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Study on H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism related to hydroxyl radical with a fluorescent probe

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ABSTRACT

The generation of hydroxyl radical (HO•) in H_2O_2/TAED and H_2O_2/TBCC systems for cotton fabric bleaching was proved and detected with a novel fluorescent probe benzenepentacarboxylic acid (BA). Effect of HO• generation on the cotton fabric bleaching performances was further discussed. The results show that HO• yield in H_2O_2/TAED and H_2O_2/TBCC systems was respectively about 11 and 15 times higher than that in H_2O_2 system without activators under the same alkali condition. Meanwhile, TAED and TBCC apparently promoted fabric whiteness and H_2O_2 decomposition rate. As the addition of HO• scavenger dimethylsulfoxide (DMSO), fabric whiteness decreased while tensile strength increased. It proves that HO• played a significant role in H_2O_2/TAED and H_2O_2/TBCC bleaching. Two main bleaching routes were suggested. The present work brought new insight into H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism. It is quite instructive to develop efficient and ecological bleaching processes.

1. Introduction

Pretreatment of cotton fabric is required to remove size and impurities including pigments, pectin, waxiness and also cotton shell, etc., to facilitate subsequent dyeing and finishing process. Conventional pretreatment contains three separate courses of desizing, scouring and bleaching. The long duration process has been gradually substituted by one bath pretreatment of desizing, scouring and bleaching, which is commonly called H_2O_2-alkali one bath treatment. Although shorten pretreatment duration greatly, H_2O_2 bleaching consumes large amounts of energy for near boiling temperatures to obtain excellent fabric performances. In order to save energy, great efforts have been made to explore low temperature bleaching with activators. Tetra-acetylethylenediamine (TAED) and N-[4-(triethylammoniomethyl)-benzoyl]-caprolactam chloride (TBCC) are promising activators to improve fabric bleaching performances at lower temperature (Cai, Evans, & Smith, 2001; Hou, Zhang, & Zhou, 2010; Lim, Hinks, & Hauser, 2004; Lim, Lee, Hinks, & Hauser, 2005; Shao, Huang, Wang, & Liu, 2010; Willey et al., 1997; Xu, Shamey, & Hinks, 2010; Xu, Long, Du, & Fu, 2013). Further research of H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism will be instructive for industrially application of TAED and TBCC to H_2O_2 activated bleaching in large-scale.

The generally considered H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism is a perhydrolysis process (James, & MacKirdy, 1990; Kott, Willey, & Miracle, 1996; Lim et al., 2004; Sain, Daneault, & Parenteau, 1997). TAED and TBCC react with HOO^- ionized in alkali H_2O_2 solution to form organic peroxide acid (RCOOOH) with higher redox potential and stronger oxidizing properties than HOO^- (Castiglione, Baggioli, Citterio, Mele, & Raos, 2012; Proska, 1952). Thus, RCOOOH would directly interact with natural pigments to improve fabric whiteness at lower temperatures. However, RCOOOH is probably unstable, especially peroxyacetic acid (PAA) formed by reaction between TAED and H_2O_2 is quite unstable under dilute solution (Coucharriere, Mortha, Lachenal, Briois, & Larnicol, 2002; Ebrahimi, Kolehmainen, Oinas, Hietapelto, & Turunen, 2011). H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism involved in free radicals still remains ambiguous. Detailed research about whether relevant free radicals are further generated after the formation of RCOOOH has not been conducted.

