

# Review of Bleach Activators for Environmentally Efficient Bleaching of Textiles

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## Abstract

Textile wet processing is the most polluting aspect of textile manufacturing and contributes to the global textile industry's substantial carbon footprint. Textile preparation of cotton typically includes scouring and bleaching at high temperature and high pH. Substantial amounts of wastewater are produced that must be treated prior to being released to receiving fresh water. Recent research in our laboratories has focused on the development and application of compounds that enhance the bleaching process. We have developed novel cationic bleach activators that can be used to bleach cellulosic fabrics and fiber blends at reduced temperature, pH, and time. Results show that after optimization equivalent or improved performance in whiteness, absorbency and strength are possible relative to conventional bleaching methods. One of the most promising bleach activators to date is N-[4-(Triethylammoniomethyl) Benzoyl]-Butyrolactam Chloride (TBBC). TBBC was designed in our group to a) exhibit strong oxidation potential when activated; b) have reasonable hydrolytic stability; and c) be inherently substantive towards cellulosic and other fibers. In this paper, we review the development of bleach activators for textile bleaching, and discuss the opportunities and potential hurdles involved in commercialization of bleach activators for textile wet processing.

*Keywords:* Bleaching; Bleach Activator; Hydrogen Peroxide; Cotton; Textile Processing

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## 1 Introduction

Cotton cellulose is an ideal material for clothes, bedding and linens due to its high water absorbency, comfort and relative ease of dyeing and finishing. However, raw cotton contains natural colored impurities which significantly impair the inherent white appearance of cotton cellulose. Unless cotton is dyed deep or with dark shades, bleaching is required to remove the natural colored impurities prior to dyeing and finishing for the preparation of cotton textiles [1].

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Hydrogen peroxide is the most widely used bleaching agent in textile industry [2]. Rapid  $\text{H}_2\text{O}_2$  bleaching is traditionally carried out under alkaline conditions (pH 10.5–12) at a temperature close to the boil, which results in high energy consumption and also gives rise to significant fiber damage. Additionally, it is essential to neutralize the bleach solution and rinse the fabric with copious amounts of water when the bleaching process is complete. Reduction of energy and water consumption in the textile industry is becoming increasingly important. Hence, the impetus to develop new and more eco-friendly systems for bleaching cotton and other fibers under benign conditions (low temperature, reduced pH, short treatment period, and low chemical concentration) is highly desirable.

A promising approach towards bleaching of cotton textiles involves so-called bleach activators in peroxide bleaching systems. Bleach activators are peracid precursors, which form peracids *in situ* by reacting with hydrogen peroxide in an aqueous solution (Fig. 1) [3, 4]. The generated peracids are more kinetically powerful bleaching species than hydrogen peroxide, which allow bleaching to be conducted under more benign conditions, such as under reduced temperatures. Bleach activators were originally developed for incorporation into domestic and industrial laundry detergents [5–8]. In recent years, they have been proposed for use in industrial textile bleaching in an attempt to overcome the drawbacks of traditional hydrogen peroxide bleaching [9–27]. While neutral (e.g. N, N, N', N'-tetra acetyl ethylene diamine, TAED) and anionic (e.g. nonanoyl benzene sulfonic acid, NOBS) bleach activators continue to be of interest, much of the recent research has focused on synthesis and application of Cationic Bleach Activators (CBAs).

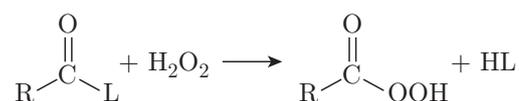


Fig. 1: Formation of peracids.

CBAs reported to date are based on a class of lactam-based peracid precursors (Fig. 2). They contain at least one cationic group, intended to provide water solubility and substantivity towards the negatively charged cellulosic substrates under aqueous conditions, and thus are very appropriate for bleaching of cellulosic textiles. In this paper, we review the development of CBAs for textile bleaching, and discuss the opportunities and potential hurdles to commercialization of CBAs for textile wet processing.

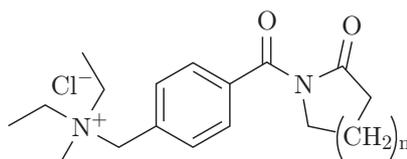


Fig. 2: Cationic bleach activators (where n=1–5).

## 2 Historical Development

CBAs were originally conceived and designed by the Procter and Gamble Company in the mid 1990s for incorporation into laundry and automatic dishwashing detergents [28, 29]. However,

the oxidation potential the CBAs exhibited was too high to be appropriate for laundry and automatic dishwashing detergent applications. Consequently, CBAs were evaluated for industrial textile bleaching.

