Optimization of non-silicate stabilizers for bleaching of cotton knitted goods

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The study gives a comparative view on the performance of silicate and non-silicate stabilizers and optimizes the peroxide concentration in the bleaching process. The uniformity of dyeing and the colour difference have also been studied on fabrics bleached using silicate and non-silicate stabilizers. Results indicate that the optimum amount of peroxide needed for bleaching is 3% owf, the inference being made from the whiteness index and yarn tenacity data. The bending length of the samples bleached using non-silicate stabilizers is smaller compared to that for the samples bleached by using sodium silicate, indicating a softer handle. The values of relative unlevelness index show that the uniformity of dyeing is excellent in all the cases i.e. the use of non-silicate stabilizer during bleaching has no adverse effect on dyeing.

Keywords: Bleaching, Cotton knitted fabric, Non-silicate stabilizer, Yarn tenacity, Whiteness index

1 Introduction
Preparatory process has great significance in textile wet processing. In cotton knitted goods, the preparatory processes involve scouring, bleaching and washing. There has been a continuous development in the area of bleaching. Sodium hypochlorite bleaching is definitely cheaper, but it gives rise to high AOX (adsorbable organic halogen compounds) value1,2. Further, it leads to yellowing of the fabric on storage3. Sodium chlorite bleaching, developed subsequently, has problems due to the corrosive nature of ClO₂ evolved during the process. A major disadvantage is that it cannot be combined with other processes. Though it has AOX value lower than hypochlorite, it is not completely free from this problem.

Considering the ecological requirements, hydrogen peroxide has emerged as the most viable bleaching chemical. It is free from AOX problems and can be combined with the scouring operation. However, sodium silicate, employed as a stabilizer in peroxide bleaching, tends to precipitate out of solution in hard water or upon acidification4. Hence, thrust has been on substituting silicate with non-silicate stabilizers. In fact, improvement in the properties of jute fibres has been reported with the use of non-silicate stabilizer during bleaching5.

In the present study, an attempt has been made to study silicate and non-silicate stabilized peroxide bleaching. Both single-stage and two-stage bleaching processes have been studied aiming at the optimization of the peroxide concentration in the bleaching bath. The uniformity of dyeing and the colour difference have also been studied on fabrics using silicate and non-silicate stabilizers.

2 Materials and Methods
2.1 Materials
2.1.1 Fabric
Cotton knitted (single jersey) fabric having the following specifications was used: courses/inch, 44; wales/inch, 38; weight, 128 g/m²; and count, 26s Ne.

2.1.2 Water
The water used during scouring and bleaching operations had the following qualities: pH, 7.26; hardness, 296 ppm; TDS, 589 ppm; and total alkalinity, 46 ppm. Alkalinity and hardness were
measured in terms of calcium carbonate. During dyeing, 0.857 g/l EDTA was added to water to make it soft.

2.1.3 Non-silicate Stabilizer

Non-silicate stabilizer having the following characteristics was used:

<table>
<thead>
<tr>
<th>Physical form</th>
<th>Off-white paste/liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical nature</td>
<td>Anionic</td>
</tr>
<tr>
<td>Solubility</td>
<td>Soluble in water</td>
</tr>
<tr>
<td>Compatibility</td>
<td>Stable to hard water and alkali; compatible with anionic and non-ionic auxiliaries.</td>
</tr>
</tbody>
</table>

2.2 Methods

2.2.1 Whiteness

The Hunter whiteness has been reported as the whiteness index (WI). The Hunter whiteness is expressed as $L-3b$, where $L$ and $b$ are CIE coordinates. WI was measured on computer colour matching system (Macbeth Color-Eye 3100).

2.2.2 Absorbency

This was determined as per IS : 2349-1963.

2.2.3 Yarn Tenacity

Yarn tenacity was measured on Tensorapid instrument using 20cm yarn gauge length and 50 mm/min elongation speed.

2.2.4 Relative Unlevelness Index (RUI)

The RUI was calculated using the following formula:

$$\text{RUI} = \frac{\sum_{\lambda=400}^{700} C_\lambda \cdot V_\lambda - \sum_{\lambda=400}^{700} (S_\lambda / \bar{R}) \cdot V_\lambda}{\sum_{\lambda=400}^{700} C_\lambda \cdot V_\lambda}$$

where $C_\lambda$ is the coefficient of variance of reflectance values; $V_\lambda$, the phototopic relative luminous efficiency function; $S_\lambda$, the standard deviation of the reflectance values; and $\bar{R}$, the average of reflectance values.

2.2.5 Colour Evaluation

Colour evaluation was done on a computer colour matching system (Macbeth Color-Eye 3100). The colour difference $DE$ is measured in terms of $D_\lambda'$, $D_{a'}$ and $D_{b'}$ which are the differences in CIE coordinates.

2.2.6 Bending Length

Bending length was measured as per ASTM standard (D-1388-64).

2.2.7 Bleaching

Laboratory hank dyeing machine (open vessel) and laboratory high-temperature high-pressure dyeing machine (closed vessel) were used for recipes I and II, whereas for all other recipes (III-VI), only laboratory hank dyeing machine was used. The recipes used in the experiments were as per the guidelines of the manufacturers.

Bleaching was carried out on scoured samples (for recipes I and II) and grey samples (for recipes III - VI). The grey fabric was scoured with the following recipe:

- Caustic soda : 2-3% owf
- Lissapol D : 0.5% owf
- pH : 10-11
- Temperature : At boil
- Time : 5 h

The details of the various experimental bleaching recipes are given below. In all the experiments, the M:L ratio was kept at 1:20. Hydrogen peroxide has been referred as peroxide in these recipes.

