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Low temperature bleaching and dyeing properties of modified cellulose fabrics with triazine derivative

Kongliang Xie[a,*], Chuanguo Hu[b], Xufang Zhang[a]

[a] College of Chemistry, Chemical Engineering and Biotechnology, Donghua University, Shanghai 201620, PR China
[b] Zhejiang Jiaxin Silk Corporation Ltd., Jiaxing 314000, PR China

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ABSTRACT

Cellulose fabrics were modified with triazine derivative containing multi-cationic groups, 2,4,6-tri [(2-hydroxy-3-trimethyl-ammonium)propyl]-1,3,5-triazine chloride (Tri-HTAC). Low temperature bleaching of modified cellulose fabrics with tetra acetyl ethylene diamine (TAED), tetra acetyl glycineurea (TAGU) or (N-[4-triethylammoniomethyl]-benzoyl) caprolactam chloride (TBCC) as H_2O_2 activator was investigated. Bleaching properties and mechanisms of three activators were discussed. The dyeing properties and colorimetric data of the bleaching fabrics were investigated. The results show that the modified cellulose fabrics with Tri-HTAC could be bleached with hydrogen peroxide at low temperature, using activators. All whiteness indexes and wettabilities of modified fabrics under low temperature bleaching conditions were significantly higher than those of control samples. The exhaustion of the bleached modified fabrics was significant higher than that of unmodified cellulose fabric. Color degree of all bleaching fabrics with activators was also improved. The fastnesses of bleached fabrics were excellent. The quality of modified fabrics bleached under low temperature activator systems was improved. It has potential application in new composites and cleaner production of cellulose biomaterials.

1. Introduction

Cellulose fiber is one of the biomaterials from nature and has wide application in different production due to biodegradability, biocompatibility and nontoxicity (Hou, Zhou, & Wang, 2009; Jung, Wolters, & Berlin, 2007). Some properties of cellulose fabric are dependent on the chemical structure modification. Many approaches have been reported to modify cellulose for making materials with enhanced performance in some specific areas (Hou, Yu, & Chen, 2010; Pisuntornsug, Yanumet, & O'Rear, 2002; Xie, Gao, & Zhao, 2010). In our previous work, modification of cellulose fabrics with triazine derivative containing multi-cationic groups, 2,4,6-tri [(2-hydroxy-3-trimethyl-ammonium)propyl]-1,3,5-triazine chloride (Tri-HTAC) was reported (Xie, Hou, & Wang, 2008; Xie, Sun, & Hou, 2007). Tri-HTAC is an excellent and efficient grafting chemical for cellulose. It contains multi reactive groups and multi-cationic groups. After cellulose fabric was modified with the 1,3,5-triazine derivative, the modified cellulose exhibits different behaviors towards dyeing property compared with that of unmodified cellulose. Chemical modification of cellulose could change its chemical and morphological structures (Xie, Hou, & Sun, 2006). The modified cellulose would have potential application in nanomaterials and biofunctional materials.

In order to further improve the quality and property of modified cellulose for different aims, the cellulose fabrics modified need to be bleached. Non-cellulose substances have to be removed and natural pigments are discolored. Hydrogen peroxide bleaching systems can achieve excellent whiteness for cellulose fabrics and it is an environmental friendly process. However, the bleaching of cellulose fabrics with hydrogen peroxide requires high temperature at a high pH value. A large amount of energy is consumed and the aggressive treatment conditions frequently damage the cellulose macromolecular chain in bleaching process. How to reduce the consumption of energy and water, to achieve cleaner processing, is the key to sustainable development in textile dyeing industry (Agarwal & Bhattachachacharya, 2010; Hou, Chen, Dai, & Zhang, 2010; Hou, Yu, et al., 2010; Hou, Zhang, & Zhou, 2010; Jiang, Yuan, Bi, & Sun, 2010). Recently, some attempts have been made to investigate low temperature bleaching process for energy conservation. Much attention has been paid on the utilization of activators for hydrogen peroxide. To date, peroxide-activator bleaching systems and some activators, such as tetra acetyl ethylene diamine (TAED) (Shao, Huang, Wang, & Liu, 2010), nonanoyloxybenzene sulphonate (NOBS) (Shafie, Fouda, & Hashem, 2009), and (N-[4-triethylammoniomethyl]-benzoyl) caprolactam chloride (TBCC) are reported (Baillely, Hardy, & Guedira, 1997; Hou, Zhang, et al., 2010; Lee, Hinks, Lim, & Hauser, 2010; Lim, Lee, Hinks, & Hauser,

