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Activated peroxide bleaching of regenerated bamboo fiber using a butyrolactam-based cationic bleach activator

Changhai Xu · Renzo Shamey · David Hinks

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Abstract Regenerated bamboo fibers are potentially a valuable source of renewable fibers for use in a wide variety of applications. As with almost all natural fibers, inherent yellowness must be reduced or eliminated in order for the fibers to be used effectively in processes such as dyeing. Oxidative bleaching in the form of hot alkaline hydrogen peroxide is the most common method for bleaching cellulosic fibers. However, significant fiber damage results, especially in the case of regenerated bamboo. Recently, more benign oxidative bleaching methods have been developed using so-called bleach activators. Reported is an effective bleaching method using a novel bleach activator, N-[4-(triethylammoniomethyl)benzoyl]butyrolactam chloride (TBBC). The ratio of TBBC to hydrogen peroxide and pH were found to be critical to achieving effective bleaching at low temperature. Using equimolar amounts of TBBC and hydrogen peroxide at pH 7 and 50 °C, comparable whiteness and less fiber damage compared with conventional peroxide bleaching was obtained. However, at pH 11.5, TBBC had no effect on whiteness.

C. Xu · R. Shamey · D. Hinks (✉)
Polymer and Color Chemistry Program, Department of Textile Engineering, Chemistry and Science, North Carolina State University, PO Box 8301, Raleigh, NC 27695, USA
e-mail: david_hinks@ncsu.edu

Keywords Regenerated bamboo fiber · Hydrogen peroxide · Peracid · Bleaching · Bleach activator · Lactam

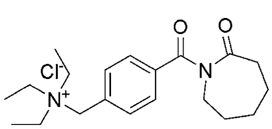
Abbreviations

AATCC	American Association of Textile Chemists and Colorists
ANOVA	Analysis of variance
CBA	Cationic bleach activators
CCD	Central composite design
CIE	Commission Illumination de L'Eclairage (International Commission on Illumination)
DP	Degree of polymerization
F	Fluidity
RSM	Response surface method
t	Time
T	Temperature
TBBC	N-[4-(triethylammoniomethyl)benzoyl] butyrolactam chloride
TBCC	N-[4-(triethylammoniomethyl)benzoyl] caprolactam chloride
2FI	Two factor-interaction model
WI	Whiteness index

Introduction

Regenerated bamboo fiber is made from bamboo pulp using alkali pulping and solution spinning (Sui 2005). The abundance and rapid regeneration of bamboo plants make regenerated bamboo fiber a promising source as a textile material (Liese 1987). While regenerated bamboo fiber is similar to conventional viscose fibers in structure and physical properties (Shen et al. 2004; Xu et al. 2007; Wang et al. 2009), it contains undesirable yellow impurities. Therefore, bleaching is commonly required for the preparation of regenerated bamboo fiber to remove the colored impurities prior to dyeing and finishing (Zeronian and Inglesby 1995; Brooks and Moore 2000; Mussatto et al. 2008). Unfortunately, conventional peroxide bleaching is typically conducted under conditions of high temperature (e.g. 95 °C) and pH 11.5, which causes significant strength loss of fibers (Andrews and Singh 1979). This is especially problematic to regenerated bamboo fiber owing to its inherent poor strength properties relative to cotton (Sui 2005; Erdumlu and Ozipek 2008; Wang et al. 2009). Hence, a more benign bleaching method is important to maximize the potential of bamboo as a source of commercial textile fibers.

Bleach activators are organic peracid precursors which liberate more kinetically active peracid in situ in the presence of hydrogen peroxide in a weakly alkaline aqueous solution (Grime and Clauss 1990; James and Mackirdy 1990; Hofmann et al. 1992; Beck et al. 2007). Using bleaching activators, a relatively benign bleaching system can be conducted at lower temperature and reduced time relative to conventional peroxide bleaching, thereby leading to reduced fiber damage. Cationic bleach activators (CBAs) are a class of quaternary ammonium peracid precursors (Willey et al. 1997), as exemplified by N-[4-(triethylammoniomethyl) benzoyl]caprolactam chloride (TBCC) (**1**) (Gursoy et al. 2004a, b; Lim et al. 2004; Lim et al. 2005). However, TBCC is sensitive to hydrolysis in aqueous solutions (Lee et al. 2005). Hence, N-[4-(triethylammoniomethyl)benzoyl]butyrolactam chloride (TBBC) (**2**) was developed with enhanced hydrolytic stability without substantial loss of bleaching performance (Hauser et al. 2007; Lee et al. 2009; Križman et al. 2007).



