

Short Communications

Sodium perborate bleaching of cotton by using tetraacetyl ethylenediamine activator

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In the present work, attempt has been made to bleach the scoured cotton fabric with sodium perborate as bleaching agent and tetraacetyl ethylenediamine as bleach activator. The concentrations of these chemicals, temperature and time of the treatment have been varied. When sodium perborate comes in contact with water in bleaching bath, it releases hydrogen peroxide which is responsible for bleaching action on cotton. The findings of this novel process on the quality of bleached fabric are compared with the fabric bleached conventionally using only hydrogen peroxide. The results show that the utilization of generated hydrogen peroxide reaches as high as 96% in the modified bleaching process, which is only 70% in the case of conventional bleaching. Further, the modified bleaching process requires less water and energy, thus fulfilling the requirements of ecofriendly process. Loss in weight, tearing strength and tensile strength are found less comparing to conventional bleaching without compromising with whiteness index. CIE whiteness with conventional bleaching is 57, whereas modified recipe shows 61 whiteness.

Keywords: Cotton, Bleaching, Hydrogen peroxide, Sodium perborate, Tetraacetyl ethylenediamine

Today's textile industry is very much aware of conserving energy in wet processing for eco-friendly approach as well as for increasing the profit margins. Hydrogen peroxide bleaching is beneficial only when it is either carried out at low temperature or in as minimum time as possible. In conventional peroxide bleaching, efficient decomposition of peroxide requires higher temperature since at lower temperature it becomes ineffective¹. Low temperature bleaching with hydrogen peroxide is possible only by producing per-acetate anion. However, this reaction is corrosive and also not stable. Tetraacetyl ethylenediamine (TAED) is being used as bleach activator in the laundry along with a detergent so that bleaching can be carried out at low temperature². TAED activated

bleaching system can be used for cellulosic textiles for improved quality.

Sodium perborate is a white, odourless, water-soluble powder. It undergoes hydrolysis in contact with water, producing hydrogen peroxide and borate^{3,4}. Reaction of hydrogen peroxide, water and TAED releases per acid or peroxy acid, which is a very strong oxidant⁵. The redox potential value is nearly 2.3 V, whereas aqueous mixture of peroxide is giving redox potential 1.8V.

In this study, sodium perborate has been used as an oxidizing agent with TAED as an activator. The effect of temperature, time, and concentrations of sodium perborate and TAED on whiteness of treated fabrics has been studied by using Taguchi analysis. The quality of bleached fabric is assessed by evaluating whiteness index, strength loss, carboxyl group content, and copper number of treated cotton and the results are compared with conventional bleaching process. Also, the residual hydrogen peroxide concentrations of both bleaching baths are evaluated.

Experimental

The 72 × 56 plain woven scoured 100% cotton fabric having 240 g/m² weight was used for the experiments. Both warp and weft yarns were of 20^s count. Fabric (scoured) properties are given below:

Property	Value
Tensile strength, kgf	
Warp wise	:35.16
Weft wise	:34.49
Tearing strength, gmf	
Warp wise	:1721.60
Weft wise	:1846.28
Whiteness index (CIE)	:33.16

Sodium perborate (bleaching), hydrogen peroxide (bleaching) and sodium silicate (stabiliser) obtained from Loba Chemicals; TAED (activator)– obtained from Noida Chemicals; sodium hydroxide (alkali)– obtained from Fissher Chemicals; and magnesium sulphate (water hardness) – obtained from Merck(I) Ltd, were used for the study. All the above chemicals used were of LR grade.

Methods

Low temperature bleaching was carried out on 10 g sample of scoured cotton fabric in the laboratory using

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'Mathis Labomat' (high temperature high pressure beaker machine), keeping material-to-liquor ratio 1:20. After this treatment, fabric samples were rinsed with fresh water and dried in air. Bleaching methodology by both conventional and proposed process are described below:

(i) Conventional Process

Scoured cotton fabric → Conventional bleaching →

Hot wash → Cold Wash → Cold wash

The conventional recipe comprises hydrogen peroxide (3 vol.), sodium hydroxide (1.0 % owf), stabiliser- sodium silicate (3g/L), temperature (90°C), and time (4 h).