* Corresponding author. Tel.: +86 21 6779 2413; fax: +86 21 6237 8392.
E-mail address: klxie@dhu.edu.cn (K. Xie).

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2005). TAED is a nonionic compound and not soluble in water. NOBS is a anionic compound and TBCC is a cationic compound. Some performances of bleach activators in a cold pad-batch and hot peroxide bleaching of cotton have been evaluated. These compounds have been widely employed as bleaching activators in the detergent industry (Baillely et al., 1997; Qi & Yu, 2003). The compounds as activators for H_2O_2 can improve bleaching efficiency under mild conditions. Pre-cationization of cellulose fabrics with 3-chloro-2-hydroxypropyl trimethyl ammonium chloride to activate hydrogen peroxide also had been reported (Hashem, El-Bisi, Sharaf, & Refaie, 2009). Results obtained showed that pre-cationization of cellulose fabrics provides comparable fabric whiteness at much shorter reaction times and lower bleaching temperature. Low temperature bleaching using active systems for hydrogen peroxide is one of the effective processes to save energy and reduce consumption. These results are significant for the energy savings and the sustainable development (Rong, 2008).

In this paper, cellulose fabric was chemically modified with Tri-HTAC. Low temperature bleaching of modified cellulose fabrics with H_2O_2 used TBCC, TAED or tetra acetyl glycineurea (TAGU) as activator was investigated. The dyeing properties of bleaching fabrics were also discussed. It has practical and academic significance for achieving high-quality matrix of composites and cleaner production of cellulose materials.

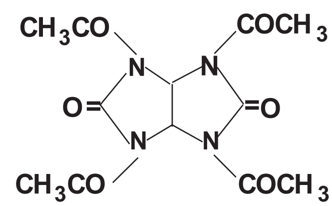
2. Experimental

2.1. Materials

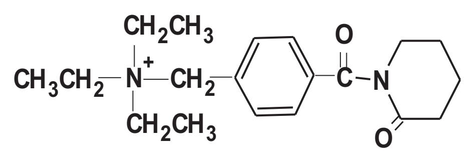
Desized cotton fabric, weight 230 g/m^2, was obtained from Shaoxing Jinqiu Textile Company, Shaoxing, China. The 1,3,5-triazine derivative, 2,4,6-tri [(2-hydroxy-3-trimethyl-ammonium)propyl]-1,3,5-triazine chloride (Tri-HTAC), was obtained from Modern Textile Institute, Shanghai, China. Low temperature activators, tetra acetyl ethylene diamine (TAED), tetra acetyl glycineurea (TAGU), and (N-[4-triethylammoniomethyl]-benzoyl) caprolactam chloride (TBCC) were obtained from National Engineering Research Center for Dyeing and Finishing of Textiles, Shanghai, China. Their chemical structures are shown in Scheme 1.

Reactive Red BF-3B was obtained from Shanghai Eighth Dyes Chemical Company, Shanghai, China. Other chemicals used were obtained from Shanghai Chemical Reagent Plant, Shanghai, China.

Tetra acetyl ethylene diamine (TAED)



Tetra acetyl glycineurea (TAGU)



(N-[4-triethylammoniomethyl]-benzoyl) caprolactam chloride (TBCC)

Scheme 1. Chemical structures of three used activators.

