fig. 2); the recorded WI for the bleached cotton fabric at a pH of 3 was the same as that of the grey cotton fabric with a WI of 28 (Table 1). This helps confirm the fact that the performance of TAED perhydrolysis is almost zero in the strong acid media. As the pH of the bleaching medium increased to 5.12, the performance of TAED perhydrolysis increased to some extent. The 20% hydrogen peroxide decomposition (Fig. 2) and the medium WI for the bleached cotton fabric of 45 (Table 1) support this observation.

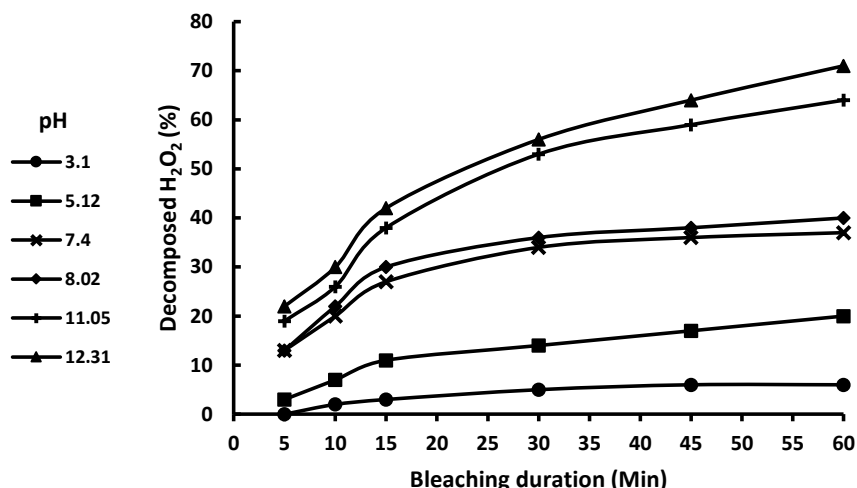


Fig. 2. Effect of bleaching medium pH on H₂O₂ decomposition

The maximum hydrogen peroxide decomposition rate of 40% (Fig. 2) and maximum WI value (Table 1) were obtained when bleaching the cotton fabric with TAED/H₂O₂/TEA at a pH of 8; this implies that the most efficient performance of TAED perhydrolysis to peracetic acid, through its reaction with H₂O₂, is obtained at this pH value.

Effect of TAED concentration

TAED is the precursor of the species responsible for cotton fabric bleaching, namely peracetic acid. Assuming the presence of an excess amount of hydrogen peroxide in the bleaching medium as is in this study (5 g/L) and the adequate system pH suitable for the perhydrolysis reaction (pH 8), the generation of peracetic acid as a result of the perhydrolysis reaction is expected to depend on the availability of TAED molecules in the bleaching medium. It should be noted that the excess amount of H₂O₂ was just added to drive the perhydrolysis reaction of TAED in the direction of peracetic acid formation, but H₂O₂ itself does not impart on the bleaching effect of the cotton fabric at this low temperature and faint alkaline pH, as hydrogen peroxide is known to give satisfactory bleaching effect to cotton fabric in a strong alkaline medium near the boiling temperature.

The WI data are presented in Table 2, which shows that the WI value increased gradually and continuously upon increasing TAED concentration until reaching 72 with utilizing 4.5 g/L TAED and greater. Considering the results obtained for the percent loss in fabric weight and fabric tenacity, the same trend as the case of WI was observed. From a practical and economic point of view, it is concluded that 2.5 g/L is an optimum concentration of TAED, which can give a suitable amount of peracetic acid for bleaching cotton fabric to give a satisfactory WI of 69 and a reasonable loss in fabric weight of 3.2% while maintaining a good deal of the fabric's tenacity at 38 CN/Tex. Table 2 shows that the utilization of TAED concentrations higher than 2.5 g/L is not desired and leads to negligible improvement in the WI, and higher loss in fabric weight and tenacity. This higher loss in fabric weight and tenacity is due to the severe oxidation of cotton fabric due to the generation of large amounts of peracetic acid at high TAED concentrations, and this is evident by the high carboxyl and carbonyl contents due to this severe oxidation at TAED concentrations of 3 g/L and greater.