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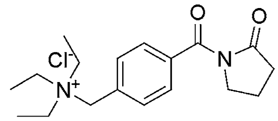

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In the present study, TBBC was investigated as a bleach activator for hydrogen peroxide bleaching of regenerated bamboo with the goal of providing acceptable whiteness without significant loss of fiber strength and at the same time reducing energy consumption relative to conventional bleaching.

Experimental

Materials

TBBC was synthesized and purified to >97% according to the procedure reported previously (Hauser et al. 2007). Regenerated bamboo knitted fabric (139 g/m^2) was donated by Donghua University, China. Hydrogen peroxide (H_2O_2, 35% w/w) and sodium hydroxide (NaOH, 50% w/w) were purchased from Sigma–Aldrich Co. (St. Louis, MO, USA). The wetting agent (Kieralon MFB) and stabilizer (Prestogen N-D) were purchased from BASF (Charlotte, NC, USA). All other chemicals were obtained from Fisher Scientific Co. (Fairlawn, NJ, USA). Untreated city water from Raleigh, NC, was used throughout the experiments to approximate industrial wet processing conditions.

Central composite design

Response surface method (RSM) is a tool for quantifying the relationship between multiple input variables and one or more output variables, and is therefore useful for process optimization. Central composite design (CCD) is one of the most favored RSM approaches (del Vecchio 1997). In this study, the experimental design and statistical analysis were performed using Design-Expert 7.0 software (Stat-Ease Inc., USA). Four factors, namely time (t, min), temperature (T, °C), and the concentrations of H_2O_2 (35% w/w) (g/L) and TBBC (g/L) were investigated, as shown in Table 1. The full factorial experimental design contained a total of 30 samples, including 16 at factorial points, eight at axial points and six at

Table 1 Coded and actual levels of the designed factors

Symbol	Factor	Level				
		−2	−1	0	+1	+2
X_1	TBBC, g/L	0.0	1.0	2.0	3.0	4.0
X_2	H_2O_2 (35% w/w), g/L	4.0	5.5	7.0	8.5	10.0
X_3	*t*, min	10	20	30	40	50
X_4	*T*, °C	60	70	80	90	100

central points. The pH values for all the bleach baths were fixed at 11.5 by addition of NaOH (50% w/w).

Effect of pH

The effect of pH on bleach activation was investigated using bleach baths buffered at various pH values listed in Table 2. The buffer solutions for pH 7.0, 9.0 and 10.0 were prepared by adding 0.1 mol/L weak base to its conjugate acid (0.1 mol/L); and the buffer solutions for pH 8.0 and 11.0 were prepared by adding H_2SO_4 (98% w/w) to 0.1 mol/L Na_2HPO_4 and NaOH (50% w/w) to 0.1 mol/L Na_2HPO_4, respectively, until the desired pH was obtained. In this series of experiments, 5 g/L TBBC was added to each bleach bath with an equimolar amount of H_2O_2 (35% w/w, 1.43 g/L). Bleaching experiments were undertaken at 50 and 80 °C for 30 min. A control bleaching was run under the same conditions except that TBBC was excluded from the experiment.

Bleaching procedure

All bleaching experiments were performed using an Ahiba Nuance Infrared Laboratory Dyeing Machine (Datacolor International, USA) at a liquor-to-goods ratio of 20:1. Each bleach bath contained 1.0 g/L wetting agent and 1.0 g/L stabilizer. After a desired amount of TBBC was dissolved in a 200 mL bleach bath, 10 g fabric was added. The bath was heated to a target temperature at the rate of 4 °C/min. After a desired time, the bleaching was quickly quenched using copious amounts of cold water, and the fabric was then dried under ambient conditions.