2.2. Modification of cellulose fabric

Modification of cellulose fabric with Tri-HTAC was carried out according to the reference method (Xie, Liu, & Wang, 2009). Tri-HTAC was used at the concentration of 40 g/L and sodium hydroxide of 10 g/L added as catalyst. Cellulose fabric was treated with the solution at the liquor ratio being 1:10 and kept at room temperature for 10 h. The modified fabric was then washed with tap water until neutral and again washed in warm water using a domestic washing machine to remove unfixed compounds. The fabric was dried at ambient conditions.

2.3. Low temperature bleaching

The bleaching process was performed using the exhaustion method according to the reference method (Hou, Zhang, et al., 2010). The conditions of low temperature bleaching were activator 4 g/L, sodium hydroxide 1 g/L, and sodium silicate 2 g/L, and hydrogen peroxide 1–6 g/L, respectively. The bleaching process was performed at 60 °C for 60 min. The bleached sample was washed three times with hot water (80 °C) then three times with cold water and finally dried at ambient conditions.

The whiteness index, which expressed degree of sample whiteness, was measured on Color-Eye 7000A spectrophotometer (Gretag Macbeth, USA) by using the CIE method according to ISO 105-J02: 1997(E) standard.

Wettability was monitored. Three specimens in one group were tested. The length of specimen was 300 mm and the width being 50 mm. Three specimens in one group were hanged. The bottoms of samples were immersed in the distilled water at room temperature. After 30 min, the length of specimens with water was measured. The mean value of three specimens was calculated.

2.4. Dyeing of bleaching cellulose fabric

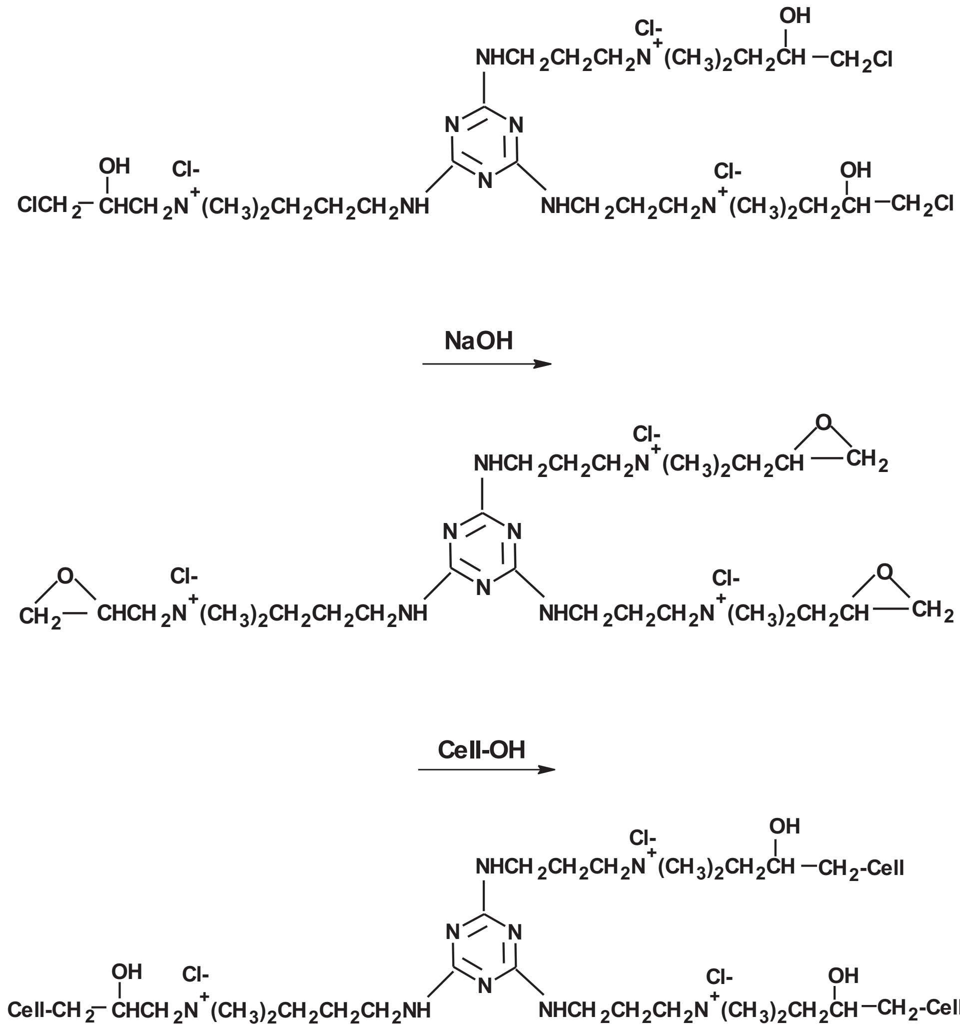
The modified cotton fabric was dyed according to the reference method (Xie et al., 2008). The liquor ratio was 1:15. Sodium chloride, 50 g/L, sodium carbonate, 10 g/L and dyes, 2% (o.w.f.) were used.