Table 2. Effect of TAED Concentration on the Physiochemical Properties of Cotton Fabric

TAED (g/L)	0.5	1	1.5	2	2.5	3	3.5	4	4.5	5	↓ Raw Cotton
Loss in Weight (%)	0.3	1.2	2.2	2.9	3.2	3.6	4	4.4	4.9	6	
WI	33	40	52	65	69	69	70	71	72	72	28
Tenacity (CN/Tex)	40	39	39	38	38	36	35	33	33	32	40
COOH (m.eq/100 g)	7	7.8	9.4	11.5	14	15	16	18	20	21	6
C=O (m.eq/100 g)	3.5	3.9	4.9	6.3	7	8	9	9.9	11.1	12.5	3

* [H₂O₂], 5 g/L; pH, 8; temperature, 70 °C; duration, 60 min

Figure 3 shows the results recorded for hydrogen peroxide decomposition upon the utilization of different concentrations of TAED in the bleaching of the cotton fabrics. For a given TAED concentration, there was a gradual increase in the percent-decomposed H₂O₂ as the bleaching duration increased. Regarding the percent decomposition of H₂O₂

at the end of the bleaching duration, it was found that it is related proportionally to the initial TAED concentration incorporated in the bleaching medium.

Based on the best WI/tenacity combination, it is concluded that a TAED concentration of 2.5 g/L is the optimum concentration. As shown in Fig. 3, this TAED concentration let to the decomposition of one third of the initial hydrogen peroxide concentration, which means that this TAED concentration requires only 33% of the already added 5 g/L H₂O₂ to achieve complete perhydrolysis to peracetic acid; this means that 1.65 g/L H₂O₂ (or 1.75 g/L H₂O₂) would be enough.

So far, it is concluded that 2.5 g/L TAED and 1.75 g/L H₂O₂ are optimum concentrations for producing bleached cotton fabrics having satisfactory WI and tenacity.

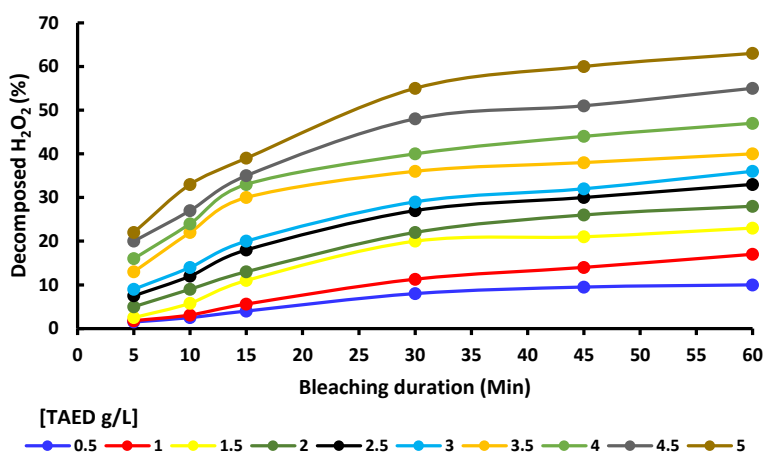


Fig. 3. Effect of TAED concentration on H₂O₂ decomposition

Temperature effect in reaction

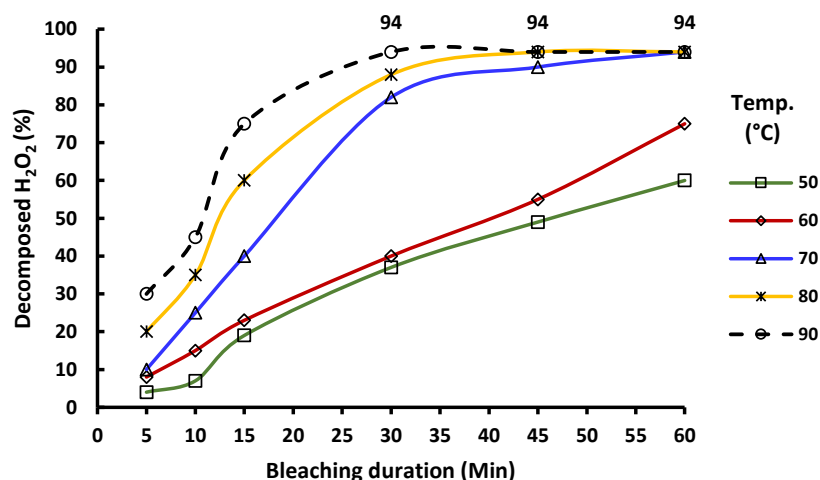
As shown by Table 3, bleaching the cotton fabric at 50 °C resulted in a WI of 50, loss in fabric weight of 1.2%, tenacity of 38.5 CN/Text, and carboxyl and carbonyl contents of 10 m.eq/100 g and 5 m.eq/100 g, respectively. Raising the bleaching temperature to 60 °C then to 70 °C resulted in corresponding improvement in the physiochemical properties of the bleached fabrics. Further increase in the bleaching temperature above 70 °C did not cause any further improvement in the physiochemical properties of the bleached cotton fabric.