Early stages of development of CBAs for application in bleaching of cotton focused on a prototype of CBAs, N-[4-(triethylammoniummethyl) benzoyl] caprolactam chloride (TBCC) (Fig. 2, where  $n = 3$ ) [12-15]. However, a successive study found that TBCC exhibited poor hydrolytic stability, which could impair its bleaching performance [16]. Hence, a CBA with enhanced hydrolytic stability was developed without substantial loss of bleaching effectiveness, consequently leading to the current prototype of CBAs, N-[4-(triethylammoniummethyl) benzoyl] butyrolactam chloride (TBBC) (Fig. 2, where  $n = 1$ ) [30]. Recent research has demonstrated that TBBC reacts with  $H_2O_2$  and bleaches cotton under neutral or near neutral pH conditions and at low temperatures [25-27]. This finding provides new impetus for advancing activated peroxide systems for industrial textile bleaching.

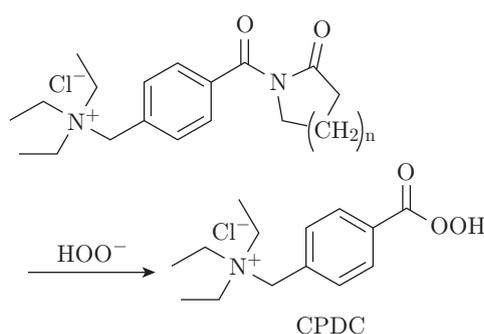
### 3 Chemistry

When mixed with  $H_2O_2$ , CBAs may undergo a series of competing reactions, depending on the conditions employed. Under aqueous conditions,  $H_2O_2$  can dissociate to produce the perhydroxyl anion ( $HOO^-$ ) (Reaction 1). CBAs react with  $HOO^-$  to form N-(4-carboperoxybenzyl)-N, N-diethylammonium chloride (CPDC), a low temperature bleaching agent (Reaction 2). Usually, an amount of alkali is added to facilitate dissociation of hydrogen peroxide and thus perhydrolysis of CBAs may result [2-4]. However, under alkaline conditions, the hydroxyl anion ( $HO^-$ ) present in the activated peroxide system may give rise to rapid decomposition of CPDC (Reaction 3) as well as hydrolysis of CBAs (Reaction 4), producing an inactive species for bleaching, N-(4-carboxybenzyl)-N, N-diethylammonium chloride (CBDC) [24, 31-33]. Therefore, reaction conditions must be controlled to maximize perhydrolysis of CBAs and minimize unwanted side reactions.

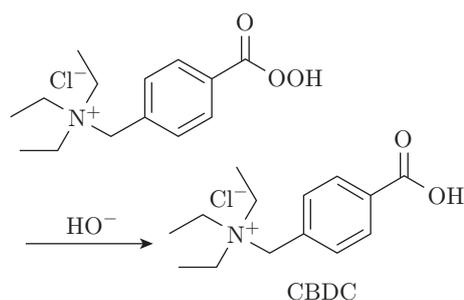
Reaction 1: Dissociation of hydrogen peroxide.



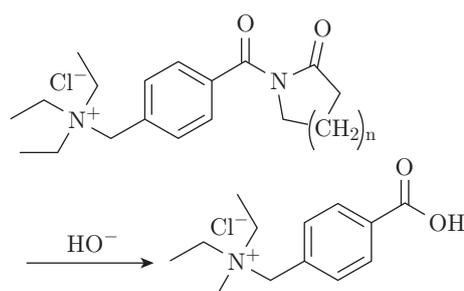
Reaction 2: Perhydrolysis of CBA.



Reaction 3: Decomposition of CPDC.



Reaction 4: Hydrolysis of CBA.



## 4 Sorption Behavior

As shown in Fig. 2, a lactam-based CBA contains one cationic group. In addition to providing water solubility, it is hypothesized that the cationic group exhibits inherent substantivity to negatively charged cellulosic substrates in aqueous solution and brings the activator into close proximity to cellulosic fibers as well as the target, colored impurities present in fibers. Such a physical interaction could facilitate efficient oxidation of the colored impurities.