The exhaustion of dyes on fabric was calculated by measuring the absorbance of the residual dye bath liquor. The percentage of dye bath exhaustion (E (%)) was calculated according to Eq. (1)

$$E\ \ (\%) = \left[1 - \left(\frac{C_2}{C_1}\right)\right] \times 100 \qquad (1)$$

where C_1 and C_2 are the concentrations of the dyes in the dye bath before and after dyeing, respectively.

The color yield (K/S) and colorimetric data of the dyed fabric were determined by Color-Eye 7000A spectrophotometer (Gretag



Scheme 2. Grafted reaction mechanism of cellulose with Tri-HTAC.

Macbeth, USA). The dye absorbance was measured in the visible region of the spectrum from 400 nm to 700 nm and the reflectance at the wavelength of maximum absorption (λ_{max}) was used to calculate the color yield of dyed fabric by the Kubelka–Munk equation (Eq. (2))

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (2)$$

where K is the absorption coefficient of the substrate, S is the scattering coefficient of the substrate and R is the reflectance of the dyed fabric at λ_{max}.

2.5. Measurements

Color fastness was evaluated according to the respective international standards: fastness to rubbing, ISO 105-X12 (1993); fastness to washing, ISO 105-C04 (1989); fastness to perspiration, ISO 105-E04 (1994).

3. Results and discussion

3.1. Low temperature bleaching of modified cellulose fabric

2,4,6-Tri [(2-hydroxy-3-trimethyl-ammonium)propyl]-1,3,5-triazine chloride (Tri-HTAC) is a compound, which contains the multi-reactive and multi-cationic groups. Tri-HTAC was able to form covalent bonds with cellulose fiber under alkaline conditions. The modified cellulose fiber formed new molecular structures containing a lot of cationic groups. Graft reactions of cellulose are shown in Scheme 2.

Chemical graft reaction and characterization of cellulose with Tri-HTAC were reported in previous works (Xie et al., 2006, 2007). The whiteness index of the modified fabrics with Tri-HTAC but unbleached was 45.33.

Three compounds as activators for H_2O_2, TBCC, TAED, and TAGU, were employed in the low temperature process. Effects of varied activators on whiteness index of modified fabrics under low temperature bleaching conditions were investigated. The modified cellulose fabrics were bleached with 1–6 g/L hydrogen peroxide at 60 °C for 60 min, respectively. The results are shown in Figs. 1–3, respectively. They indicate that all whiteness indexes of modified and unmodified fabrics bleached under low temperature were significantly higher than that of unbleached modified sample (45.33). It can be seen that the modified cellulose fabrics could be bleached at low temperature 60 °C, using three activators, respectively. Meantime, Fig. 1 also indicates that the whiteness index of the modified fabric was slightly higher than that of unmodified fabric when TBCC was applied as an activator. Figs. 2 and 3 show that the whiteness indexes of the modified fabrics were similar with that of unmodified cellulose with TAED or TAGU as activator, respectively. The phenomenon shows that cationic groups of modified cellulose

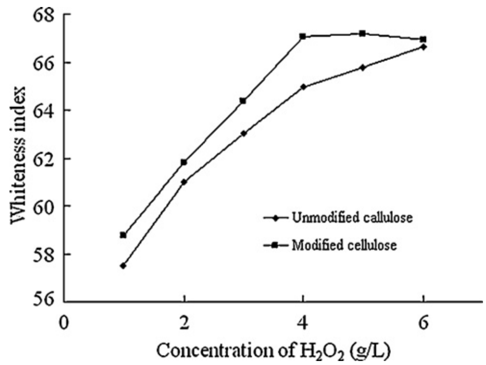


Fig. 1. Effect of hydrogen peroxide concentrations on whiteness with TBCC.

