Combined scouring-bleaching of cotton using potassium persulphate

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An attempt has been made to use hydrogen peroxide in a combined scouring and bleaching process on mill grey mercerized cotton fabric at a lower temperature in the presence of a bleach activator (potassium persulphate) and an ecofriendly commercial stabilizer. The results on assessment of various quality parameters such as whiteness and tensile strength of the bleached cotton fabric indicate no adverse effect of new process as compared to conventional combined scouring and bleaching process carried out at higher temperature. The new process is found to be successful in terms of savings in energy and time.

Keywords: Bleaching, Cotton, Energy conservation, Minitab 13, Potassium persulphate, Scouring

1 Introduction

Bleaching is the removal of unwanted colour from the textile fibres and typically involves the use of one of the four main bleaching agents, namely sodium hypochlorite, calcium hypochlorite, sodium chlorite and hydrogen peroxide. The bleaching process includes three main steps, namely (i) saturating the fabric with the bleaching agent and other necessary chemicals; (ii) raising the temperature to the recommended level for the particular textile and maintaining that temperature for necessary duration; and (iii) thoroughly washing and drying the fabric.

Conventionally, bleaching with bleaching powder or sodium hypochlorite was carried out at room temperature. However, hypochlorite bleaching units of textile bleaching processes generate more AOX (17.2–18.3 mg L\(^{-1}\)) and hence they belong to the category of banned chemicals, being non-ecofriendly in nature. From quality control point of view, the drawback is that the whiteness produced by these bleaching agents is not permanent. They also consume more water (45–80 L/kg of yarn/cloth). On the other hand, bleaching with hydrogen peroxide hardly generates the AOX in the effluents and the water consumption is also comparatively less (40 L/kg of yarn/cloth).

Prior to bleaching, most of the impurities present in the cotton material are removed by pressure boil under alkaline conditions. Hydrogen peroxide bleaching is also performed under alkaline conditions and, as a result, may be combined with the scouring process. Hydrogen peroxide bleaching is ecofriendly and it is carried out at pH 10.5 - 11 and temperature 80 – 85°C for 4 h conventionally in the presence of sodium silicate as a stabiliser. However, sodium silicate is not ecofriendly and also imparts harshness to cotton material. Bleaching with hydrogen peroxide requires a large amount of steam (2.5 kg/kg of fabric), thereby adding to the fuel cost. Now-a-day’s fuel is becoming expensive due to fast depletion in fuel reservoir. Thus, although it is environment friendly, the rising fuel cost tends to make hydrogen peroxide bleaching an uneconomical process.

Conservation of energy is the only step essential to overcome the mounting problems of the worldwide energy crisis and environmental degradation. The approach, therefore, should be based on the process intensification, making most of the energy used in a process and, if not possible, process modification so as to reduce the energy requirements. One such approach is the combined scouring and bleaching of cotton, which has the potential of saving the energy as well as time required for making cotton ready for further value additions such as dyeing, printing, finishing, etc.

The other possibility is to keep the temperature low for bleaching. This may appear possible since the scouring process removes almost all impurities from cotton, leaving behind only the colouring matter to be removed through oxidative bleaching.

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In this study, an attempt has been made to explore the possibility of bleaching cotton fabric with hydrogen peroxide at lower temperature without compromising the bleaching quality in terms of whiteness and strength. Mill grey mercerized cotton fabric was subjected to combined scouring and bleaching process using 25 different recipe formulations (R1-R25) of alkaline hydrogen peroxide bath containing simultaneously potassium per sulphate (PPS) as an activator and Contavan GAL of CHT (I) Ltd. as a commercial stabiliser based on hydroxyl carbonic acid and claimed to be ecofriendly. Various tests, such as CIE whiteness index, strength loss, weight loss, carboxyl group content and copper number, have been carried out for assessing the quality of bleached fabric treated with various formulations. In present industrial scenario, the quality control in processing is becoming more important besides the cost control. Therefore, in addition to energy saving benefits, there is emphasis on prediction of quality parameters like whiteness index, strength loss, weight loss, carboxyl group content and copper number. Such a prediction will be very much beneficial for obtaining required quality processing in bleaching.

2 Materials and Methods
2.1 Materials
The 60 × 59 plain woven mill grey mercerised 100% cotton cambric fabric (164 g/m²) was used for the study. Both warp and weft yarns were of 20s count. The tensile strength (warp-wise) of grey and mercerized fabric was 59.72 kgf and 71.26 kgf respectively. Whiteness index (CIE) of grey and mercerised fabric was -2 and 33.50 respectively.

The chemicals namely sodium hydroxide, hydrogen peroxide (100 vol), potassium sulphate and magnesium sulphate of LR grade, manufactured by Merck (I) Ltd., were used for securing functions of scouring, bleaching, activator and hardness respectively. The commercial sample ‘Contavan GAL’ of CHT (I) Ltd. make was used as stabiliser.