This hypothesis has been supported by the sorption behavior of TBBC on bamboo viscose fiber [25]. Fig. 3 shows that TBBC is rapidly adsorbed onto the substrate and the sorption system reaches equilibrium after approximately 20 min. Fig. 4 shows that the amount of TBBC adsorbed onto bamboo viscose fiber increases nonlinearly as a function of the concentration of TBBC

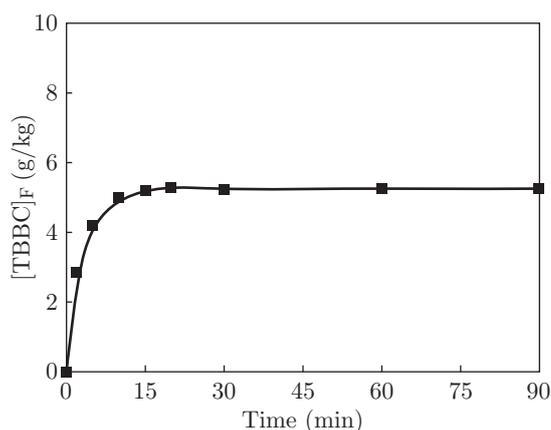


Fig. 3: Kinetic curve of sorption of TBBC onto bamboo viscose fiber in 5 g/L TBBC solution at 25°C.

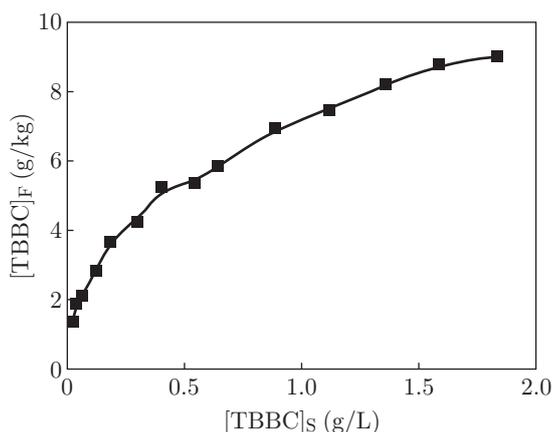


Fig. 4: Sorption isotherm of TBBC on bamboo viscose fiber in aqueous solution at 25°C.

in solution, and no saturation of TBBC sorption was observed within the concentration range investigated. The rapid and nonsaturated sorption behavior of TBBC on cellulosic substrates has practical importance for low temperature and rapid bleaching.

According to the sorption of TBBC on bamboo viscose fiber and the mode of activation to hydrogen peroxide, a model has been proposed to explain the effect of physical interaction between activator and cellulosic substrate on bleaching performance (Fig. 5). As can be seen, the whole process includes three feasible stages. In the first stage, TBBC exhausts onto the cellulosic fiber substrate through the sorption process, and the amount depends on the nature of the substrate surface and aqueous conditions (e.g. pH, temperature, and concentration of TBBC). In the second stage, TBBC reacts with  $H_2O_2$ , which may or may not be dissociated from a source of  $H_2O_2$  (for example, sodium perborate), to produce CPDC in the bulk solution and on the cellulosic substrate. In the third stage, cellulosic fibers are bleached by CPDC present in both bulk solution and on the cellulosic substrate. Results of these experiments have shown that the pre-sorption of TBBC on the cellulosic substrate (first stage) plays a key role in improving the bleaching performance of the TBBC-activated peroxide system [25]. Though the sorption-activation of the peroxide bleaching model can explain the effect of the cationic group on bleaching performance, it is essential to incorporate an anionic, cationic or nonionic surfactant in a practical bleaching system to ensure the wettability of cotton textiles. It is not yet clear whether the use of surfactants in the CBA-activated peroxide system impacts the physical interaction between CBA and the cellulosic substrate. Further investigation is on-going to determine the effect of the cationic group on bleaching performance in the presence of a surfactant.

## 5 Hydrolytic Stability

It is important that CBAs have sufficient hydrolytic stability for practical utility in industrial textile bleaching because aqueous bleach solutions are often prepared and stored for a short period of time prior to textile bleaching. For example, TBCC was found to be inappropriate for practical textile bleaching due to its poor hydrolytic stability although it exhibited satisfactory bleaching performance in cold-pad batch and hot peroxide bleaching processes for cotton textiles in a shorter treatment period and at lower temperatures than conventional peroxide bleaching [12-16].

