

Combined Scouring and Bleaching of Cotton/Linen Blends by a Near-Neutral Activated Peroxide System

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¹ Wuhan Textile University,
College of Advanced Textile,
Wuhan 430200, China

² Wuhan Textile University,
College of Chemistry and Chemical Engineering,
Wuhan 430200, China

³ Wuhan Textile University,
Hubei Key Laboratory of Biomass Fibres
and Eco-Dyeing & Finishing,
Wuhan 430200, China,
* e-mail: 2014020@wtu.edu.cn

Abstract

The simultaneous scouring and bleaching of cotton/linen blends was performed in a near-neutral activated peroxide system (pH = 7.2). A response surface quadratic model (RSQM) based on the central composite design (CCD) was established to investigate and optimise the bleaching performance. Research results showed that hydrophobic impurities in the blends had little impact on the bleaching. Temperature was the most significant factor affecting the fabric's whiteness index (WI), followed by the concentration and duration of the activator tetraacetythylenediamine (TAED). The optimised process was performed at 70 °C for 40 min, incorporating 20 mmol/l of TAED and 42 mmol/l of H₂O₂. Compared with the conventional process carried out at 95 °C for 60 min and adding 80 mmol/l H₂O₂, the activation process provided the fabric with comparable WI and wettability, a smoother and cleaner microcosmic surface, as well as the advantages of saving energy and preserving fabric. This study confirmed the feasibility of an efficient one-step process for neutral scouring and bleaching.

Key words: low-temperature bleaching, TAED, peracetic acid, cotton, linen.

Introduction

Natural cellulosic greige contains many coloured impurities which significantly impair a fabric's whiteness. Usually, these undesired impurities should be removed via bleaching to give the fabric a white appearance prior to dyeing and finishing. Traditional bleaching is performed using hydrogen peroxide (H₂O₂) at approximately boiling temperature and in strongly alkaline media [1]. Such harsh conditions bring about large energy consumption and severe fibre damage. Therefore, a low-temperature bleaching process is eagerly expected by the whole textile and apparel industry.

Peracids are more kinetically active oxidants and have been confirmed to bleach cellulosic fibres in a low-temperature medium [2]. Nevertheless, they are seldom directly used in industrial bleaching when considering the risks associated with preparation, storage and transport [3]. At the same time, peracids can be obtained in-situ via the reaction between H₂O₂ and bleaching activators, thus bleaching activators make low-temperature bleaching based on peracids possible [4].

Nowadays, the most commonly used bleaching activator is tetraacetythylenediamine (TAED). Its approved activation mechanism during bleaching

is revealed in **Figure 1** [5-7]. TAED reacts with two molecules of hydrogen peroxide ion (HO₂⁻) in the nucleophilic manner to generate one molecule of diacetythylenediamine (DAED) with the release of two molecules of peracetic acid (PAA). PAA functions as an oxidant destroying the coloured substances of the fibre at low temperature (about 70 °C). In spite of some activators with another structure having been reported [8-13], TAED continues to attract researchers' extensive attentions. Related works have mainly focused on investigating the in-depth activation bleaching mechanism and low-temperature bleaching performances of various fibres [14-16], especially those with poor heat and alkali resistance [17-19]. Additionally, TAED is an industrial product with the advantages of being non-toxic, non-sensitising and biodegradable [2]. Therefore, to promote the industrialisation of low-temperature

bleaching, TAED is still a preferred activator.

In previous studies it was verified that TAED-activated bleaching functioned more effectively for cotton under near-neutral conditions [14, 20]. The conclusion was based on the fact that the fabric had a good wicking capability before bleaching. But in most cases the wicking capability is poor for cotton greige due to some hydrophobic impurities, such as wax, pectin, fat, etc [1], which may prevent the penetration of bleaching agents. Moreover, the near-neutral condition is not conducive to the removal of hydrophobic impurities. To develop an efficient short process combining scouring and bleaching, it is necessary to investigate the neutral bleaching effect in the presence of hydrophobic impurities. For this purpose, desized, unscoured and unbleached cotton/linen blended woven