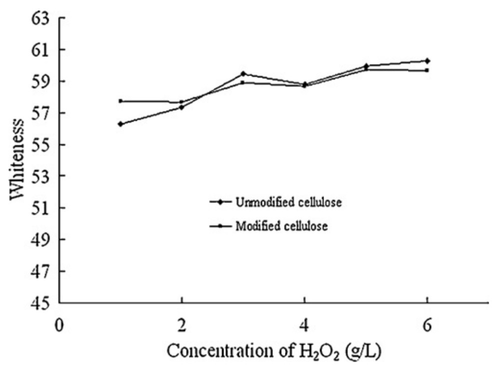


Fig. 2. Effect of hydrogen peroxide concentrations on whiteness with TAED.

improved bleaching reaction in the presence of TBCC at low temperature. However, the cationic groups of modified cellulose did not obviously affect bleaching reaction in the presence of TAED or TAGU.

Hydrogen peroxide is main component in bleaching system and plays an important role in fabric bleaching. The results in Fig. 1 indicate that the whiteness indexes of bleached fabrics sharply increased with increasing the concentration of hydrogen peroxide, from 1 g/L to 6 g/L in the presence of TBCC at low temperature. The whiteness indexes of bleached fabrics were similar with increasing the concentration of hydrogen peroxide in the presence of TAED at low temperature. However, the whiteness indexes of bleached fabrics were slight decreased with increasing the concentration of hydrogen peroxide in the presence of TAGU at low temperature. Compared with the whiteness indexes of bleached fabrics with different activators at low temperature, TBCC low temperature system for modified cellulose fabrics was efficient.

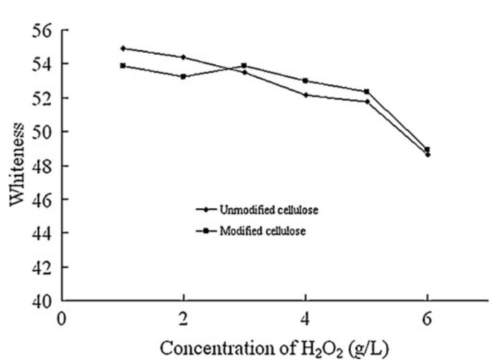


Fig. 3. Effect of hydrogen peroxide concentration on whiteness with TAGU.

Table 1
Wettability of bleaching cellulose fabrics.

Activator	Hydrogen peroxide (g/L)					
	1	2	3	4	5	6
Unmodified control without activators	4.3	4.2	4.4	4.5	4.5	4.7
Modified control without activators	4.5	4.7	4.6	4.5	5.3	5.5
Bleaching with TBCC	11.8	11.7	10.9	10.8	11.3	11.4
Bleaching with TAED	11.8	11.9	11.4	11.4	11.6	12
Bleaching with TAGU	12.0	12.1	12.0	11.9	12.1	11.9

Table 2
Exhaustion of dyes on the cellulose fabrics.

Samples	Hydrogen peroxide (g/L)					
	1	2	3	4	5	6
Unmodified control	69.31	69.34	68.73	69.56	69.55	69.01
Modified control	89.65	89.34	90.11	90.28	89.75	88.92
Bleaching with TBCC	91.25	91.36	92.26	91.76	92.82	91.79
Bleaching with TAED	92.45	93.59	94.78	92.6	93.51	92.92
Bleaching with TAGU	92.83	93.62	93.96	92.58	93.84	93.81

3.2. Wettability of the bleaching fabric and mechanism of bleached reaction

Wettability is an important parameter for bleaching fabrics. The better the wettability of the fabrics, the higher the water absorbency of the fabrics is. The wettabilities of the bleaching fabrics with and without activators at low temperature 60 °C are listed in Table 1. It shows that wettabilities of the bleached fabrics of the unmodified and modified control samples without activator were both low. The wettabilities of the bleached modified fabrics with activator were obviously higher than those of the bleached modified fabrics without activator. The results show that the activators played very important role in low temperature systems.