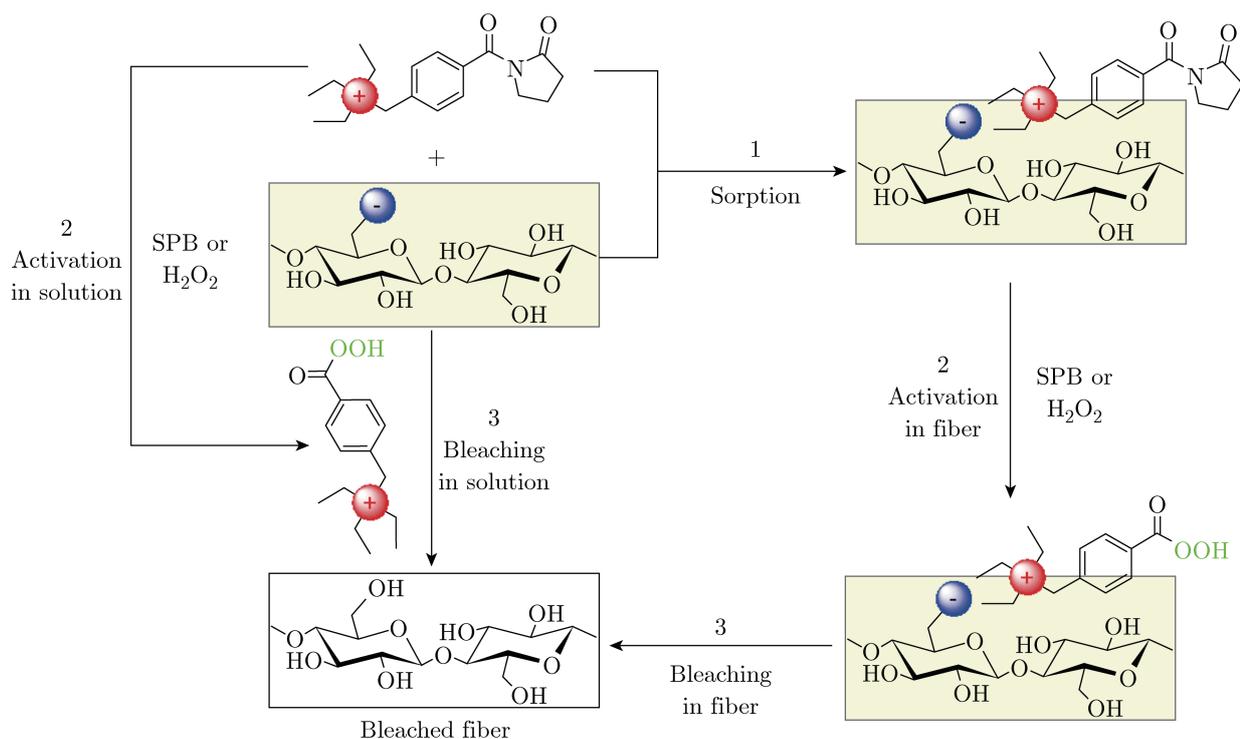


Fig. 5: A proposed model for sorption-activation peroxide bleaching based on TBBC.

It has been shown that the rate of hydrolysis of CBAs is related to the type of leaving group [30]. In an effort to find a CBA with sufficient hydrolytic stability as well as bleaching effectiveness, a series of CBAs containing lactam-based leaving groups of various ring size were synthesized and their performance was assessed (Fig. 2,  $n=1, 2, 3, 4,$  and  $5$ ) [24]. The hydrolytic stability of CBAs was found to be as follows (in descending order of hydrolytic stability):



The bleaching performance of CBAs, i.e. the degree of whiteness of bleached cotton textiles, directly correlated with hydrolytic stability.

Although the  $\omega$ -octalactam-based CBA ( $n=5$ ) is the most stable compound in aqueous solution, the cost of manufacture of this compound is relatively high compared to other compounds due to the high cost of  $\omega$ -octalactam. Since butyrolactam ( $n=1$ ) is a low cost alternative that delivers near equivalence in hydrolytic stability and oxidative power, TBBC is currently the most promising CBA.

## 6 Bleaching Parameters

Bleaching conditions must be controlled to maximize desired reactions and minimize side reactions. The performance of the CBA-activated peroxide system depends strongly on bleaching

parameters, such as concentration of CBA, concentration of  $\text{H}_2\text{O}_2$ , pH or concentration of NaOH, temperature and treatment period [12-15, 18, 23, 26, 27].

## 6.1 Concentration of CBA

CBAs themselves do not bleach cotton fibers. They may react with hydrogen peroxide to form CPDC (Reactions 1 and 2) which is a more powerful bleaching species than  $\text{H}_2\text{O}_2$ . In the presence of sufficient  $\text{H}_2\text{O}_2$ , the concentration of CBA will directly determine the abundance of CPDC in the bleaching solution and consequently the bleaching performance, assuming that perhydrolysis of CBA is achieved with minimal side reactions.