2.2 Methods
Low temperature combined scouring and bleaching was carried out on mill grey mercerised cambric fabric in the laboratory using ‘Mathis Labomat’, keeping material-to-liquor ratio at 1:30. After the treatment, fabric samples were rinsed with fresh water and dried in air.

The process sequences for conventional and modified methods are given below:

Conventional Process
Mill grey mercerised fabric → Conventional scouring & bleaching → Hot wash → Cold wash → Cold wash

The conventional recipe comprises hydrogen peroxide (3 vol), sodium hydroxide (1.5 % owf), sodium carbonate (0.5 %), stabiliser sodium silicate (3 g/L), soap (1.0 g/L), temperature (90 °C), and time (3 h)

Modified Process
Mill grey mercerised fabric → Modified scouring & bleaching → Hot wash → Cold wash → Cold wash

To find out optimum combination for best results, Taguchi analysis was carried out using Minitab 13 statistical data software. Twenty-five different recipe formulations (R1-R25) were selected. The details of the recipes are shown in Table 1.

To study the rate of decomposition of hydrogen peroxide, same twenty-five number of batches were carried without using fabric (blank). Following parameters were studied for quality assurance:

Parameter | Values
--- | ---
Hydrogen peroxide, vol. | 1, 2, 3, 4, 5
Sodium hydroxide, % owf | 1.5, 2.5, 3.5, 4, 5
Activator (PPS), % owf | 0.2, 0.3, 0.4, 0.5, 0.6
Stabiliser (Contavan GAL), g/L | 0.3, 0.4, 0.5, 0.6, 0.7
Temperature, °C | 30, 40, 50, 60, 70
Time, h | 1, 1.5, 2, 3, 4

2.3 Testing and Analysis
Residual hydrogen peroxide content of the bleaching bath was calculated by potassium permanganate titration method using the following relationship:

\[ \text{H}_2\text{O}_2, \text{g/L} = \frac{0.0017 \times A \times 1000}{V} \]

where \( A \) is the burette reading of 0.1 N KMnO₄; and \( V \), the volume (mL) of sample.

Copper number of the cotton fabric bleached by conventional and modified techniques was determined as per the IS: 200: 1989 method.
Carboxylic group content of the cotton fabric bleached by conventional and modified techniques was determined using IS : 1560 –1974 (Reaffirmed 1999).

Whiteness index of bleached samples was measured on Konica Minolta 3600d spectrophotometer employing CIE formula, keeping D-65 light source and 10° observer.

Tensile strength of grey as well bleached fabric was measured warp-wise by following ASTM-D-5035 (1995) method using Instron tensile tester (Model No. 5565).

Wettability of bleached fabric was measured by IS 2349:1963 method.

3 Results and Discussion

Preparation of cotton fabric for further value addition processes like coloration and finishing plays a very important role in maintenance and enhancement of the quality. Proper whiteness and absorbency need to be achieved without any significant strength loss or oxidation. In order to conserve energy and reduce the time of operation, combined scouring and bleaching is worth considering. However, in most of the approaches dealt so far the results are nowhere near the conventional process, particularly in terms of whiteness and absorbency, which involves sequential scouring and bleaching.

Potassium per sulphate (PPS) is known to be a catalyst finding major use in polymerization through generation of free radicals which initiate and propagate the polymer chain. It has also been shown to activate the decomposition of hydrogen peroxide. Controlled peroxide decomposition must be warranted
to effectively bleach the cotton fabric and hence use of a stabilizer becomes necessary. Sarma et al.\textsuperscript{2} reported the use of PPS in combined scouring and bleaching process via padding of cotton fabric. The authors have indicated that PPS can be used for getting satisfactory bleaching employing statistical analysis. No other work on the use of PPS in bleaching has been reported so far.

In the present work, a mathematical software (Minitab 13) has been used to predict the results of combined scouring and bleaching of cotton by exhaust method using PPS as activator and Contavan GAL (abbreviated as GAL) as a commercial stabilizer simultaneously in the bath. Taguchi analysis technique was used to generate 25 recipes with variations in the values of components such as NaOH, H\textsubscript{2}O\textsubscript{2}, temperature, time, activator PPS and stabilizer GAL. Experiments were conducted in the laboratory using these recipes and the results were obtained in terms of weight loss, strength loss, whiteness index, copper number, carboxylic group content, residual peroxide and absorbency.

The results generated by MINITAB-13 software are shown in Fig 1. It is clear that among the various components of all the recipes used the effect of change in hydrogen peroxide concentration on various results is insignificant. From Taguchi analysis, it is observed that the change in temperature causes maximum variations in all properties, whereas the change in concentration of hydrogen peroxide causes the least.