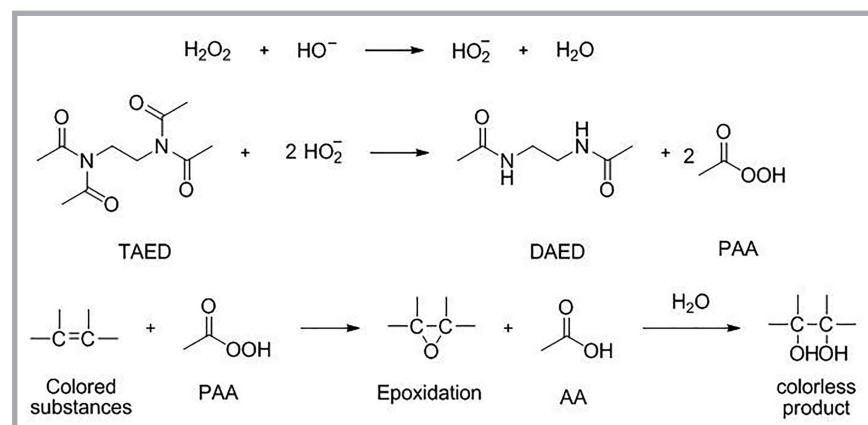


Figure 1. Conceivable activated bleaching mechanism of TAED.

fabric was chosen. Generally, linen contains more impurities compared with cotton, especially lignin in significant quantity [21], thus the impact of hydrophobic impurities on bleaching may be more evident in the present study.

A one-step process for scouring and bleaching in the TAED/H₂O₂ activation system was performed under near-neutral conditions. The central composite design (CCD) was used to analyse the performance in bleaching. CCD experimental data were fitted to create a response surface quadratic model (RSQM) describing the fabric's whiteness index (WI). Then, the low-temperature activated bleaching was compared with traditional H₂O₂ bleaching in terms of WI, the capillary effect (CE), strength of the fabric, and the theoretical energy consumption (ΔQ) of the process. Finally, SEM was performed to observe the micro-morphology of cotton/linen blended fabric after having undergone the activated bleaching.

Materials and methods

Materials

Cotton/linen blended woven fabric, 230 g/m², was purchased from Yixing Kaiyue Textiles Co., Ltd. The blending ratio of the cotton to linen was 45/55. TAED (cream coloured granule, purity 91.3%) was generously provided by Zhejiang Jinke Peroxides Co., Ltd. H₂O₂ (30% w/w) was purchased from Sino-pharm Chemical Reagent Co., Ltd. Sodium hydroxide (NaOH), H₂O₂ stabiliser disodium ethylene diamine tetraacetate (EDTA-2Na), sodium dihydrogen phosphate (NaH₂PO₄) and disodium hydrogen phosphate (Na₂HPO₄) used for the preparation of buffer solution, were of analytical reagent grade. The non-ionic wetting agent was of industrial grade. Except for rinsing, deionised water was used for all experiments.

CCD of experiments

Experimental design and statistical analysis were performed using Design-Expert (version 8.0.5.0) statistical software (Stat-Ease Inc., USA). Three variables were assessed, each including five levels (-1.68, -1, 0, 1, 1.68). The actual levels of variables are listed in **Table 1**. As shown in **Table 2**, the total number of experiments was 20, including eight factorial points (coded as -1 or 1), six axial points (coded as -1.68 or 1.68) and six replica-

Table 1. Code and actual levels of design variables. **Note:** [TAED] – concentration of tetraacetythylenediamine, mmol/l; T – temperature, °C; t – duration.

Variable	Unit	Coded and actual levels				
		-1.68	-1	0	1	1.68
[TAED]	mmol/l	3.18	10	20	30	36.82
T	°C	16.36	30	50	70	83.64
t	min	6.36	20	40	60	73.64

Table 2. Design matrix and CCD response data. **Note:** [TAED] – concentration of tetraacetythylenediamine, mmol/l; T – temperature, °C; t – duration, min; WI – whiteness index.