(ii) Proposed Process

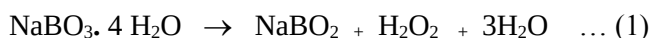
Scoured cotton fabric → Modified bleaching →

Hot wash → Cold wash → Cold wash

To find out optimum combination of parameters for best results, Taguchi analysis was carried out using Minitab 13 statistical data software. The Taguchi method can reduce research and development costs by improving the efficiency of generating information needed to design systems that are insensitive to usage conditions, manufacturing variation, and deterioration of parts⁶. The values of affecting parameters used are given in Table 1. Sixteen different recipe formulations were selected. The details of the recipes are as shown in Table 2.

Testing and Analysis

Sodium perborate undergoes hydrolysis in the presence of water, producing hydrogen peroxide and borate as shown in following reaction⁷:



The molecular weight of sodium perborate is 154. As per reaction (1), 154 parts of sodium perborate are equivalent to 34 parts of hydrogen peroxide. Residual sodium perborate content in the bleach bath was

Table 1— Parameters studied for quality assurance

Parameter	Value
Sodium perborate , g/L	10, 15, 20, 25
TAED, g/L	1, 2, 3, 4
Temperature, °C	40, 50, 60, 70,
Time, min	30, 60, 90, 120

calculated by potassium permanganate titration method using the following formula:

$$\text{H}_2\text{O}_2(\text{g/L}) = \frac{0.0017 \times A \times 1000}{V}$$

where A is a burette reading of 0.1 N KMnO₄; and V, the volume (mL) of the sample taken. Similar method was used for estimating residual hydrogen peroxide content in bleach bath.

Whiteness indices of bleached samples were measured on Konica Minolta 3600d spectrophotometer by using of CIE formula, D-65 light source and 10° observer.

Tensile strength of scoured as well as bleached fabric samples was measured both in warp and weft directions on Instron tensile tester (Model No. 5565) by using ASTM-D- 5035 (1995) method.

Tearing strength of scoured as well as bleached fabric was measured both in warp and weft direction on Instron tensile tester (Model No. 5565) by using IS - 6489 (1971) method.

Carboxylic group content of the samples was determined by IS: 1560 – 1974 (Reaffirmed 1999) method.

Copper number of the cotton fabric bleached by modified and conventional process was determined by IS: 200: 1989 method.

Results and Discussion

There are various impurities and ingredients present in cotton which need to be removed efficiently during pre-treatment for value-added processes like dyeing, printing and finishing to improve quality of the fabric.

Table 2 — Taguchi analysis chart

Recipe	Sodium perborate, g/L	TAED g/L	Temp °C	Time min
T1	10	1	40	30
T2	10	2	50	60
T3	10	3	60	90
T4	10	4	70	120
T5	15	1	50	90
T6	15	2	40	120
T7	15	3	70	30
T8	15	4	60	60
T9	20	1	60	120
T10	20	2	70	90
T11	20	3	40	60
T12	20	4	50	30
T13	25	1	70	60
T14	25	2	60	30
T15	25	3	50	120
T16	25	4	40	90

Experiments were carried out on scoured cotton fabrics by exhaust method in the laboratory using recipes as given in Table 1. The effect of parameters like temperature, time and concentrations of sodium perborate and TAED on weight loss, whiteness index, tensile strength, tearing strength, carboxylic group content, copper number and residual hydrogen peroxide is shown in Figs 1 and 2.

From the general linear model analysis from Figs 1 and 2, it is observed that weight loss increases with increasing concentration of sodium perborate and TAED. But as time progresses there is decrease in weight loss. Weight loss of modified bleaching process is found marginally low (3.51%) as compared to that of conventional bleaching process (4.63 %).