Taguchi analysis has given ranking to all components as highest affecting to lowest affecting component, which is as follows:

Temperature > Stabilizer GAL > Activator PPS > Time > Alkali NaOH > Hydrogen peroxide

From the Taguchi analysis of the means it is inferred that as the component hydrogen peroxide shows the lowest effect on the mean results, it may be discarded from the general linear model analysis.

The highest effect of temperature variations on the properties of bleached fabric, as compared to others, may be attributed to the fact that the decomposition of PPS to generate free radicals, which, in turn, gives rise to formation of peroxy radicals is governed mainly by the temperature. The chemical reactions involved are given below:

\[
\begin{align*}
K_2\text{S}_2\text{O}_8 + \text{H}_2\text{O} &\rightarrow 2\text{KHSO}_4 + \frac{1}{2}\text{O}_2 \\
\text{H}_2\text{O}_2 + \text{OH}^- &\rightarrow \text{HO}_2^- + \text{HO}_2 \\
\text{HO}_2^- + \text{H}_2\text{O}_2 &\rightarrow \text{HO}_2^- + \text{HO}^- + \text{HO}^- \\
\text{HO}_2^- &\rightarrow \text{O}_2^- + \text{H}^+
\end{align*}
\]

The major requirement of any textile process house is the whiteness of fabric. This is also important from the point of view of perfect shade matching of the coloured goods. Out of 25 recipes experimented, recipe No. R 23 (Table 1) is found to be the most

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Theoretical predictions</th>
<th>Laboratory results</th>
<th>Difference in theoretical predictions and lab results, %</th>
<th>Shop floor trial No.1</th>
<th>Shop floor trial No.2</th>
<th>Difference in lab results and bulk trial results, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight loss, %</td>
<td>3.83</td>
<td>3.72</td>
<td>2.87</td>
<td>3.58</td>
<td>3.69</td>
<td>2.42</td>
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<tr>
<td>Whiteness index, CIE</td>
<td>68.69</td>
<td>68.46</td>
<td>0.3</td>
<td>68.85</td>
<td>69.10</td>
<td>0.7</td>
</tr>
<tr>
<td>yellowness index</td>
<td>0.09</td>
<td>0.06</td>
<td>33</td>
<td>0.06</td>
<td>0.05</td>
<td>8.33</td>
</tr>
<tr>
<td>Carboxyl content, mg/g</td>
<td>0.60</td>
<td>0.623</td>
<td>3.83</td>
<td>0.63</td>
<td>0.65</td>
<td>2.73</td>
</tr>
<tr>
<td>Residual peroxide, %</td>
<td>0.28</td>
<td>0.31</td>
<td>10.71</td>
<td>0.29</td>
<td>0.28</td>
<td>8.06</td>
</tr>
<tr>
<td>Copper number</td>
<td>0.38</td>
<td>0.39</td>
<td>2.63</td>
<td>0.3</td>
<td>0.3</td>
<td>23.07</td>
</tr>
<tr>
<td>Strength loss, %</td>
<td>8.36</td>
<td>8.29</td>
<td>0.83</td>
<td>8.75</td>
<td>8.90</td>
<td>6.51</td>
</tr>
<tr>
<td>Absorbency, s</td>
<td>&lt;2</td>
<td>&lt;2</td>
<td>0</td>
<td>&lt;2</td>
<td>&lt;2</td>
<td>0</td>
</tr>
</tbody>
</table>
suitable one for the whiteness requirement of two different industrial trials employing jiggers for exhaust method of combined scouring and bleaching. Therefore, a bigger lot of grey mercerized fabric (150 kg) of same construction as that used in laboratory trials is subjected to the combined process using this recipe (R23) and testing of the bleached fabrics has been carried out for various parameters. The results are given in Table 2. It is found that the shop floor results are largely (> 90%) matching with the results of the laboratory trials.

The multiple regression equations with fitting parameters are given in Table 3. Higher \( R^2 \) value indicates better predictability of a parameter. The data clearly indicates that the % deviation between the laboratory trial values and the theoretically predicted values for various parameters is less than 4%, except for peroxide concentration (about 10%), perhaps due to its unstable nature. Similar agreement is observed with the results of laboratory trials and the shop floor trials. This confirms that the optimization of different components of recipes is perfect and can be confidently used for bulk scale operations. It may be observed from all these results that in the proposed experimental set-up, it is possible to carry out combined scouring and bleaching with a good amount of saving in time (from 3 h for conventional to 2 h) and temperature (from 90°C for conventional to 70°C) of the process.

4 Conclusions
The proposed combined scouring and bleaching method using potassium persulphate as an activator along with a commercial stabilizer has proved to be efficient in giving results at par with the conventionally followed sequential scouring and bleaching. This will lead to substantial reduction in time of pre-treatment and also reduce the energy requirements.

References