

Recognizing a limitation of the TBLC-activated peroxide system on low-temperature cotton bleaching



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ABSTRACT

In this study, cotton was bleached at low temperatures with an activated peroxide system which was established by incorporating a bleach activator, namely, *N*-[4-(triethylammoniomethyl)benzoyl]caprolactam chloride (TBCC) into an aqueous solution of hydrogen peroxide (H₂O₂). Experimental results showed that the bleaching performance was unexpectedly diminished as the TBCC concentration was increased over the range of 25–100 g/L. Kinetic adsorption experiment indicated that this was most likely ascribed to the adsorptive interactions of TBCC and the in situ-generated compounds with cotton fibers. Such a limitation was especially fatal to cold pad-batch bleaching process of cotton in which a high TBCC concentration was often required. The results of this study may stimulate further research to avoid or overcome the limitation of the TBCC-activated peroxide system on low-temperature cotton bleaching.

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1. Introduction

Bleaching is an important stage in preparation of cotton-based textiles, aiming to remove the natural yellowish impurities present in cotton fibers and as such provide a white cotton substrate for dyeing and finishing (Lewin, 1984). Hydrogen peroxide (H₂O₂) is the most widely used bleaching agent in the textile industry owing to its low cost and eco-friendliness. H₂O₂ works on bleaching by dissociation into perhydroxyl anion (HOO[−]), as shown in Scheme 1, which is generally thought to be the main active species (Zeronian & Inglesby, 1995). However, H₂O₂ has an extremely low dissociation constant ($K_d = 1.78 \times 10^{-12}$ at 20 °C) and dissociates hardly in water. Therefore, strong alkalis such as sodium hydroxide (NaOH) and sodium carbonate (Na₂CO₃) are often added to promote the formation of HOO[−] anions (Brooks & Moore, 2000). Practically, cotton fabric can be bleached with H₂O₂ under alkaline conditions in rapid exhaustion process at a high temperature (>95 °C), or alternatively in cold pad-batch process for a long dwell time (approximately 24 h). Therefore, it would be highly desired to lower the bleaching temperature in the rapid exhaustion process for saving energy and reduce the dwell time of cotton fabric in the cold pad-batch process for shortening production cycle.

An alternative approach for cotton bleaching is to use an activated peroxide system which is established by incorporating a so-called bleach activator into an aqueous H₂O₂ solution (Hofmann, Just, Pritzkow, & Schmidt, 1992). The bleach activator can react with H₂O₂ to in situ generate peracid, as shown in Scheme 2, which is a more kinetically active oxidant than H₂O₂ and allows cotton fabric to be bleached in rapid exhaustion process at low temperatures (Cai & Evans, 2007; Scarborough & Mathews, 2000), or in cold pad-batch process for a short dwell time (Shao, Huang, Wang, & Liu, 2010; Wang & Washington, 2002).

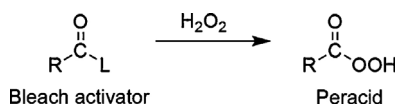
N-[4-(triethylammoniomethyl)benzoyl]lactam chlorides (TBLCs) are a promising class of bleach activators specially designed for industrial cotton bleaching (Lee, Hinks, Lim, & Hauser, 2010). As shown in Scheme 3, TBLCs contain a cationic charge which provides not only good solubility in water but also affinity towards negatively charged cellulosic fibers (Xu, Hinks, & Shamey, 2010). The TBLC-activated peroxide system was initially proposed for low-temperature bleaching of cotton fabric under alkaline conditions (e.g. using NaOH) by adding TBLC to a largely excessive amount of H₂O₂ (e.g. in a molar ratio of H₂O₂ to TBLC from 5:1 to 10:1) (Gursoy, El-Shafei, Hauser, & Hinks, 2004; Gursoy, Lim, Hinks, & Hauser, 2004; Lim, Gursoy, Hauser, & Hinks, 2004; Lim, Lee, Hinks, & Hauser, 2005). However, the most recent study revealed that the TBLC-activated peroxide system was most effective under near-neutral pH conditions by using equimolar amounts of H₂O₂ and TBLC (Fei et al., 2015). This gave rise to the proposal of

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Scheme 1. Dissociation of hydrogen peroxide.



Scheme 2. Reaction of bleach activator with H_2O_2 .

conducting the TBLC/ H_2O_2 / NaHCO_3 system for low-temperature cotton bleaching in rapid exhaustion process (Xu, Hinks, Sun, & Wei, 2015). It was reported that the TBLC/ H_2O_2 / NaHCO_3 system was comparable to the conventional peroxide bleaching method in improving the degree of whiteness of cotton fabric and resulted in no apparent damage to cotton fibers (Luo et al., 2015). In this study, however, a limitation of the TBLC-activated peroxide system on low-temperature cotton bleaching will be revealed, which is especially fatal to cold pad-batch bleaching process of cotton. Therefore, this study provides important insights into understanding such a limitation, and may stimulate further research to avoid or overcome it.