Free radicals have been frequently studied in physiological and pathological processes of biological molecules, such as DNA, protein and fat (Lagouge & Larsson, 2013; Miral & Pawel, 2012). In textile industry, various processes especially H_2O_2 bleaching also involve in free radicals, though the bleaching mechanism is still under debate (Dannacher & Schlenker, 1996). Among the free radicals

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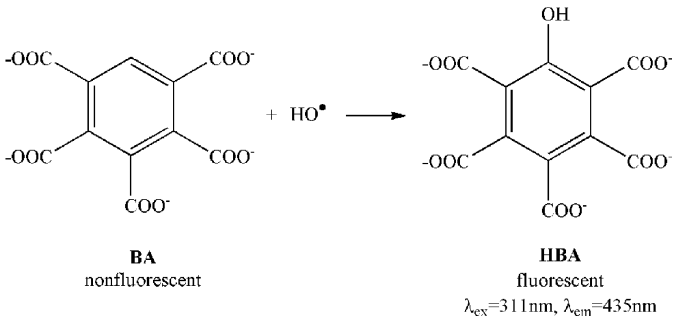


Scheme 1. The process of HO• addition to BA.

probably generated in H_2O_2 bleaching, HO• possesses the strongest oxidizing ability owing to its high standard redox potential in aqueous solution (Klaning, Sehested, & Holcman, 1985; Koppenol & Liebman, 1984). HO• can induce chain reaction and interact with various organic compounds primarily by means of electronic transfer, electrophilic addition, dehydrogenation reaction (Ek, Gierer, & Jansbo, 1989). Hence, the presence and generation of HO• are prior to investigate H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism in the paper.

In the present study, benzenepentacarboxylic acid (BA), as a specific and reproducible fluorescent probe of HO• developed by our lab (Zhang, Si, &Yan, 2013), was utilized to prove and detect the generation of HO• in H_2O_2/TAED and H_2O_2/TBCC alkali systems with and without HO• scavenger dimethylsulfoxide (DMSO). The present probe has only one reaction site for HO• addition that results in the formation of a single fluorescent product, hydroxybenzenepentacarboxylic acid (HBA) (Scheme 1), which has been demonstrated by ESI-MS analysis system in our previous work. The generation of the single pure fluorescent product by BA made the present probe sensitive, accurate and reproducible in detection of HO•. Effect of HO• generation on the whiteness and tensile strength of the cotton fabrics was further studied, to discuss the role of HO• plays in H_2O_2/TAED and H_2O_2/TBCC bleaching.

2. Experimental

2.1. Apparatus and materials

2.1.1. Apparatus

The fluorescence intensity was measured on Hitachi F-7000 spectrophotofluorometer with a xenon lamp and a quartz cuvette (1.0 cm optical path) as the container. The spectrometer slit was set for 5.0 nm band-pass, with the PMT voltage 400 V.

All bleaching processes were carried out by RY-25012 room temperature oscillation dyeing machine (Shanghai Long Ling Electronic Technology Co., Ltd.).

2.1.2. Materials

Benzenepentacarboxylic acid (BA) was provided by TCI (purity > 98%, Japan). Sodium benzenepentacarboxylate stock solution was prepared from benzenepentacarboxylic acid through neutralization with NaOH, and diluted to prepare relative working solutions.

Gray fabric used was 100% woven twill cotton. TBCC was synthesized and purified according to the patent reported previously by Willey et al. (1997). TAED (purity 99%), DMSO and H_2O_2 (30 wt% in H_2O) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). All other chemicals were of analytical grade. Distilled water was used throughout except the washing process of fabrics.

2.2. Experimental procedure

2.2.1. Detection of HO•

Molarity of fluorescent substances near or lower than 10^{-3} mol/L is acceptable to detect fluorescence intensity by the spectrophotofluorometer, or the fluorescent substances need to be diluted before determination. Reagents used in detection of HO• contained BA (400 μmol/L), NaOH (4 mmol/L), TAED (400 μmol/L), TBCC (400 μmol/L), H_2O_2 (220 μmol/L), and reacted for 60 min at a certain temperature. Then the reactions were promptly cooled and quenched. A certain amount of working solution was placed in the four-pass optical quartz cuvette, and the fluorescence intensity was measured at maximum excitation wavelength (311 nm) to characterize the concentration of HO•.