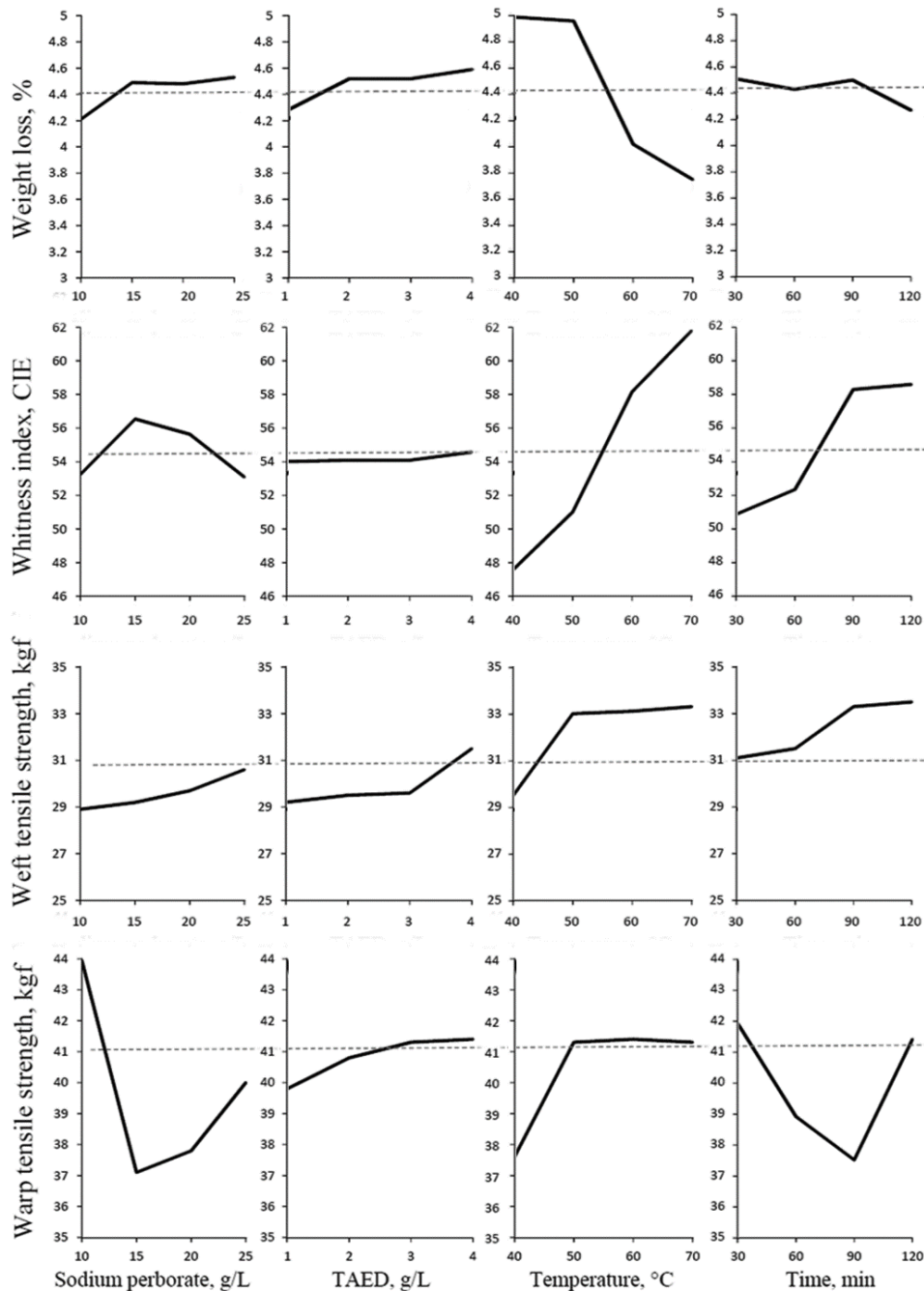


Fig. 1 — Effect of treatment condition on tensils strength, whiteness index and weight loss of fabric (Main effect plots data meant values)

The CIE whiteness index increases with increase in concentration of TAED, temperature and time. Up to 15g/L concentration of sodium perborate, the whiteness index is increased, which decreases slightly thereafter. Thus, temperature and

time has significant effect on whiteness. Samples bleached with recipes from T4 onwards show little higher whiteness (CIE index-61) as compared to conventional pre-treatment process (CIE index-60).