2. Experimental

2.1. Materials

Cotton greige knitted fabric was kindly provided by Hongdou Group Textiles Co., Ltd., China. N-[4-(triethylammoniomethyl)benzoyl]caprolactam chlorides (TBCC) was used as a prototype TBLC ($n=3$), and was synthesized with a purity greater than 97% according to the previously reported method (Wei, Sun, Wang, Du, & Xu, 2014). H_2O_2 (30%, w/w), NaOH (50%, w/w) and NaHCO_3 were purchased from Sinopharm Group Co. Ltd., China. Wetting agent (Penetrant JFC) and peroxide stabilizer (DM 1403) were purchased from Dynamic Chemicals Ltd., China.

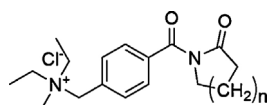
2.2. Bleaching method

2.2.1. Preparation of bleach bath

Table 1 shows the recipes for preparation of bleach baths which were applied to bleaching of cotton fabric in rapid exhaustion process (REP) and cold pad-batch process (CPP). Unless otherwise stated, the TBCC-activated peroxide system was conducted by adding a 1:1.2:1.4 molar ratio of TBCC, H_2O_2 and NaHCO_3 to a solution containing 1 g/L peroxide stabilizer and 1 g/L wetting agent for rapid exhaustion process, or a solution containing 3 g/L peroxide stabilizer and 3 g/L wetting agent for cold pad-batch process. A conventional peroxide system was also conducted for bleaching of cotton fabric in rapid exhaustion process using a commercial recipe, according to which equal amounts of H_2O_2 (30%, w/w) and NaOH (50%, w/w) was added to a solution containing 1 g/L peroxide stabilizer and 1 g/L wetting agent.

2.2.2. Rapid exhaustion process

A sample of 5 g of cotton fabric was immersed into the bleach bath using a liquor-to-goods ratio in the range from 5:1 to 100:1. The sample in bleach bath was heated to 50 °C for bleaching



Scheme 3. Chemical structure of TBLCs ($n=1-5$).

with the TBCC-activated peroxide system, and 95 °C for bleaching with the conventional peroxide system. Bleaching was carried out for 60 min on an Ahiba Nuance infrared laboratory dyeing machine (Datacolor International, USA) with a ramp rate of 3 °C/min. The bleached cotton fabric was rinsed thoroughly with copious amounts of water, and dried under ambient conditions.

2.2.3. Cold pad-batch process

A sample of 10 g of cotton fabric was padded through the bleach bath on a laboratory padder (Werner Mathis, Switzerland) with a 100% wet pick-up. The impregnated sample was sealed into a plastic bag and conditioned at room temperature for a desired time. The bleached cotton fabric was rinsed thoroughly with copious amounts of water, and dried under ambient conditions.

2.3. Kinetic adsorption of TBCC to cotton fabric

A sample of 2 g of bleached cotton fabric was added to 100 mL of water solution containing 1 g/L TBCC. Adsorption of TBCC to cotton fabric was allowed for various time at a desired temperature (e.g. 25 and 50 °C) under constant stirring. The concentration of TBCC on cotton fabric (C_{Cotton} , mg/g) was obtained using Eq. (1),

$$C_{\text{Cotton}} = 50(1.0 - C_t) \quad (1)$$

where C_t is the residual concentration of TBCC in solution, and can be determined by a spectrophotometric method as reported previously (Xu et al., 2010).

2.4. Whiteness measurement

The bleached cotton fabric was folded into a four-layer sample, and measured on a Datacolor 650 spectrophotometer (Datacolor International, USA). The instrument was set with the CIE Illuminant D65, CIE 1964 Supplemental Standard Observer, large area view (30 mm), and specular included. CIE whiteness index (WI) was calculated according to the AATCC Test Method 110-2010. Each sample was measured four times with 90° rotation between measurements to give an average value of CIE WI.

3. Results and discussion

The TBCC-activated peroxide system works on bleaching of cotton fabric using 4-(triethylammoniomethyl)perbenzoic acid (TPA) which is generated in situ by the reaction of TBCC with H_2O_2 , as shown in Scheme 4. When bleaching is completed, TPA would be converted to 4-(triethylammoniomethyl)benzoic acid (TBA), as shown in Scheme 5. The in situ generation of TPA and TBA in the bleaching solution was verified by using liquid chromatography-mass spectrometry (Chen et al. (2015)). In the present work, the TBCC-activated peroxide system was conducted using a 1:1.2:1.4 molar ratio of TBCC, H_2O_2 and NaHCO_3 , and the bleaching performance was evaluated in terms of the degree of whiteness of bleached cotton fabric.

Cotton fabric was bleached at 50 °C with the TBCC-activated peroxide system in rapid exhaustion process using a liquor-to-goods ratio of 20:1. Fig. 1 shows the concentration effect on the bleaching performance, in which the concentration of H_2O_2 (30%, w/w) is used for the convenience of comparison with the conventional peroxide system. It can be seen that the TBCC-activated peroxide system was quite effective for cotton bleaching and the maximum degree of whiteness was attained at a relatively low H_2O_2 concentration. Unlike the conventional peroxide system that gradually increased the degree of whiteness of cotton fabric as the H_2O_2 concentration was increased in the whole investigated range, however, the TBCC-activated peroxide system exhibited an

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