2.2.2. Scavenging of HO•

DMSO was used as HO• scavenger to decrease HO• concentration and clarify its effect on fabric performances during bleaching processes. Before that, HO• scavenging efficiency of DMSO (2.4 mmol/L) was tested at 70 °C, other reaction regents and test conditions are the same as that used in detection of HO•.

2.2.3. Fabric bleaching

Gray cotton fabric samples were impregnated with distilled water for a while to avoid the influence of transitional metal ions which were probably carried on the surface of fabric during previous processes, then air dried under ambient temperature. The process did little to fabric performances. Impregnation bleaching method was used and performed at a certain temperature for 60 min with a liquor ratio of 1:20. Each bleaching solution contained NaOH (1.5 g/L), 30% H_2O_2 (5 g/L), TAED 2 g/L or TBCC 4 g/L. After reactions, the bleached fabric was rinsed thoroughly in copious amounts of tap water, then air dried under ambient temperature.

Corresponding to scavenging of HO• in Section 2.2.2, varying amounts of DMSO (1–4 g/L) was added into each fabric bleaching bath to evaluate effects of HO• generation on fabric bleaching performances. Other reaction conditions are the same as that used in fabric bleaching without DMSO.

2.3. Measurement

CIE Whiteness Index (WI) of cotton fabric samples was measured by Datacolor 65° color matching instrument (USA) according to ISO 105-J02: 1997 standard. Each sample was measured for five times in different positions to give an average value.

Tensile strength of cotton fabric samples was determined by H10KS fabric strength tester and the test method referred to ISO 13934.1, 1994 standard.

H_2O_2 decomposition rate (DR) was measured by a certain concentration of potassium permanganate solution and calculated by the following equation.

$$\mathrm{DR} = \frac{V_0 - V_1}{V_0} \times 100\% \quad (1)$$

where V_0 is the volume of potassium permanganate solution consumed by titration of 10 mL bleaching liquid before reaction and V_1 is the volume of potassium permanganate solution consumed by titration of 10 mL bleaching liquid after reaction.

3. Results and discussion

3.1. Detection of HO•

HO• is the strongest single electron oxidant existed in aqueous solution, leading to its poor selectivity in rapid reaction with

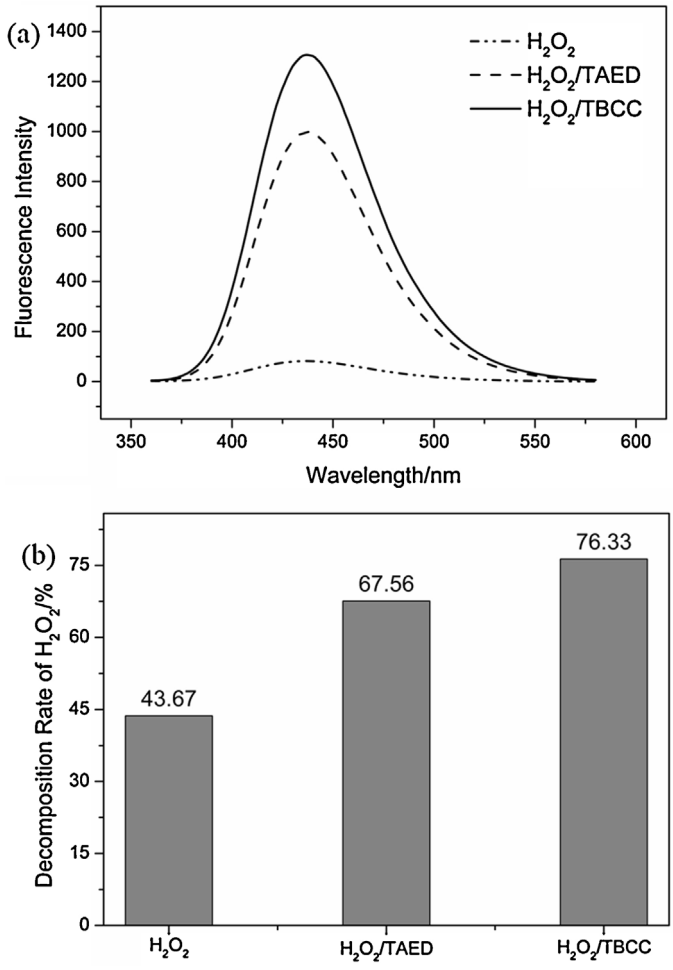


Fig. 1. HO• generation and H_2O_2 decomposition rate in each system. (a) HO• generation, (b) H_2O_2 decomposition rate. T = 70 °C.