Table 2 Buffer solutions

pH	0.1 M buffer solution
7.0	Na_2HPO_4/NaH_2PO_4
8.0	Na_2HPO_4/H_2SO_4
9.0	$Na_2CO_3/NaHCO_3$
10.0	$Na_2CO_3/NaHCO_3$
11.0	$Na_2HPO_4/NaOH$

Whiteness measurement

The reflectance spectra of all fabrics were measured using a Datacolor Spectraflash SF600X (Datacolor International, USA), following the guidelines stated in AATCC Evaluation Procedure 11 (AATCC 2009) and using UV calibrated, specular component included, illuminant D_{65} and the CIE 10 degree supplemental standard observer settings. CIE Whiteness Index (CIE WI) was calculated from the reflectance spectrum of each sample using AATCC Test Method 110 (AATCC 2009). A four-layer fabric sample was measured four times by rotating the sample at 90 degrees between each measurement. The average value was recorded.

Fiber damage

Fiber damage was evaluated by measuring the degree of polymerization (*DP*) calculated from the cuen fluidity (*F*) of fiber dispersion, as shown in Eq.1 (Anonymous 1981),

$$\mathrm{DP} = 2032 \log_{10}\left(\frac{74.35 + F}{F}\right) - 573 \quad (1)$$

where *F* was measured according to AATCC Test Method 82 (AATCC 2009).

Results and discussion

CCD analysis

The whiteness values of the bleached samples based on CCD are illustrated in Table 3. Model summary statistics suggests that the experimental data are sufficient to fit a two-factor interaction (2FI) model. The analysis of variance (ANOVA) for the response surface (CIE WI) 2FI model is summarized in Table 4; as $p < 0.0001$ the 2FI model is significant. The "lack of fit p-value" of 0.4466 implies the lack of fit is not significant relative to the pure error. The

Table 3 Conditions and testing results of the CCD with actual levels of factors

Run	TBBC, g/L	H_2O_2 (35% w/w), g/L	*t*, min	*T*, °C	CIE WI
1	1.0	5.5	20	70	45.64
2	3.0	5.5	20	70	47.32
3	1.0	8.5	20	70	44.73
4	3.0	8.5	20	70	44.77
5	1.0	5.5	40	70	46.66
6	3.0	5.5	40	70	48.02
7	1.0	8.5	40	70	50.66
8	3.0	8.5	40	70	50.19
9	1.0	5.5	20	90	48.93
10	3.0	5.5	20	90	48.61
11	1.0	8.5	20	90	48.60
12	3.0	8.5	20	90	48.74
13	1.0	5.5	40	90	51.81
14	3.0	5.5	40	90	53.75
15	1.0	8.5	40	90	54.45
16	3.0	8.5	40	90	55.99
17	0.0	7.0	30	80	49.39
18	4.0	7.0	30	80	47.67
19	2.0	4.0	30	80	45.78
20	2.0	10.0	30	80	51.05
21	2.0	7.0	10	80	44.41
22	2.0	7.0	50	80	52.69
23	2.0	7.0	30	60	41.76
24	2.0	7.0	30	100	52.66
25	2.0	7.0	30	80	48.77
26	2.0	7.0	30	80	50.82
27	2.0	7.0	30	80	49.46
28	2.0	7.0	30	80	48.74
29	2.0	7.0	30	80	48.32
30	2.0	7.0	30	80	51.21

effects of main factors and the 2-factor interactions are presented in Figs. 1, 2, 3, 4, 5, 6.

Using backward regression, the model terms having p-values greater than 0.05 in Table 4 were removed from the 2FI model, since they were not statistically significant at 95% confidence level. Table 5 shows the coefficient estimates yielded by fitting the modified 2FI model to the experimental data based on coded levels for each significant model term. In Fig. 7, the normal probability plot shows the residual was normal. Therefore, the modified model's prediction of whiteness for the bleached bamboo fiber under the investigated experimental conditions is statistically significant.