Standard	Run	[TAED]	T	t	Hunter WI
6	1	30	30	60	46.93
19	2	20	50	40	51.72
18	3	20	50	40	51.31
7	4	10	70	60	51.79
17	5	20	50	40	50.31
8	6	30	70	60	55.17
11	7	20	16.36	40	46.52
14	8	20	50	73.64	52.26
9	9	3.18	50	40	48.17
3	10	10	70	20	50.17
16	11	20	50	40	51.34
13	12	20	50	6.36	47.71
5	13	10	30	60	47.59
10	14	36.82	50	40	52.75
12	15	20	83.64	40	54.77
4	16	30	70	20	54.29
20	17	20	50	40	51.65
2	18	30	30	20	47.70
15	19	20	50	40	52.21
1	20	10	30	20	46.47

tions of the centre point (coded as 0). WI of the treated fabrics was monitored as the response variable.

One-step process for scouring and bleaching

All experiments were carried out in sealed and conical flasks immersed in a universal dyeing machine (Xiamen Rapid, China). The weight of each fabric sample was 4 g, and the ratio of the liquor-to-fabric was 25:1. The treatment solution incorporated TAED added according to the levels shown in **Table 1**, H₂O₂ was added with a 2.1:1 molar ratio to TAED, 1g/l of EDTA-2Na, 2 g/l of the wetting agent, and 0.2 M of a phosphate buffer (NaH₂PO₄/Na₂HPO₄). The H₂O₂ slightly exceeded its stoichiometric amount to drive the generation of PAA to completion. The phosphate buffer was used to maintain the solution medium at pH 7.2 during the process. The fabrics were immersed in a series of treatment solutions. Then the solutions were heated to a required temperature at a rate of 5 °C/min, and the treatment continued

for a required time. After the treatment, the fabric was rinsed thoroughly with warm tap water (70 °C) for 3 min and cold tap water (20 °C) for 3 min, and then dried in the open air.

For comparison purposes, the conventional process was performed using a solution containing 80 mmol/l of H₂O₂, 9 g/l of H₂O₂ (30%, w/w), 2 g/l of NaOH, 1 g/l of EDTA-2Na, and 2 g/l of wetting agent at 95 °C for 60min. After the treatment, the fabric was treated as mentioned earlier.

Analytical methods

WI

WI of the fabric was directly measured with a 110 reflectance spectrophotometer (Datacolor, USA) using a D₆₅ illuminant and 10° standard observer. Each sample was folded twice, which gave a thickness of four layers of fabric. An average of three readings was calculated for each sample.

CE

CE of the fabric was measured according to the Chinese standard method

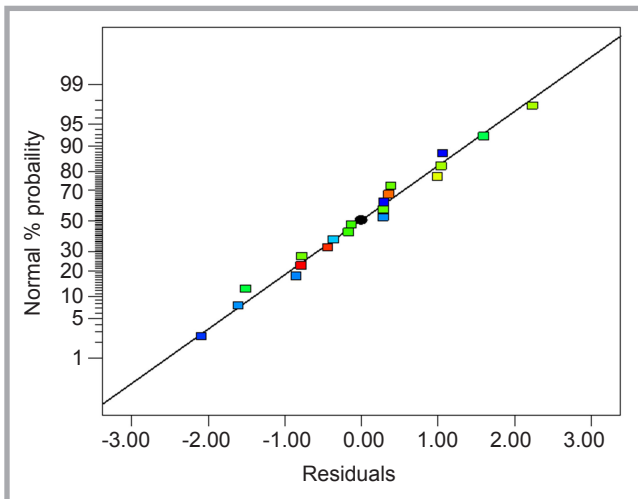


Figure 2. Normal probability plot of residuals.

(FZ/T01071-2008, “test method of capillary effect for textiles”) to evaluate the water absorbency of the fabric. A sample of 25 cm × 4 cm size was prepared and left in the laboratory at constant temperature and humidity (20 °C, 65%) for 4 h. A YG871 capillary effect tester (Shanghai Precision Instruments, China) was used to test the height of the aqueous solution which rose along the fabric within 30 min.

Fabric strength

Fabric strength was tested according to the Chinese standard method (GB/T3916-1997, “Measurement of Breaking Strength and Elongation at Break for Textiles, Yarn from Packages and Single Yarn”). The test was performed using a YG028 universal material testing machine (Wenzhou Fangyuan, China).