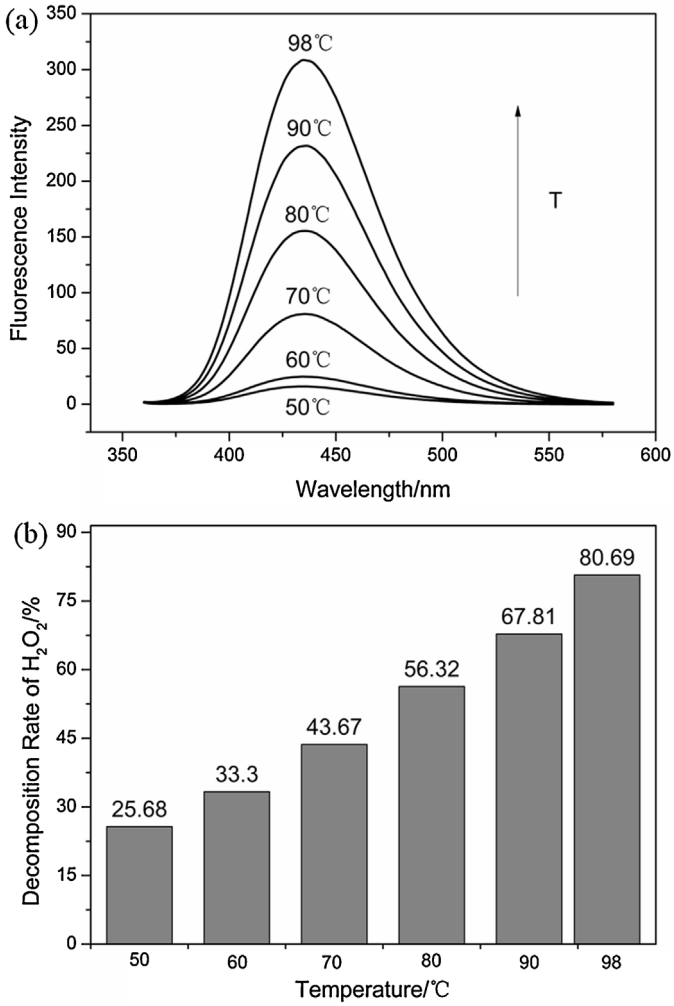


Fig. 2. Effect of temperature on HO• generation and H_2O_2 decomposition rate in H_2O_2 system without activators. (a) HO• generation, (b) H_2O_2 decomposition rate. T = 50–98 °C.

various organic substances (Hammel, Kapich, Jensen, & Ryan, 2002). Hence, detection of HO• generation is of great importance to further understand H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism. H_2O_2 decomposition rate was also considered to discuss its relationship with HO• generation.

3.1.1. *Detection of HO• generated in H_2O_2/TAED and H_2O_2/TBCC systems*

HO• generation and H_2O_2 decomposition rate in H_2O_2/TAED and H_2O_2/TBCC systems are given in Fig. 1a and b. Compared to H_2O_2 system without activators, both TAED and TBCC enhanced HO• generation significantly under the same alkali condition, respectively with 11 and 15 times increase of fluorescence intensity at 70 °C for 60 min. At the same time, TAED and TBCC also obviously accelerated H_2O_2 decomposition. It can be explained that TAED and TBCC reacted with HOO^- ionized in alkali H_2O_2 solution to form RCOOOH, which consequently promoted H_2O_2 decomposition and enormous HO• generation. Besides, HO• generation was closely linked to H_2O_2 decomposition rate.