The values of coefficient estimates in Table 5 suggest the contributions of the factors to the whiteness of regenerated bamboo fiber. It was observed that temperature is the most significant

Table 4 ANOVA summary for the response surface 2FI model

Source	Sum of squares	df	Mean square	*F* value	*p*-value Prob > *F*
Model	264.66	10	26.47	15.97	<0.0001
X_1	0.25	1	0.25	0.15	0.6997
X_2	13.40	1	13.40	8.08	0.0104
X_3	107.32	1	107.32	64.76	<0.0001
X_4	124.62	1	124.62	75.21	<0.0001
X_1X_2	0.73	1	0.73	0.44	0.5158
X_1X_3	0.50	1	0.50	0.30	0.5890
X_1X_4	0.030	1	0.030	0.018	0.8948
X_2X_3	13.52	1	13.52	8.16	0.0101
X_2X_4	0.24	1	0.24	0.15	0.7063
X_3X_4	4.05	1	4.05	2.44	0.1345
Residual	37.29	19	1.49		
Lack of fit	30.13	14	1.51	1.21	0.4466
Pure error	7.15	5	1.43		
Cor Total	296.15	29			

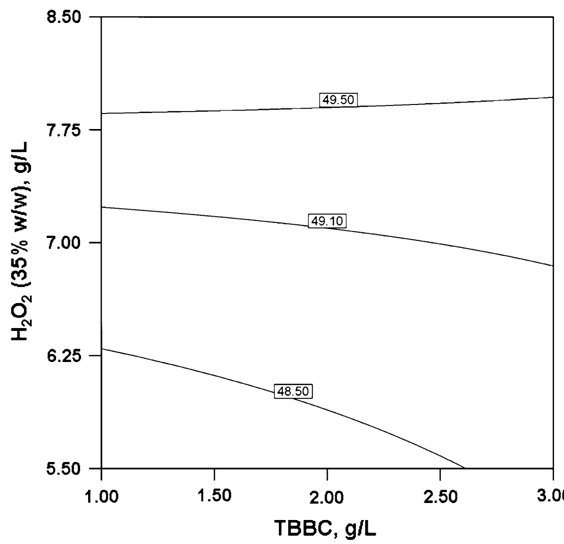

Fig. 1 Model prediction of the effect of H_2O_2 and TBBC on whiteness of regenerated bamboo fiber

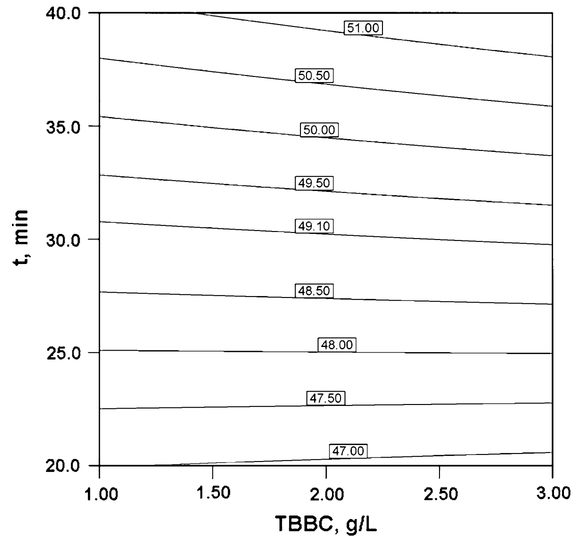

Fig. 2 Model prediction of the effect of time and TBBC concentration on whiteness of regenerated bamboo fiber