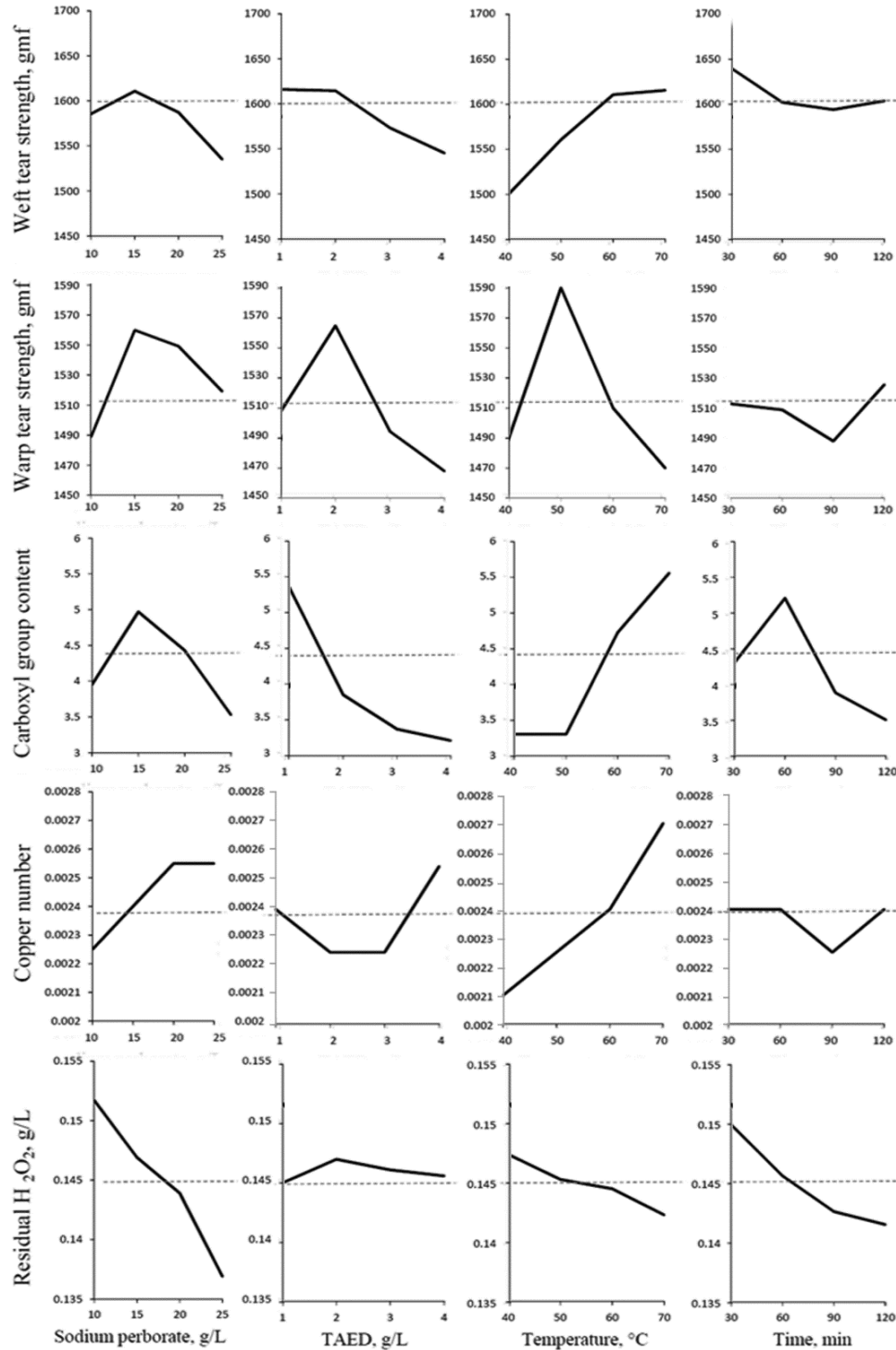


Fig. 2 — Effect of treatment condition on tear strength, carboxy group, copper number and residual peroxide of fabric (Main effect plots data mean values)

Increasing trend is observed in weft way tensile strength for all the parameters. Weft tensile strength loss of modified bleaching sample is found lower by about 10% than that by conventional bleaching process.

Warp tensile strength initially decreases up to 90 min. time of treatment and 15 g/L concentration of sodium perborate, after which it increases with increase in time and concentration of sodium perborate. TAED and temperature has no significant effect. Warp tensile strength loss of modified process is observed to be lower by 8.67% than that of conventional pre-treatment.

For proposed method the maximum tearing strength loss observed warp way is found 14% and weft way 18%. For the conventionally treated sample, the tearing strength loss is 15% (1463.38 gmf) warp way and 20% (1477.01 gmf) weft way. Concentrations of sodium perborate and TAED and the temperature has significant effect on warp way tearing strength. At higher levels of these parameters, the loss observed is higher. On weft tearing strength, TAED concentration and temperature show significant effect. As the concentration of TAED is increased, the weft tearing strength decreases. Both warp and weft tearing strength loss in new method is observed to be lower than that for conventional bleaching method.