3.1.2. *Detection of HO• generated in H_2O_2 system without activators*

Temperature is a significant variable for H_2O_2 bleaching. For the purpose of better comparison with H_2O_2/TAED and H_2O_2/TBCC systems, HO• generation and H_2O_2 decomposition rate at different temperatures (50–98 °C) in H_2O_2 system without activators are discussed. As shown in Fig. 2a and b, little HO• was produced between 50 °C and 60 °C because of low H_2O_2 decomposition rate. Then HO• generation and H_2O_2 decomposition rate increased evidently as the temperature rises from 70 °C to 98 °C. Interestingly, even HO• yield in H_2O_2 system without activators at 98 °C was still much less than that in H_2O_2/TAED and H_2O_2/TBCC systems at 70 °C (see also in Fig. 1a). H_2O_2 decomposition rates in H_2O_2/TAED and H_2O_2/TBCC systems at 70 °C were both higher than that in H_2O_2 system without activators at 90 °C. It indicated that RCOOOH formed in H_2O_2/TAED and H_2O_2/TBCC systems must play a key role in promoting HO• generation and H_2O_2 decomposition rate at lower temperature. In addition, BA as the novel probe of HO• was found to exhibit excellent stability and reproducibility even at high temperature (more than 90 °C). The detection method allowed more specific and precise characterization of HO• generation during complex H_2O_2 bleaching systems.

3.2. *H_2O_2/TAED and H_2O_2/TBCC bleaching*

High temperature steaming condition is required for H_2O_2 bleaching without activators to gain satisfactory fabric performances. Addition of TAED or TBCC is intended to improve bleaching performances at lower temperatures. Fig. 3a and b displays performances of cotton fabric bleached in H_2O_2/TAED and H_2O_2/TBCC systems at variant temperatures (50–98 °C). It can be suggested that the fabric whiteness depended highly on H_2O_2 decomposition rate.