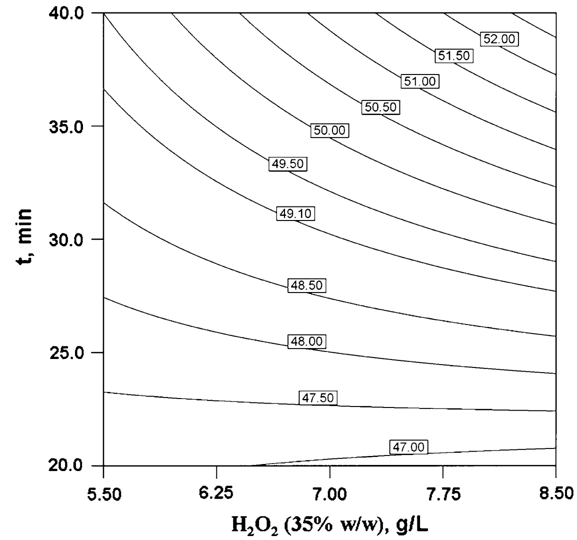

Fig. 4 Model prediction of the effect of time and H_2O_2 concentration on whiteness of regenerated bamboo fiber

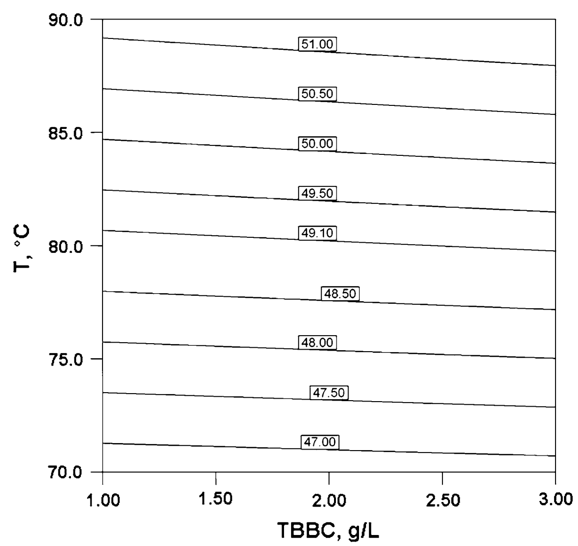

Fig. 3 Model prediction of the effect of temperature and TBBC concentration on whiteness of regenerated bamboo fiber

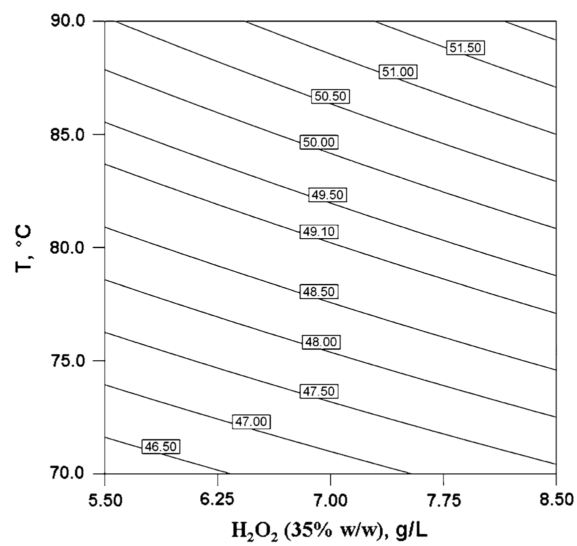

Fig. 5 Model prediction of the effect of temperature and H_2O_2 concentration on whiteness of regenerated bamboo fiber

factor in contributing to the resultant whiteness, in turn followed by time and concentration of H_2O_2. The model term X_2X_3 indicates that there is a significant interaction between time and concentration of H_2O_2 in the bleaching process; the coefficient estimate of the interaction is roughly equal to that of concentration of H_2O_2, which indicates that, at the low level of time, concentration of H_2O_2 has essentially no effect. Surprisingly, the contribution of the concentration of TBBC to the whiteness of regenerated bamboo fiber under the investigated conditions was found to be insignificant as evidenced by ANOVA analysis, $p = 0.6997$, shown in Table 4.