The carboxylic group content significantly decreases with increase in concentration of TAED but increases with increase in temperature. At 15g/L concentration of sodium perborate and 60 min treatment time, the carboxylic group content is found maximum, which decreases thereafter. Carboxylic group content of modified process is marginally lower than that of conventional bleaching process, which is estimated to be 4.3 in modified process.

The copper number increases with increase in concentration of sodium perborate and temperature of the process. There is no significant effect of concentration of TAED and time. Copper number of modified process bleached samples is found lower as compared to conventional bleaching process. Copper number of conventionally treated sample was 0.003.

The residual content of sodium perborate, as decomposed into peroxide in the bleach bath decreases with increase in its initial bath concentration, treatment temperature and the time. With TAED concentration as a parameter, the residual hydrogen peroxide increases up to 2 g/L TAED concentration. A further increase in concentration of TAED decreases the residual hydrogen peroxide. Residual hydrogen

peroxide content is found 5-7%, which is lower as compared to conventional bleaching process (18%).

Combination T4 is observed to give highest whiteness value of 61 CIE. When sodium perborate is substituted by hydrogen peroxide in this combination, the whiteness is found 57 CIE. This may be due to the rapid decomposition of peroxide in bleach bath where stabilizer has not been used.

The results on main effect plots generated by MINITAB-13 software, as shown in Fig.1 and Fig.2, clearly indicate that among the various components of all the recipes experimented; the effect of sodium perborate concentration on various results is insignificant. From Taguchi analysis, it is observed that the change in temperature causes maximum variation in all results whereas the change in process time causes the least.

Taguchi analysis has given ranking to all affecting parameters of the process as highest (1) to lowest (4) as follows: Temperature (1) > TAED (2) > sodium perborate (3) > Time (4)

The major requirement of any textile process house is the maximum whiteness of fabric with lowest degradation. This is also important from the point of view of perfect shade matching of the coloured goods. Out of 16 recipes experimented, recipe No. T4 (Table 2) is found to be the most suitable one for the whiteness requirement.

As shown by Taguchi analysis mean graph residual content of sodium perborate, decreases with its initial concentration of sodium perborate in bath and treatment time. It is marginally lower as compared to peroxide bleaching. Hence, it is adapted for taking bulk scale industrial trial employing jigger for exhaust method of bleaching.

Greig mercerized and scoured fabric (150 kg) of same construction as that used in the laboratory trials is subjected to the new proposed bleaching process using the optimum values of parameters and the fabric after bleaching is evaluated for various parameters (Table 3). It may be clearly observed from the data that the results on all the evaluation parameters of bleaching by laboratory experiments and the bulk trial experiments nearly match with those of the theoretical predictions and the variations in every parameter are marginal. Thus, the modified bleaching process with sodium perborate using TAED as activator has proved to be successful.

The modified bleaching process using TAED as an activator along with sodium perborate as an oxidizing

Table 3 — Comparison of bulktrial results with the laboratory results

Property	Theoretical predictions (A)	Laboratory results (B)	Difference (A-B) %	Bulk trial (C)	Difference (B-C) %
Weight loss, %	3.71	3.61	5.39	3.64	3.70
Whiteness index, CIE	63.54	61	3.99	62.97	3.22
Carboxyl content, mg/g	3.16	3.46	9.49	3.32	4.04
Residual sodium perborate, g/L	0.16	0.14	11.42	0.17	17.65
Copper number	0.002	0.002	0	0.002	0
Tensile strength loss (warp), %	8.50	8.34	1.88	8.79	5.39
Tensile strength loss (weft), %	7.54	7.49	0.66	7.62	1.73
Tearing strength loss (warp), %	13.49	13.62	0.96	13.01	4.48
Tearing strength loss (weft), %	16.94	17.16	1.29	17.89	4.25

agent has proved to be efficient in giving results at par with the conventional peroxide bleaching process. Modified process can conserve 55% energy and time as well as 30% saving of water since maximum whiteness is observed at 70°C and 2 h treatment time as compared to conventional peroxide bleaching carried out at 90°C for 4 h. Also, the effluent load is reduced due to elimination of sodium silicate stabilizer and reduced number of washings that are required in conventional combined bleaching process. Thus, it can be concluded that, in the modified process, the utilization of active species (H_2O_2) that is responsible for bleaching action is better as compared to that in conventional peroxide bleaching.

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