In early studies on the CBA-activated peroxide system, the concentration of CBA was found to be statistically significant but its contribution to improving bleaching performance was reduced compared to other parameters [12-15]. It was suspected that, under those bleaching conditions, decomposition of CPDC (Reaction 3) and hydrolysis of CBA (Reaction 4) occurred simultaneously, thus lowering the concentration of the active species.

The latest studies confirmed that the occurrence of decomposition of CPDC and hydrolysis of CBA were likely to be the main reasons for the diminished influence of the concentration of CBA on bleaching performance [23]. It was proposed that the CBA-activated peroxide system exhibited its best performance under neutral or near neutral pH conditions with the use of equimolar CBA and hydrogen peroxide and its performance was remarkably improved by increasing the concentration of CBA [26, 27]. However, further studies are needed to optimize the concentration of CBA to match various bleaching conditions. Since CBA involves a higher cost of manufacture than  $\text{H}_2\text{O}_2$ , a criterion for deciding a concentration of CBA used in a bleaching system should be to use a concentration as low as possible to produce acceptable bleaching performance.

## 6.2 Concentration of Hydrogen Peroxide

In the early studies of cationic bleach activators, a large excess of  $\text{H}_2\text{O}_2$  compared to the activator was deliberately used [9-17, 22]. Since the system contained both  $\text{H}_2\text{O}_2$  and peracid, it was hypothesized that the two compounds had an additive effect on bleaching performance.

However, this additive effect is hard to achieve under alkaline bleaching conditions (e.g.  $\text{pH} > 11$ ), because of the rapid decomposition of CPDC and hydrolysis of CBA. Therefore, the excess  $\text{H}_2\text{O}_2$  continued to play a dominate role in bleaching [23]. This explains why the concentration of  $\text{H}_2\text{O}_2$  was found to be more significant than the concentration of bleach activator in most studies. The additive effect of CBA and  $\text{H}_2\text{O}_2$  is also hard to achieve under a neutral or near neutral pH conditions because  $\text{H}_2\text{O}_2$  alone is not active under these pH conditions even in a large excess. However, CBAs were found to exhibit good bleaching effectiveness at neutral to mildly-alkaline conditions, when combined with  $\text{H}_2\text{O}_2$  or a precursor like SPB.

Therefore, our research group investigated the use of  $\text{H}_2\text{O}_2$  in equimolar or slightly excess relative to the concentration of CBAs under a neutral or near neutral pH conditions [26, 27].

## 6.3 Effect of pH

Of course, pH plays a critical role in the CBA-activated peroxide system. When mixed  $\text{H}_2\text{O}_2$ ,

CBAs are converted to CBDC which readily dissociates to release hydrogen ions ( $H^+$ ). The pH of bleaching solution would decrease so as to decelerate or even terminate the dissociation of  $H_2O_2$ . Hence, the subsequent perhydrolysis of CBAs and cotton bleaching declines if the released  $H^+$  is not neutralized by adding a quantity of alkaline reagent and/or appropriate buffer. To achieve an effective CBA-activated peroxide system for bleaching cotton, optimizing the pH of the bleach bath is the key.

Most of the previous studies on CBAs focused on utilizing NaOH in the CBA-activated peroxide system and maintaining pH 10-12 [12-16]. Such a high pH inevitably caused competing decomposition of CPDC and hydrolysis of CBA so that the efficiency of the CBA was reduced. It seemed plausible that increasing the concentration of NaOH could improve the bleaching performance of the CBA-activated peroxide system. However, this was only possible with a large excess of  $H_2O_2$  used in bleaching.

Bleaching was therefore carried out under a series of buffered pH values (pH 7-11), and the CBA-activated peroxide system was found to be most effective under neutral or near neutral pH conditions [23, 26, 27]. This finding provided new insights into the CBA-activated peroxide system as an environmentally benign approach to industrial textile bleaching.

## 6.4 Temperature

In almost all statistical design of experiment studies on the CBA-activated peroxide system for hot rapid bleaching of cotton textiles, temperature has been shown to exhibit the largest effect on bleaching performance [14, 15, 23, 26, 27]. This is mainly due to the temperature dependence of reaction rates. An increase of temperature gives rise to a higher rate of bleaching, and thus effectively improves the bleaching performance. However, one key goal of using a bleach activator is to reduce the temperature. Experimental results showed that TBBC was capable of creating an effective bleaching system over a temperature range of 40-60°C for a rapid bleaching process [26, 27]. Compared to conventional hot peroxide bleaching at around 95°C, the low temperature bleaching significantly reduces energy cost and saves time associated with heating a bleach bath to elevated temperatures.