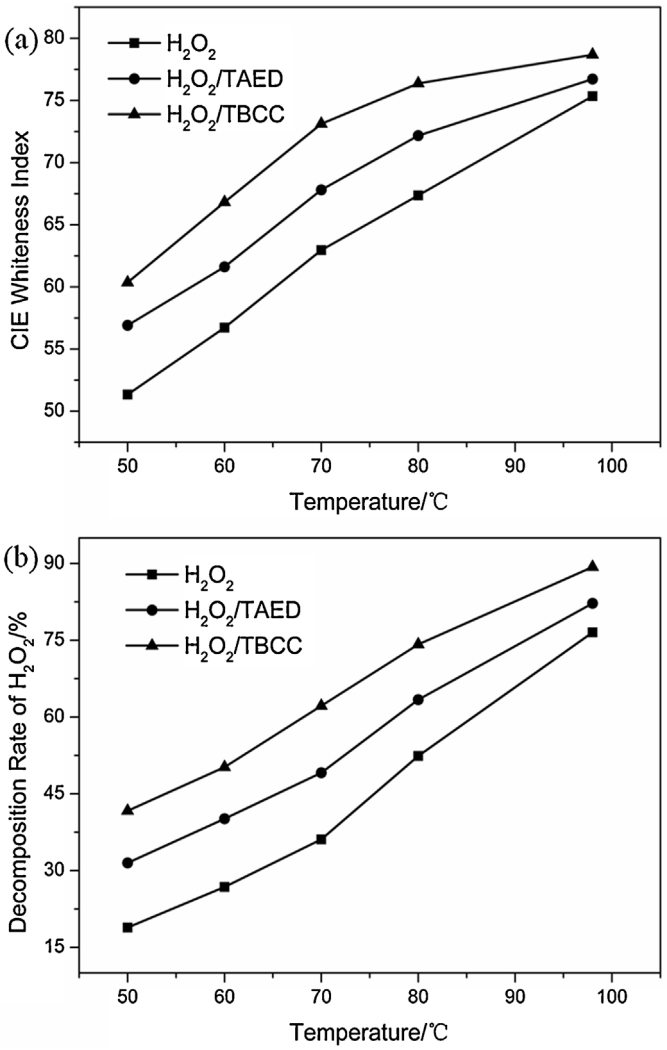


Fig. 3. Effect of temperature on fabric whiteness and H_2O_2 decomposition rate. (a) Fabric whiteness, (b) H_2O_2 decomposition rate. T = 50–98 °C.

Compared to H_2O_2 bleaching without activators, both H_2O_2/TAED and H_2O_2/TBCC systems apparently enhanced fabric whiteness and H_2O_2 decomposition rate at lower temperature (50–80 °C), while slightly enhanced at high temperature (98 °C). That is why H_2O_2/TAED and H_2O_2/TBCC bleaching are commonly preferred to be conducted at lower temperature (lower than 80 °C). Particularly, fabric bleached in H_2O_2/TBCC system exhibited excellent whiteness (73.12) at 70 °C, similar to that (75.35) in H_2O_2 system without activators at 98 °C. The results were consistent with that displayed in Figs. 1 and 2. Whiteness of cotton was both in proportional to H_2O_2 decomposition rate and HO• concentration (fluorescent intensity) in H_2O_2 bleaching system with/without activators. TAED and TBCC enormously enhanced HO• generation and fabric whiteness. High temperature promoted HO• generation and fabric whiteness. It can be concluded that beside RCOOOH, which is generally considered as the main effective component of H_2O_2/TAED and H_2O_2/TBCC bleaching, HO• may also has non-ignorable influence on fabric bleaching performances.

3.3. Scavenging effect of HO•

DMSO has been extensively applied to selectively scavenge HO• (Fukui, Hanasaki, & Ogawa, 1993; Kohno, Mizuta, Kusai, Masumizu, & Kakino, 1994; Ren, Kim, & Zhong, 2009), producing methyl sulfinic acid (CH_3SOOH) and methyl radical ($^{\bullet}CH_3$) with weak oxidizing ability. Kohno used DMSO to eliminate HO• disturbance with superoxide anion radical ($O_2^{-\bullet}$) detection by electron paramagnetic resonance spectroscopy (ESR) (Kohno et al., 1994). Ren added DMSO into single carbon nanotube system to demonstrate the generation of HO• by capillary electrophoresis (CE) (Ren et al., 2009). In the current work, DMSO was employed as HO• scavenger and the scavenging efficiency was proved. As shown in Fig. 4, HO• yield in H_2O_2/TAED and H_2O_2/TBCC systems as well as H_2O_2 system without activators all decreased dramatically after adding a certain amount of DMSO. Hence, DMSO could be used to control HO• concentration efficiently and explore H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism concerning HO•.

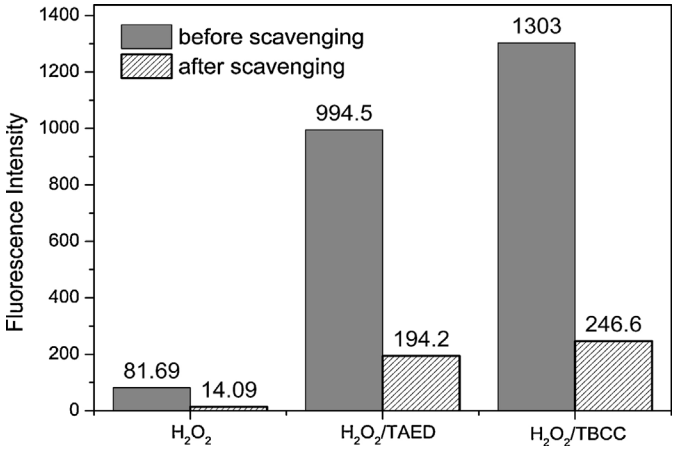


Fig. 4. Scavenging effect of HO• in each system. T = 70 °C.