Figure 4 shows that carrying out the bleaching reaction at 50 °C for 60 min resulted in a decomposition of only 60% of the amount of H₂O₂ incorporated initially to the bleaching liquor. The percent-decomposed H₂O₂ reached only 75% upon raising the bleaching temperature to 60 °C. The low percent H₂O₂ decomposition at low temperatures explains the bad physiochemical properties obtained when bleaching cotton fabrics at these temperatures. The low percent H₂O₂ decomposition had a negative effect on the perhydrolysis reaction of TAED to give peracetic acid and the poor peracid generation resulted in a poor bleaching effect on the cotton fabrics. The bleaching temperature of 70 °C was the optimum temperature, which resulted in 94% H₂O₂ decomposition and accordingly led to very effective perhydrolysis of TAED to give enough peracetic acid to impart a good bleaching effect on the cotton fabric. It is also noticed from Fig. 4 that raising the bleaching temperature to 80 °C then to 90 °C led to 94% H₂O₂ decomposition in less than 60 min, but it did not lead to further improvement in physiochemical properties.

Table 3. Effect of Bleaching Temperature on the Physiochemical Properties of Cotton Fabric

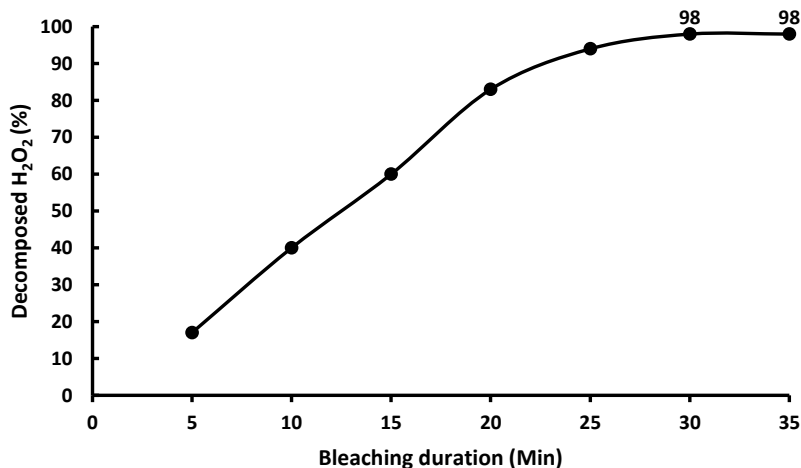
Temperature (°C)	50	60	70	80	90	↓ Raw Cotton
Loss in Weight (%)	1.2	2.3	3.2	3.2	3.2	
WI	50	55	69	70	70	28
Tenacity (CN/Tex)	38.5	38.2	38	38	38	40
COOH (m.eq/100 g)	10	12	14	14	14	6
C=O (m.eq/100 g)	5	6	7	7	7	3

* [TAED], 2.5 g/L; [H₂O₂], 1.75 g/L; pH, 8; duration, 60 min

**Fig. 4.** Effect of bleaching temperature on H₂O₂ decomposition

Ultrasound-assisted TAED/H₂O₂/TEA bleaching system

The optimum conditions obtained from the foregoing study were applied again in bleaching cotton fabric, but this time with the assistance of ultrasonic energy to examine its effect on the bleaching efficiency of the TAED/H₂O₂/TEA system. According to the data presented in Fig. 5, the hydrogen peroxide decomposition percent reached 98% after 30 min and did not increase with prolonged bleaching duration for an additional five minutes.

**Fig. 5.** Effect of ultrasonic assistance on H₂O₂ decomposition

The characterization showed that the cotton fabric bleached via the ultrasonic-assisted TAED/H₂O₂/TEA bleaching system had a WI of 72, tenacity of 37.5 CN/Tex, and a weight loss of 3.3%. These physiochemical properties are very comparable to those of cotton fabric bleached by the same bleaching liquor for a duration of 60 min without the assistance of ultrasonic energy. This means that the application of ultrasonic energy to a bleaching process could help to reduce the bleaching time by 50% and produce bleached fabrics having the same physiochemical properties.

CONCLUSIONS

1. A tetraacetythylenediamine/hydrogen peroxide/triethanolamine (TAED/H₂O₂/TEA) system was used in low-temperature bleaching of cotton fabric, with and without ultrasonic assistance.
2. The application of TAED/H₂O₂/TEA without ultrasonic assistance to grey cotton fabric having a whiteness index (WI) of 28 and tenacity of 40 CN/Tex resulted in full bleaching of cotton fabric at 70 °C within 60 min to give bleached cotton fabric with a WI of 69 and tenacity of 38 CN/Tex.
3. The application of ultrasound to the low-temperature bleaching system could reduce the duration required for full bleaching from 60 to 30 min to give bleached fabrics having the same physiochemical properties.

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