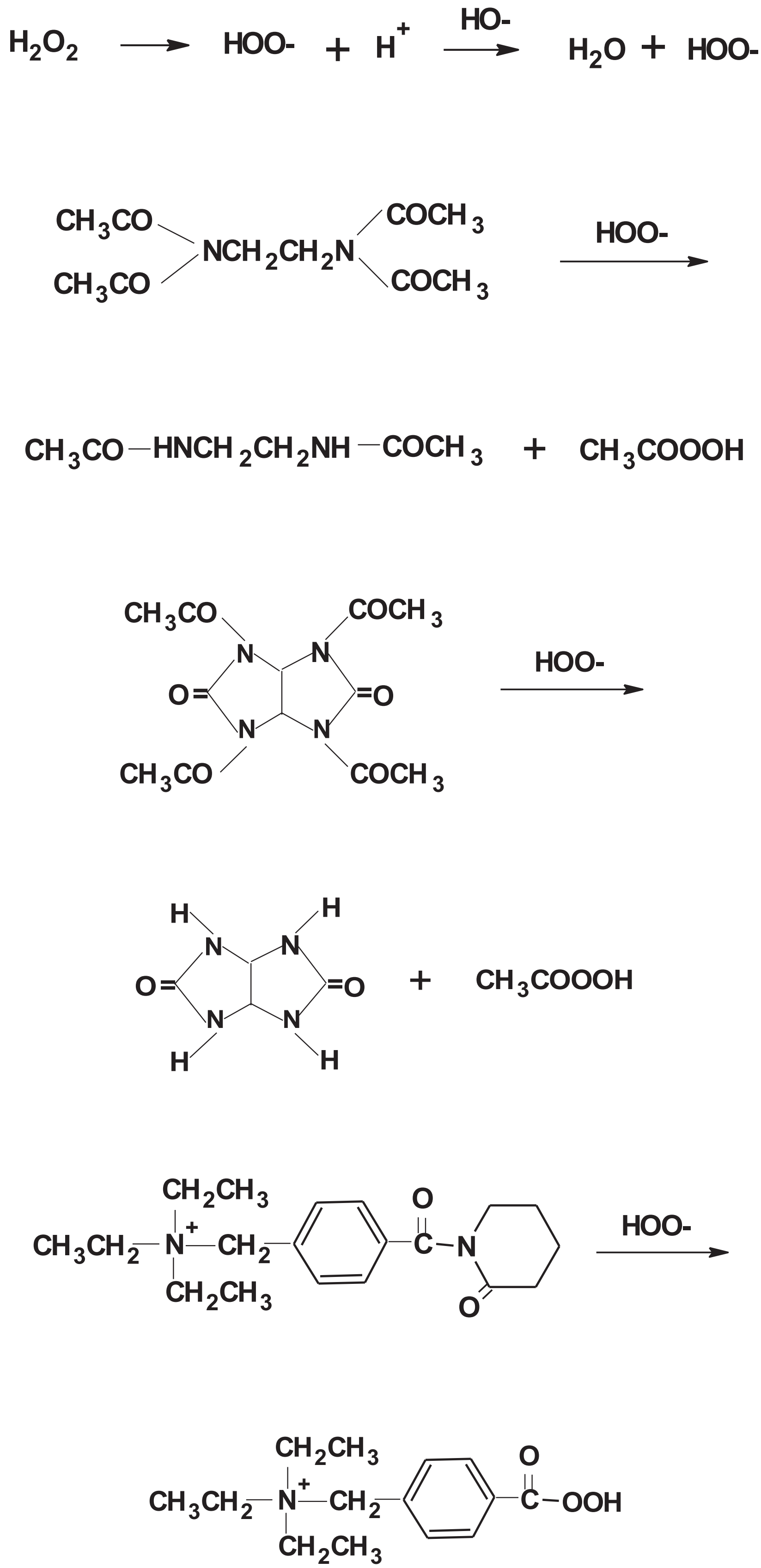
It is well known that peracids, such as peracetic acid, show stronger oxidative bleaching ability than hydrogen peroxide. In the bleaching process of cellulose fabrics, hydrogen peroxide liberates perhydroxyl anion (HOO^-) in alkali condition. Then peracids are formed by perhydrolysis reaction between the activators and HOO^- anion. The peracetic acid is formed by the reaction of TAED or TAGU with perhydroxyl anion. HOO^- could also react with TBCC and cationic benzoyl hydroperoxide is obtained. In fact, bleaching activators are peracid precursors, which generate peracids in situ in an alkaline hydrogen peroxide solution. The formed intermediate products, peracids, are safe and efficient bleaching agent at mild condition. The mechanisms of bleaching catalyst are shown in Scheme 3.