3.4. Effect of HO• concentration on the cotton fabric bleaching performances

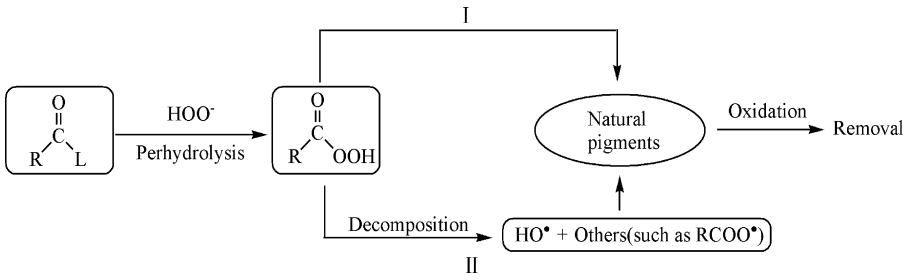
The large amounts of HO• generated in H_2O_2/TAED and H_2O_2/TBCC bleaching may affect bleaching performances and mechanical properties of cotton fabrics. In order to better understand the role of HO• plays in H_2O_2/TAED and H_2O_2/TBCC bleaching, varying amounts of DMSO (1–4 g/L) was added to describe effect of HO• concentration on the fabric bleaching performances by evaluating whiteness and tensile strength.

3.4.1. Effect of HO• concentration on fabric whiteness

As displayed in Table 1, with growing addition of DMSO, whiteness of fabric bleached in H_2O_2/TAED and H_2O_2/TBCC systems decreased more evidently at 70 °C, respectively from 68.01 to 60.58 and 73.12 to 61.69 in the presence of 2 g/L DMSO, even lower than that in H_2O_2 system without activators and DMSO (62.96). As DMSO addition increased to 4 g/L, whiteness of fabric bleached in H_2O_2/TAED and H_2O_2/TBCC systems continued to reduce rapidly, until the value came similar to that of fabric bleached in H_2O_2 system without activators. Superfluous addition of DMSO more than 4 g/L would probably cause solvent effect which impedes bleaching processes without selectivity.

Table 1
Effect of HO• concentration on fabric whiteness.

DMSO (g/L)	CIE Whiteness Index			
	Gray fabric	H_2O_2	H_2O_2/TAED	H_2O_2/TBCC
0	20.12	62.96	68.01	73.12
1	20.12	61.94	62.98	66.58
2	20.12	59.13	60.58	61.69
4	20.12	57.96	56.95	56.73



Scheme 2. Two main routes of H_2O_2/TAED and H_2O_2/TBCC bleaching.

The results proved that HO• is significantly responsible for H_2O_2/TAED and H_2O_2/TBCC bleaching, while plays a smaller role in H_2O_2 bleaching without activators. It indicated that other effective constituents except HO• contributed to the high whiteness of cotton sample bleached in H_2O_2 system without activators at high temperature (>90 °C) (Fig. 3a). TAED and TBCC accelerated the generation of enormous HO• which could oxidize and remove impurities to enhance fabric whiteness. As the addition of DMSO, HO• was scavenged promptly, resulting in a sharp decrease of fabric whiteness. Whiteness of fabric bleached in H_2O_2/TBCC system exhibited greatest decrease with the addition of DMSO because TBCC promoted most HO• generation in the absence of DMSO.