Chemistry of TBBC

In the CCD-based bleaching experiments, the inefficiency of TBBC in activating hydrogen peroxide is

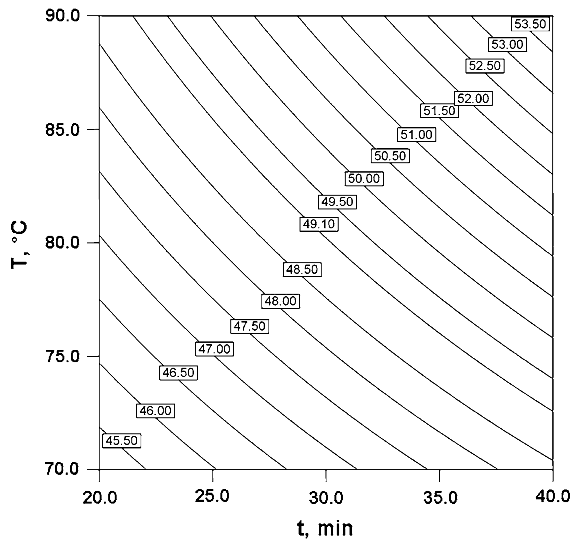

Fig. 6 Model prediction of the effect of temperature and time on whiteness of regenerated bamboo fiber

Table 5 Effect of significant model terms ($R^2 = 0.8741$)

Model term	Coefficient estimate	Standard error
Constant	49.05	0.22
X_2	0.75	0.25
X_3	2.11	0.25
X_4	2.28	0.25
X_2X_3	0.92	0.31

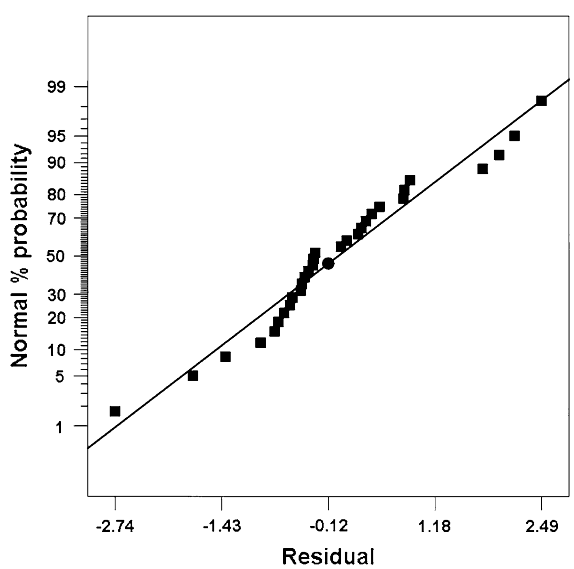

Fig. 7 Graph of normal probability of residual

unexpected. A clear explanation must address a series of chemical reactions related to TBBC. In most of the research related to bleach activators attempted for industrial textile bleaching, it has been stated that the activated peroxide bleaching system could provide an enhanced whiteness by using bleach activators and a large excess of hydrogen peroxide at a molar ratio of around 1:10 and at relatively high pH of around 11.5, assuming that bleach activators and alkaline hydrogen peroxide could simultaneously work on bleaching (Matthews 1999; Scarborough and Matthews 2000; Cai et al. 2001; Wang and Washington 2002; Gursoy et al. 2004a, b; Lim et al. 2004; Lim et al. 2005; Cai and Evans 2007). This is plausible because hydrogen peroxide dissociates at pH 11 to 11.5 to release perhydroxyl anions that directly bleach fibers (Zeronian and Inglesby 1995). On the other hand, perhydroxyl anions react with bleach activators to generate peracid species, which can bleach fibers under milder conditions (Grime and Clauss 1990; James and Mackirdy 1990; Hofmann et al. 1992; Beck et al. 2007).

However, in the case of regenerated bamboo fiber, TBBC failed to provide significantly enhanced bleaching efficiency under the conditions tested in the CCD. One possible explanation is that the high pH causes rapid hydrolysis of the bleach activator or decomposition of peracid generated from TBBC (D'ans and Mattner 1951; Goodman et al. 1962; Koubek et al. 1963; Akiba and Simamura 1970; Botsivali and Evans 1979; Evans and Upton 1985; Koberstein and Kurzke 1987; Hauthal et al. 1990; Janicot et al. 1996).