3.3. Dyeing properties of the bleached cellulose fabrics

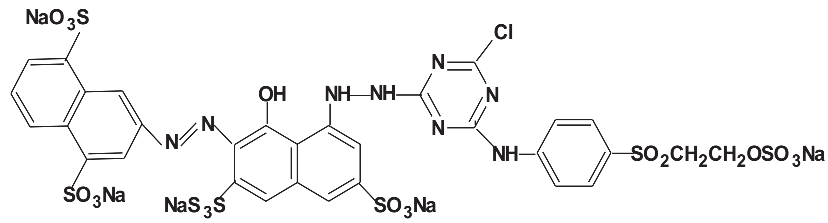
After cellulose fabrics modified with multi cationic groups were bleached at low temperature systems, some surface properties of the fabrics could be changed. In this work, dyeing properties of the beached cellulose fabrics were discussed. Reactive dye, Reactive Red BF-3B, was employed in the experiment. Chemical structure of Reactive Red BF-3B is shown in Scheme 4.

3.3.1. Dye exhaustion and color yields of the cellulose fabrics

Unmodified control, modified control, and the bleached modified fabric with the presence of activator were dyed with Reactive Red BF-3B. Unmodified control and modified control were bleached without activators in low temperature systems. The exhaustions of the dyes on fabrics were calculated and are summarized in Table 2. The results indicate that the exhaustions of modified control and bleached modified fabrics with the presence of activators were significant higher than those of unmodified samples. It could be



Scheme 3. Mechanism of low temperature bleaching.



Scheme 4. Chemical structure of Reactive Red BF-3B.

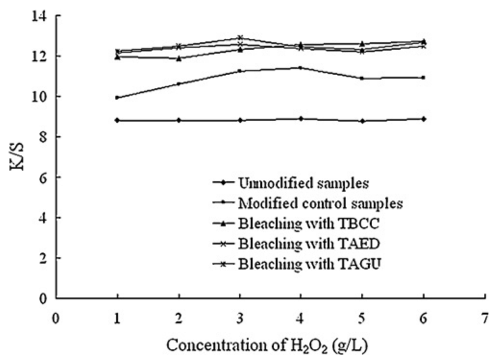


Fig. 4. Effect of various activators on *K*/*S* of the dyed fabrics.

contributed to the modified cellulose possessing a lot of cationic groups in the fiber surface. Chemical structure of modified cellulose was not damaged during low temperature bleaching process. Compared with modified control, the exhaustions of the modified fabrics bleached with the presence of activators were slightly improved. The bleached fabrics further improved their wettability and dye solution absorbency.

K/*S* represented color yield of the dyed fabrics. *K*/*S* of the dyed fabrics was determined and is shown in Fig. 4. It shows that the color yield of modified control and the modified samples bleached with the presence of activators was obviously higher than those of unmodified samples. The results of *K*/*S* were in agreement with those of their exhaustions.