## 6.5 Treatment Period

Reducing the bleaching time is beneficial in at least two ways: energy consumption is reduced and the capacity to treat more fabric is increased. Since peracid (CPDC) generated from CBAs is a more kinetically powerful bleaching species than  $H_2O_2$ , it allows bleaching to be completed more rapidly. Due to the temperature dependence of the bleaching rate, the treatment period varies as a function of temperature. Experimental results showed that an increase in temperature led to a shorter treatment period [27], with bleach times as short as 30 minutes.

## 7 Bleaching Performance

The bleaching performance of the CBA-activated peroxide system is usually estimated in terms of the CIE degree of whiteness (WI) and the degree of polymerization (DP) of the bleached cotton

textiles. A higher WI value indicates a higher bleaching effectiveness and a higher DP value indicates less fiber damage.

Table 1 summarizes the bleaching performance of TBCC and TBBC-activated peroxide systems compared to conventional peroxide bleaching. As can be seen from the table, although the TBCC-activated peroxide system results in an equivalent degree of whiteness and less fiber damage compared to conventional peroxide bleaching, it involves the use of a high concentration of sodium hydroxide and only modest reduction of temperature was observed. Therefore, the TBCC-activated peroxide system shown in Table 1 provides some benefit but it was found to be insufficient for commercialization. However, the later study that utilized TBBC-activated peroxide system under neutral pH conditions was effective at bleaching at lower temperature and pH, and thus was more environmentally benign without significant reduction of DP.

Table 1: Bleaching performance of CBA-activated peroxide systems

Investigation	System	Parameters					Performance	
		[activator] (g/L)	[H <sub>2</sub> O <sub>2</sub> ] (g/L)	pH or [NaOH] (g/L)	T (°C)	t (min)	WI	DP
1 [14]	Control						-1.24	3302
	TBCC-activated	3.00	8.55	1.50	102.5	18.3	75.56	2984
	Conventional		8.55	1.50	110.0	25.0	75.91	2779
2 [26]	Control						45.31	2324
	TBBC-activated	7.50	2.15	pH 7.0	50.0	30.0	78.94	2293
	conventional		6.00	pH 11.5	100.0	30.0	81.95	1794

## 8 Conclusions

This paper reviewed the development of Cationic Bleach Activators for textile bleaching and discussed the opportunities and potential hurdles to commercialization of bleach activators for textile wet processing. CBAs work by generating more kinetically active bleaching species (CPDC) by reacting with H<sub>2</sub>O<sub>2</sub>. However, decomposition of CPDC and hydrolysis of CBAs reduces bleaching effectiveness. Reaction conditions must be optimized and controlled to maximize perhydrolysis of CBAs and minimize side reactions.

The hypothesis that the cationic group contained in CBAs exhibits substantivity has been confirmed by the observation of sorption behavior of TBBC on bamboo viscose fiber. The physical interaction between CBAs and cellulosic substrates appears to enhance bleaching of colored impurities present in cotton.

Reasonably hydrolytic stability of CBAs is important for practical utility in industrial textile bleaching. CBAs exhibit differing degrees of hydrolytic stability when incorporating various ring sizes of lactam-based leaving groups. According to the hydrolytic stability and the manufacturing cost pertaining to lactam compounds, TBBC has been selected as the most promising CBA for industrial textile bleaching.

The performance of a CBA-activated peroxide system strongly depends on bleaching parameters, such as concentration of CBA, concentration of H<sub>2</sub>O<sub>2</sub>, pH, temperature and treatment

period. Experimental results revealed that the TBBC-activated peroxide system provides satisfactory bleaching performance under the conditions of equimolar amounts of TBBC and  $H_2O_2$ , neutral pH, 40–60°C and 30 minutes dwell time. Such an activated peroxide system provides many benefits over conventional peroxide bleaching, including saving time, energy, reducing environmental impact, reducing fiber damage from bleaching, and enhancing plant capacity.

Further work is on-going to investigate the effects, if any, of CBA-based bleaching on dyeing performance, pilot plant and full production scale-up and also the potential for improved benefits, including reduced rinsing after bleaching.

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