ΔQ

ΔQ for heating the treatment solution contained within the fabric being treat-

ed was determined by Equation (1). T_1 and T_2 are the starting and final temperature, respectively. At a liquor ratio of 1:25, if the solution contains 1 kg of fabric, 25 kg of liquor will be used. C_1 , C_2 and C_3 are the specific heat capacity of cotton (1.296 kJ/[kg·°C]), linen (1.340 kJ/[kg·°C]) and water (4.186 kJ/[kg·°C]), respectively. Since the concentration of chemical auxiliaries in the solution was very low, the specific heat capacity of water was almost equivalent to that of the treatment solution and can be used to calculate the ΔQ. Irrespective of the heat loss, this is a simplified method to estimate the energy required to raise the temperature of the treatment solution containing 1 kg of fabric [9].

$$\Delta Q = (45\%C_1 + 55\%C_2 + 25C_3) \times (T_2 - T_1) \quad (1)$$

SEM

An SEM test was carried out by mounting the fabric sample on a stub using double-sided adhesive tape and coating

with gold. The sample was then viewed using a PW-100-012 scanning electron microscope (Phenom, Netherlands) at a magnification of 5000.

Results and discussion

CCD Analysis

To investigate the bleaching effect of unscoured cotton/linen blends in near-neutral media, the interactions of three key factors, i.e., [TAED], temperature (T), duration (t) and the fabric’s WI, as well as the optimisation of the whole process were researched with CCD, which had been successfully applied in the statistical analysis of an activated bleaching system [20, 22-25].

The WI data measured are shown in Table 2 and were fitted to create a RSQM. Analysis of variance (ANOVA) for the RSQM is summarised in Table 3. A RSQM p-value less than 0.0001 and an F-value of 23.26 show the model is significant. The lack of fit is insignificant (p = 0.1917) relative to the error. The insignificant lack of fit indicates that the quadratic model is adequate to describe the actual response. The RSQM was also diagnosed by the normal probability plot of residuals to assess the error normality. As shown in Figure 2, all data points were close to a straight line, suggesting that residuals are normally distributed.

Variables effects on WI

In Table 3, values of “Prob > F” less than 0.05 indicate model terms are significant. In this case, [TAED], T, t, [TAED] * T and t^2 were the significant model terms. The extent of the significance of these five model terms can be ranked in terms of F values. It can be found that T was the most significant variable affecting WI, followed by [TAED] and t. The model terms [TAED] * T and t^2 respectively indicate that the interaction between [TAED] and T significantly affects WI as well as the quadratic relationship between t and WI. The 3D response surfaces of WI predicted from the RSQM are shown in Figure 3. The effects of [TAED], T, t and [TAED] * T on the bleaching were successively analysed as follows.

Effect of [TAED]

As shown in Figure 1, the oxidation ability of the activation system with respect to coloured substances depends on

Table 3. ANOVA for the response surface quadratic model.

Source	Sum of squares	df	Mean square	F value	P-value Prob>F
Model	139.14	9	15.46	23.26	<0.0001
[TAED]	18.22	1	18.22	27.41	0.0004
T	98.11	1	98.11	147.63	<0.0001
t	8.08	1	8.08	12.15	0.0059
[TAED]*T	6.00	1	6.00	9.03	0.0132
[TAED]*t	0.86	1	0.86	1.30	0.2806
T*t	0.58	1	0.58	0.87	0.3731
[TAED] ²	2.31	1	2.31	3.48	0.0916
T ²	1.62	1	1.62	2.44	0.1494
t ²	4.66	1	4.66	7.01	0.0244
Residual	6.65	10	0.66	–	–
Lack of Fit	4.63	5	0.93	2.29	0.1917
Error	2.02	5	0.40	–	–
Cor Total	145.78	19	–	–	–