3.3.2. *Colorimetric data of dyed fabrics*

The color parameters *L*, *a*, *b*, of the dyed fabrics were calculated by the tristimulus values *X*, *Y* and *Z*. *L* refers to brightness–darkness with values from 100 to 0 representing white to black. The *a* values run from negative (green) to positive (red). The *b* values run from negative (blue) to positive (yellow). The *C* is color degree of the dyed fabrics. ΔL, Δa and Δb are the difference in the color parameters of the dyed fabrics bleached with and without the activators. Colorimetric data for modified control and the modified samples bleached with the presence of activators are summarized in Table 3. Because the modified control sample was bleached without activator in low temperature system, its bleached effect was not good. So, the L^* value and the *C* value of the modified control were the smallest in all samples. It can be seen that the brightness of all bleached fabrics with activators were obviously improved. Redness/greenness (a^*) values of beached samples with activators were higher than that of the control sample. Yellowness/blueness (b^*) values were also greater than that of the control sample. Color degrees of all bleaching fabrics with activators were also obviously improved. It can be seen that low temperature bleaching of modified fabrics benefited to improve dye properties and quality of cellulose fabrics.

Table 3
Colorimetric data of modified control and the modified fabrics bleached with activators.

Samples	*L*	*a*	*b*	*C*	ΔL	Δa	Δb
Modified control	44.28	57.10	−3.26	57.19	0	0	0
Bleaching with TBCC	44.84	58.12	−2.77	58.18	0.56	1.02	0.49
Bleaching with TAED	44.88	58.29	−2.82	58.34	0.60	1.19	0.44
Bleaching with TAGU	45.16	58.02	−2.81	58.32	0.88	0.92	0.45

Table 4
Fastness properties of modified control and bleached modified fabrics with activators.

Samples	Fastness to rubbing		Fastness to washing		Fastness to perspiration	
	Dry	Wet	SC[a]	SW[b]	SC[a]	SW[b]
Modified control	4–5	3	3–4	3–4	3–4	3–4
Bleaching with TBCC	4–5	4	4	4	4	4
Bleaching with TAED	4–5	4	4	4	4	4
Bleaching with TAGU	4–5	4	4	4	4	4

[a] SC, staining on cotton.
[b] SW, staining on wool.

3.3.3. *Fastness properties*

The fastness properties of the dyed cellulose fabrics are summarized in Table 4. It indicates wet rubbing fastness, washing fastness and fastness to perspiration of the modified samples bleached with activators were better than those of control sample. The fastness properties of dyed cellulose fabrics bleached with the presence of activators at low temperature were excellent.

4. Conclusions

The modified cellulose fabrics with Tri-HTAC could be bleached with hydrogen peroxide at low temperature 60 °C, using activators. Three compounds as activators, TBCC, TAED, and TAGU, had excellent active properties for H_2O_2. All whiteness indexes and wettabilities of modified fabrics under low temperature bleaching conditions were significantly higher than those of control sample. TBCC low temperature system for modified cellulose fabrics was the most efficient among three activator systems. The whiteness indexes of bleached modified fabrics sharply increased with increasing the concentration of hydrogen peroxide used TBCC as activator. Chemical structure of modified cellulose could not be damaged during low temperature bleaching process. The exhaustions of modified control and the bleached modified fabrics were significant higher than that of unmodified cellulose fabric. Compared with modified control, the exhaustions of the bleached modified fabrics with activators were slightly improved. Color degree of all bleached fabrics with activators was obviously improved. The fastnesses of dyed modified cellulose fabrics were excellent. The modified fabrics, which were bleached under low temperature activator systems, improved dyeing property and quality of cellulose fabric.

Low temperature bleaching of cellulose fabrics with H_2O_2 in the presence of activator could save a large amount of energy. However, as bleaching activators for cellulose fabrics have not been widely applied in the textile industry. It is important key to reduce the cost used in their practical industrial application. The new technique to reduce the quantity of activators during applying also needs to be further investigated. In the future bleaching process should be environmentally safe, cost-effective and sparing of energy.

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