3.4.2. *Effect of HO• concentration on fabric tensile strength*

HO• could destroy cellulose chains by electrophilic addition and dehydrogenation reaction or other ways (Ek et al., 1989), leading to a certain degree of bond cleavage and strength loss. Table 2 shows the effect of HO• concentration on tensile strength in warp/weft of each bleached sample at 70 °C. In the absence of DMSO, fabric samples bleached in H_2O_2/TAED and H_2O_2/TBCC systems got larger damage than that in H_2O_2 bleaching without activators, with a respective strength loss about 7% and 8% compared to the gray fabric sample. It indicated that the large quantity of HO• generated in H_2O_2/TAED and H_2O_2/TBCC bleaching contributed to excellent whiteness as well as inevitable damage to fabric samples. As rising DMSO addition, strength of fabric bleached in H_2O_2/TAED and H_2O_2/TBCC systems increased due to less fabric damage by scavenging of substantive HO•. Especially for H_2O_2/TBCC bleaching, fabric strength gained maximum increase because of the most HO• yield in the absence of DMSO. And in the presence of 4 g/L DMSO, strength of fabric bleached in each system increased to a similar level. The results further clarified the generation of HO• and its important role in H_2O_2/TAED and H_2O_2/TBCC bleaching.

3.5. *Discussion of H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism*

Generally, peroxide acid (RCOOOH) is regarded as the main effective component of H_2O_2/TAED and H_2O_2/TBCC bleaching. Hydroperoxide (R_1OOH) is a kind of organic peroxides (R_1OOR_2) which is considered to induce formation of radicals R_1O• and •OR_2 by initial cleavage of the oxygen- oxygen bond (Braun, Cherdron, Rehahn, Ritter, & Voit, 2004; Hiatt, Mill, Irwin, & Castleman, 1968; Moad & Solomon, 2006; Sheppard & Kamath, 1979). In the current paper, RCOOOH as a kind of hydroperoxide was proved to induce the generation of enormous HO•. The strong oxidizing HO• played an important role in enhancing fabric whiteness with acceptable fabric damage by reacting with natural pigments as well as cellulose. Hence, there existed at least two main routes of H_2O_2/TAED and H_2O_2/TBCC bleaching, as shown in Scheme 2. Beside the generally considered route that RCOOOH directly interacts with natural pigments to improve fabric whiteness, as shown in Process I. Another bleaching route was proposed that HO• was decomposed from RCOOOH and could also oxidize and remove natural pigments to enhance fabric whiteness, as shown in Process II, other substances such as RCOO• may be also decomposed at the same time, which remains to be further clarified with suitable detection method.

Table 2
Effect of HO• concentration on fabric tensile strength.

DMSO (g/L)	Tensile strength in warp/weft (N)			
	Gray fabric	H_2O_2	H_2O_2/TAED	H_2O_2/TBCC
0	750.2/327.5	722.7/315.1	698.7/305.2	691.9/300.2
1	750.2/327.5	728.9/318.8	712.8/312.5	702.8/308.7
2	750.2/327.5	731.5/320.1	723.5/316.3	716.5/316.2
4	750.2/327.5	733.1/321.4	730.9/320.9	729.1/320.1

4. Conclusion

In this paper, H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism related to HO• was discussed. Benzenepentacarboxylic acid (BA) and dimethylsulfoxide (DMSO) were utilized to detect and scavenge HO• respectively. Enormous HO• was actually involved in H_2O_2/TAED and H_2O_2/TBCC bleaching, where the generation of HO• was found to be respectively increased 11 and 15 times compared to that in H_2O_2 bleaching without activators under the same alkali condition. TAED and TBCC both evidently enhanced fabric whiteness and H_2O_2 decomposition rate with an acceptable strength loss. The effect of HO• generation on fabric whiteness and strength was more significant in H_2O_2/TAED and H_2O_2/TBCC bleaching than that in H_2O_2 bleaching without activators. It proved that HO• plays a fairly important role in H_2O_2/TAED and H_2O_2/TBCC bleaching beside RCOOOH. Another bleaching route was proposed that HO• decomposed from RCOOOH could greatly oxidize and remove natural pigments to improve fabric whiteness. The present paper provided new view in H_2O_2/TAED and H_2O_2/TBCC bleaching mechanism, and potential guidance to improve the present bleaching processes.

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