Figure 8 presents the possible reactions related to TBBC, of which only perhydroxyl anions dissociated from hydrogen peroxide and TBBC peracid generated by perhydrolysis of TBBC are oxidizing agents. In alkali aqueous solution, TBBC can hydrolyze into a carboxylate anion, which reduces bleaching efficiency. On the other hand, the base-catalyzed decomposition of peracid also could cause the loss of bleaching efficiency through consumption of an equimolar amount of perhydroxyl anions (D'ans and Mattner 1951; Wiberg 1955; Jencks and Carriuolo 1960; Edwards and Pearson 1962; Pearson and Edgington 1962; Koubek et al. 1963; McIsaac et al. 1972). As a consequence, perhydroxyl anions dominate the bleaching of regenerated bamboo fiber because of a large excess of hydrogen peroxide employed in the CCD experiments. This explains

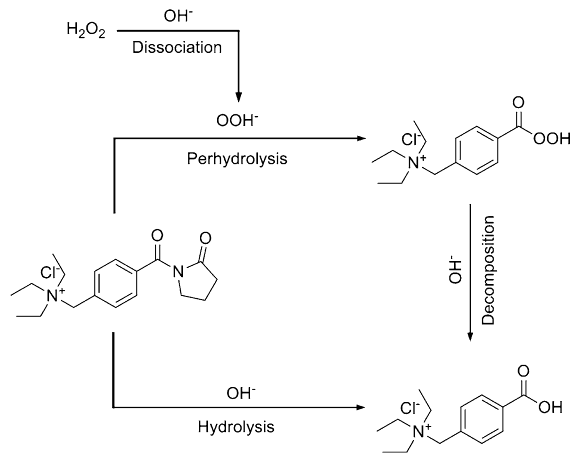

Fig. 8 Potential routes to perhydrolysis and hydrolysis of TBBC in alkali hydrogen peroxide solution

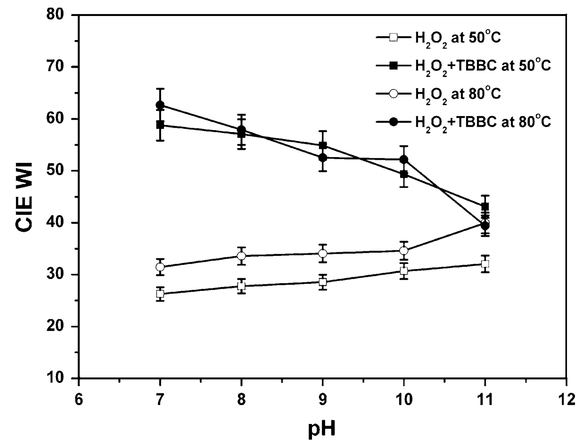

Fig. 9 Effect of pH on the resultant whiteness for varying conditions using equimolar amounts of TBBC and H_2O_2

why the typical features of hydrogen peroxide bleaching were observed in Figs. 4, 5, 6. Because hydrogen peroxide is not very active in short bleaching times and at temperatures below 95 °C, the effect of hydrogen peroxide concentration on the whiteness was relatively weak.

Effect of pH

In an attempt to address the lack of effectiveness of TBBC in the CCD experiment, regenerated bamboo fiber was bleached using an equimolar amount of TBBC and H_2O_2 (14.7 mmol/L) at various pH values. Figure 9 shows the effect of pH on Whiteness Index values of resultant bleached fabric. As expected, H_2O_2 bleaching excluding TBBC increased the whiteness values as the pH value increased. However, the increase in whiteness value was substantially reduced at low temperature.

When TBBC was included in H_2O_2 bleaching, the whiteness values increased substantially at pH 7.0. This indicates that H_2O_2 was activated by TBBC and the bleaching was effective at both 50 and 80 °C. However, the whiteness values dropped as pH value increased from 7.0 to 11.0 for both temperatures, indicating that hydrolysis of TBBC or decomposition of TBBC peracid occurs at a higher pH value. Therefore, the pH value and equimolar concentrations of peroxide and activator appear to be key factors in bleaching. Also, the effect of pH in Fig. 9 corroborates the conclusion from the CCD model that TBBC is not a significant factor at pH 11.5.