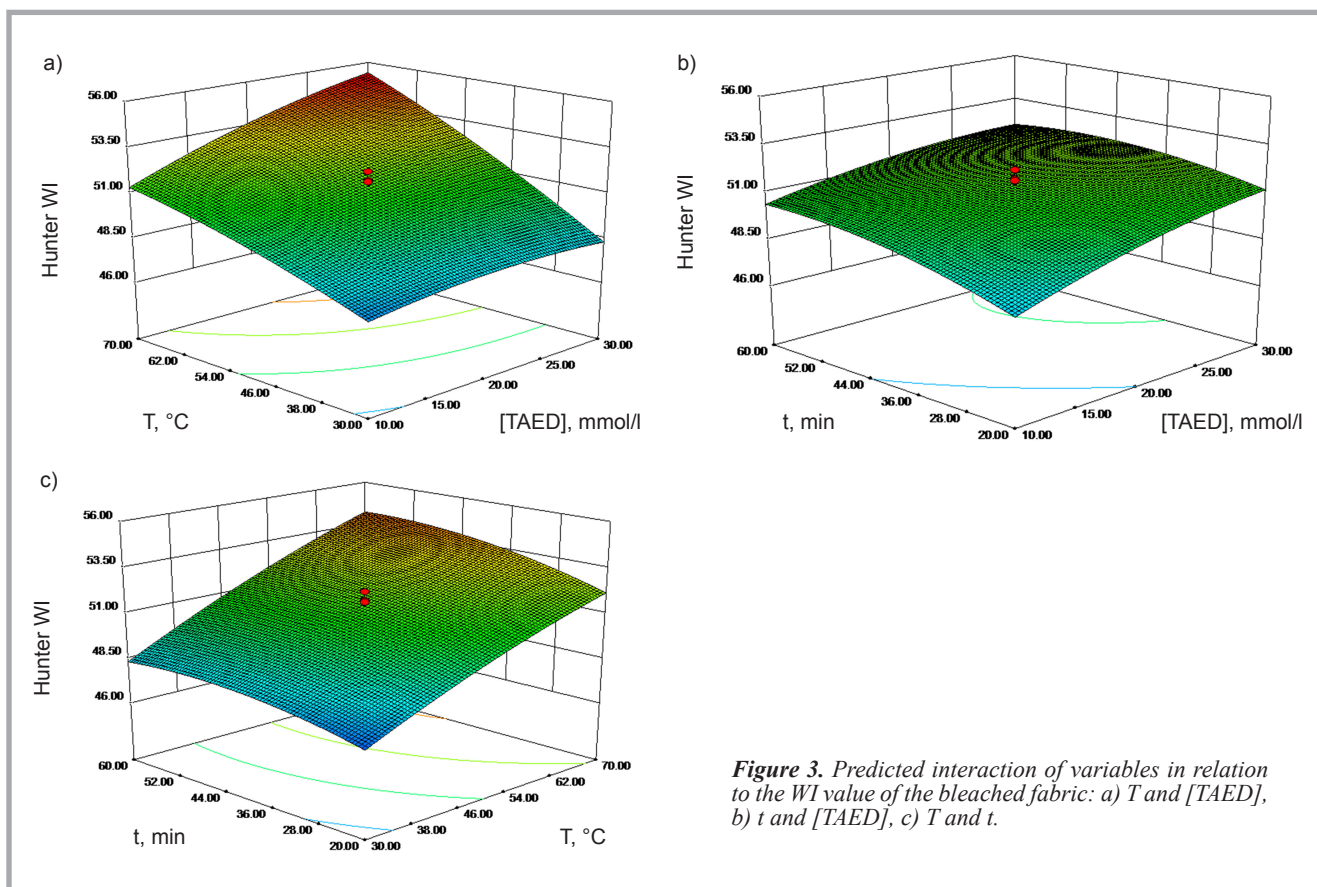


Figure 3. Predicted interaction of variables in relation to the WI value of the bleached fabric: a) T and [TAED], b) t and [TAED], c) T and t.

the amount of PAA generated. TAED is the precursor of PAA, thus it is the essential component in this system. It can be seen from **Figure 3.a** and **3.b** that, WI evidently increased with the increasing of TAED in a relatively low range. But WI promotion became flat when TAED reached a higher amount. This indicates that the PAA formed can still oxidise the coloured substances effectively to improve the fabric's WI in the presence of hydrophobic impurities. Meanwhile, the strong oxidation action is also beneficial to enhance the wicking capability of cellulosic fibre, thereby providing a path for the permeation of bleaching agents. The less obvious increase in WI with more TAED is probably attributed to the fact that the most coloured substances had already been destroyed by PAA. In this case, WI cannot be further promoted, even if more TAED is used.

Effect of temperature

According to the ANOVA in **Table 3**, temperature was the most significant variable to improve WI, whose effect even surpassed TAED. Although it has been verified that the TAED-activated system can perform low-temperature bleaching, it is shown in **Figure 3.a** and **3.c** that there was no obvious tendency of WI being flat in the temperature range analysed. That is to say, the bleaching effect would continue to increase under high-temperature conditions. Heating gives the activated system a direct energy driving the generation of APP and the oxidation of coloured substances to be more complete, which is the main reason for such a positive and significant impact of the temperature. Additionally, raising the temperature is favourable towards the removal of hydrophobic impurities, which then facilitates the bleaching.

Effect of duration

Based on the ANOVA in **Table 3**, duration was a less significant variable affecting WI compared with [TAED] and temperature. The duration of the process reflects the time required to complete the reactions shown in **Figure 1**. As shown in **Figure 3.b** and **3.c**, extending the time had little influence on WI with an increase in [TAED] or temperature, particularly at a high level of temperature. Thus, to improve the efficiency, shortening the duration is quite feasible when the process is carried out at higher temperatures. Such a short time required could be due to the fast speed of PAA generation and efficient oxidation of impurities. Besides, there was a quadratic relationship between the duration and WI, indicating that the bleaching effect will be further ensured when extending the range of time.

Table 4. Comparisons between TAED-activated and conventional scouring/bleaching of cotton/linen blended fabric. **Note:** ^a Control sample was cotton/linen blended greige; ^b pH value of the conventional processing bath was 11.8; ^c ΔQ was calculated to raise the temperature of the bath from 25 °C to those required by the two different processes.

Sample	Treatment conditions		WI	CE, cm/30min	ΔQ^c , kJ/kg fabric	Breaking strength, N		Elongation at break, %	
	T, °C	t, min				Warp	Weft	Warp	Weft
Control ^a	–	–	44.26	0.5±0.1	–	391	371	37.4	30.2
Activated	70	40	54.32	11.2±0.2	4769	385	361	51.4	32.8
Conventional ^b	95	60	54.76	10.3±0.5	7418	368	300	45.9	32.7

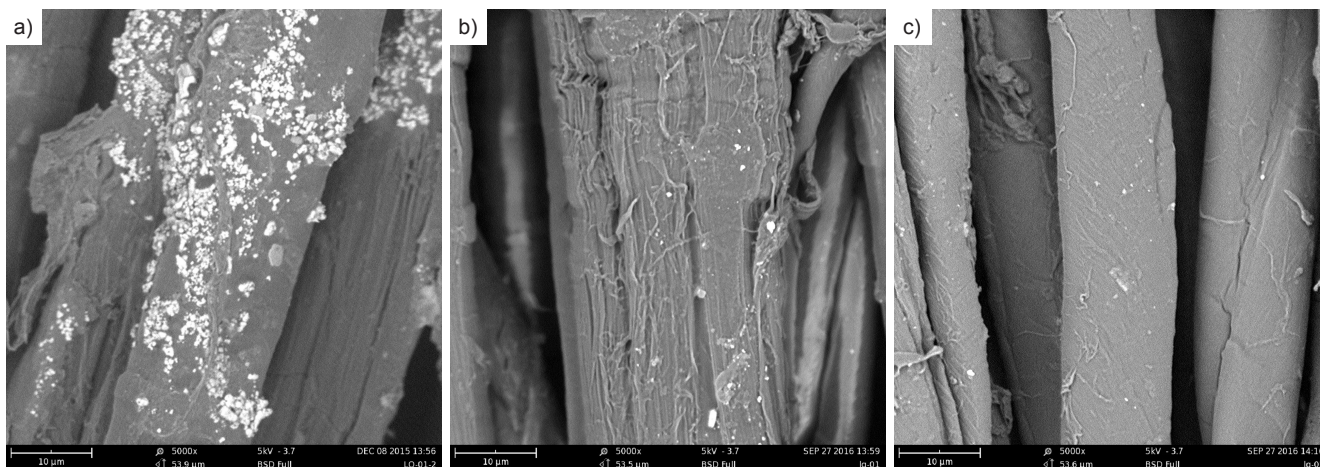


Figure 4. SEM images of greige and treated fabrics obtained by the conventional and activated processes: a) greige, b) conventional scoured and bleached fabric, c) activated scoured and bleached fabric.