Bleaching performance

Regenerated bamboo fiber was bleached by TBBC–activated bleaching system under the conditions of 5 g/L TBBC, 1.43 g/L H_2O_2 (35% w/w), 50 °C, pH 7.0 and 30 min, compared with conventional peroxide bleaching system under the conditions of 6 g/L H_2O_2 (35% w/w), 100 °C, pH 11.5 and 30 min. The bleaching performances were evaluated by the whiteness values, shown in Fig. 10, and the degree of polymerization of bleached regenerated bamboo fiber, shown in Fig. 11. In each case, a control sample was included that underwent identical bleaching, except in the absence of hydrogen peroxide or activator. Degree of polymerization is a repeatable

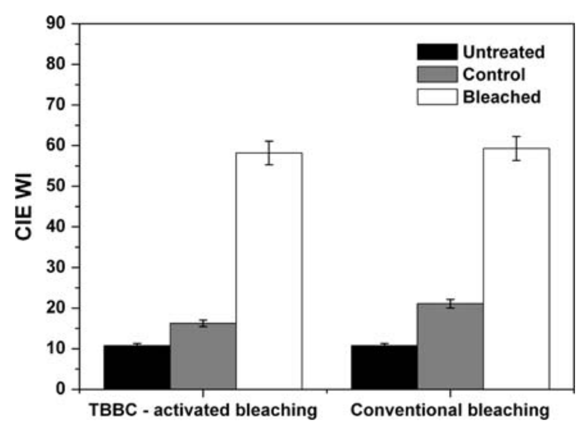

Fig. 10 Comparison of whiteness of regenerated bamboo fiber between the TBBC-activated bleaching and conventional bleaching

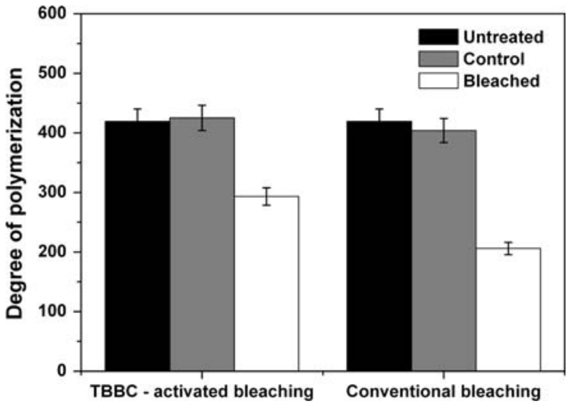

Fig. 11 Comparison of degree of polymerization of regenerated bamboo fiber between the TBBC-activated bleaching and conventional bleaching

quantitative method to indicate the level of change due to chemical processing and it is known to correlate with strength loss of yarn and fabric. Comparable whiteness was observed for TBBC-activated peroxide bleaching relative to the conventional bleaching method. In addition, the degree of polymerization was much higher for the TBBC-peroxide bleach system compared to the conventional bleach. Therefore, the use of TBBC enables effective bleaching of regenerated bamboo using relatively benign conditions with reduced fiber damage.

Conclusions

The analysis of variance of the central composite design of experiment revealed that, for the conditions investigated in the design, the TBBC concentration was not a significant factor in bleaching, possibly because TBBC lost its efficiency due to hydrolysis or decomposition at pH 11.5. The use of a large excess of hydrogen peroxide with TBBC did not increase whiteness substantially.

However, at pH 7 and 50 °C, TBBC-activated peroxide bleaching produced whiteness levels on regenerated bamboo fiber comparable to conventional peroxide bleaching, but with substantially decreased fiber damage. Neutral bleaching of bamboo fiber at 50 °C using equimolar amounts of TBBC and hydrogen peroxide was shown to be effective. These conditions may be also effective for bleaching other cellulosic fibers or protein fibers and fiber blends.

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