Effect of [TAED] * T

The interaction between [TAED] and temperature was also significant according to ANOVA. As shown in **Figure 3.a**, TAED exhibited an evidently positive effect on WI at a low level of temperature, but tended to decrease or even lose its effect with an increasing temperature. This interaction implies that the application of TAED-activated bleaching be optimised. For example, using more TAED is effective to enhance WI when the cold pad-batch process is performed; on the other hand, it could be more useful to increase the temperature when exhaustion or the pad-steam process is desired.

Comparisons between TAED-activated and conventional processes

On the basis of CCD experimental data, an optimised activated process was performed at 70 °C for 40 min, adding 20 mmol/l of TAED. WI & CE, and the strength of the fabrics was measured for comparison with the conventional process. Theoretical energy consumption (ΔQ) during the process was also estimated. The results obtained are shown in **Table 4**.

Compared with conventional bleached fabric, the activated bleached fabric exhibited nearly the same WI, indicating a similar bleaching effect of both processes. According to the test standard (FZ/T01071-2008), a CE of woven fabric above 8 cm/30 min means a qualified water absorbency which can meet the requirement of subsequent dyeing or finishing. Thus, the TAED-activated system imparted a qualified water absorbency for the unscoured cotton/linen blends. WI

and CE data confirmed that the activated system functioned effectively under near-neutral conditions, which provided a good method for the pre-treatment of those fibres with poor heat and alkali resistance.

Through a simplified method for assessing the energy consumption for the two processes, the ΔQ value in **Table 4** revealed that the energy consumption for the conventional process was 1.56 times higher than that for the activated process due to the latter's lower holding temperature. If the heat transferred from the bath to the outside conditions is included in the estimation, the advantage of saving energy should be much more evident. Besides this, compared with the control sample, the treated fabric that had undergone the activated process had a comparable breaking strength and a longer elongation at break. The mild and low-temperature conditions protect the fabric from damage, which could explain why the fabric strength is well preserved.

Morphology of cotton/linen blended fabric

SEM was conducted to exam the micro-morphology of the fabric before and after the treatment. As shown in **Figure 4**, there were parallel ridges, occasional breaks, obvious grooves and many impurities on the raw fabric surface. By contrast, the treated fabrics showed a cleaner and flatter surface, indicating that most impurities had been removed and the appearance had undergone a great improvement. It was also found that the activated treated fabric had a cleaner and smoother surface, which verifies that the activated system is beneficial to completely re-

move the impurities and protect the fabric from damage.

Conclusions

A near-neutral TAED-activated system was designed for the combined scouring and bleaching of cotton/linen blended fabric. RSQM based on CCD was used for discussing the interactions of [TAED], T and t in the bleaching and optimising of the whole process. ANOVA for the RSQM revealed that T was the most significant factor affecting WI, followed by [TAED] and t. Moreover, there was an interaction of [TAED] and T significantly affecting WI as well as a quadratic relationship between t and WI. The TAED/H₂O₂ system could be optimised for improving the WI of the fabric by raising T, increasing [TAED] or extending t. The response surface analyses showed that the near-neutral activated system can be effectively used for the bleaching of cotton/linen blends, which have a poor wicking capability.

The optimised process can be performed at 70 °C for 40 min, incorporating 20 mmol/l of TAED and 42 mmol/l of H₂O₂. Compared with the conventional system, which included 80 mmol/l of H₂O₂ and was carried out at 95 °C for 60 min, the activated system provided comparable WI and CE for the fabric. Due to the near-neutral and low-temperature conditions, the activated system had more competitive advantages than the conventional H₂O₂ system in saving energy and limiting fabric strength damage. SEM analysis revealed that the activated bleached fabric had a smoother and cleaner surface.

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