**Recipe I**

- Sodium hydroxide : 0.6% owf
- Soda ash : 1.0% owf
- Wetting agent (Lissapol D) : 0.5% owf
- Sodium silicate : 2.0% owf
- Peroxide (50%) : 0.5-5% owf
- Temperature : 95°C
- Time : 2 h
- pH : 10.5-11.0
- Fabric : Scoured

Experiments were carried out with different concentrations (0.5-5.0% owf) and residual peroxide estimated with 0.1N sodium thiosulphate solution as listed in Table 1.

**Recipe II**

- Peroxide (50%) : 2.5 ml/l (5% owf)
- Stabilizer NS (ICI) : 1.7 g/l
- Caustic soda : 1.5 g/l
- Lissapol D : 0.5 g/l
- Temperature and time : Temp. raised from 60°C to 95°C in 30 min and then maintained at 95°C for 1 h
- pH : 10-11
- Fabric : Scoured
Table I—Results of open system (hank dyeing machine) bleaching

<table>
<thead>
<tr>
<th>Stabilizer</th>
<th>( \text{H}_2\text{O}_2 ) (50%) % owf</th>
<th>( \text{H}_2\text{O}_2 ) consumed % owf</th>
<th>WI (Hunter whiteness index)</th>
<th>Yarn tenacity gf/tex</th>
<th>Yarn strength loss, %</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Recipe I</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sodium silicate</td>
<td>0.5</td>
<td>0.46</td>
<td>68.90</td>
<td>13.08</td>
<td>3.9</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>0.98</td>
<td>73.62</td>
<td>13.29</td>
<td>2.4</td>
</tr>
<tr>
<td></td>
<td>1.5</td>
<td>1.38</td>
<td>75.22</td>
<td>12.27</td>
<td>9.5</td>
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<td></td>
<td>2.0</td>
<td>1.97</td>
<td>74.49</td>
<td>12.18</td>
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<tr>
<td></td>
<td>3.0</td>
<td>2.98</td>
<td>76.53</td>
<td>11.33</td>
<td>16.8</td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>3.96</td>
<td>77.85</td>
<td>8.73</td>
<td>35.9</td>
</tr>
<tr>
<td></td>
<td>5.0</td>
<td>4.89</td>
<td>76.62</td>
<td>9.74</td>
<td>28.4</td>
</tr>
<tr>
<td><strong>Recipe II</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stabilizer NS</td>
<td>5.0</td>
<td>4.4</td>
<td>76.29</td>
<td>11.56</td>
<td>15.1</td>
</tr>
<tr>
<td>Sodium silicate</td>
<td>5.0</td>
<td>5.0</td>
<td>76.62</td>
<td>9.74</td>
<td>28.4</td>
</tr>
<tr>
<td>Scoured sample</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The experiment was also repeated by substituting stabilizer NS by sodium silicate (5.0 % owf).

**Recipe III**

Combined scouring and bleaching with non-silicate stabilizer Superstat VP, marketed by Super Products (rapid bleaching process):

- Caustic soda: 15 g/l
- Sodium silicate: 10 g/l
- Superstat VP: 5 g/l
- Imerol XI liq: 5 g/l
- Mineral turpentine: 5 g/l
- Peroxide (35%): 30 g/l
- Fabric: Grey

Imerol XI and turpentine were emulsified together under stirring. Caustic soda and sodium silicate were dissolved in water. A dispersion of Superstat VP was made separately and all the three were mixed together. Peroxide was added in the last. Fabric was impregnated in the above liquor and then batched, well covered and kept rotating for 3 h. It was then re-padded in the same liquor and steamed at 100°-120°C for 1 h and rinsed well.

**Recipe IV (Scourex Process)**

- Scouring agent*: 2% owf
- Peroxide (50%): 1% owf
- Sodium silicate: 1.1% owf
- Disodium hydrogen phosphate and NaOH in 1:1 molar ratio to give pH 11
- Time: 6 h
- Temperature: 70°C
- Fabric: Grey

* Scouring agent composition: Pine oil, 50%; Emulsifier (Niogen EA635), 40%; and Perchlooroethylene, 10%.

**Recipe IV(A)**

Experiments were done with Scourex process by taking different concentrations of \( \text{H}_2\text{O}_2 \) (1%-6% owf) and processed at 95°C for 2 h.

**Recipe V**

Combined scouring and bleaching process used in some modern mills. The chemicals used were of Clariant India Ltd.

- Sandoclean PC liq: 1 gpl at 50°C for 15 min
- Sirrix 2UDI liq: 2 gpl
- Caustic soda: 2-3% owf
- Soda ash: 0.5% owf
- Peroxide (50%): 1-2% owf
- Stabilizer AWNI: 0.25-0.5% owf
- Temperature: 95°C
- Time: 1.5 h
- pH: 10-11
- Fabric: Grey

**Recipe VI**

Conventional combined scouring and bleaching process used in some textile mills:

- Caustic soda: 3% owf
- Sodium silicate: 8% owf
- Peroxide (50%): 6% owf
- Soda ash: 1% owf
- Temperature: 95°C
- Time: 2 h
- Fabric: Grey

**Recipe VII**

Industrial trial was taken on 100 kg of fabric using this recipe.
Sandoclean PC liq : 1 gpl at 50°C for 15 min
Sirrix 2UDI liq : 2 gpl
Caustic soda : 3% owf
Soda ash : 0.5% owf
Peroxide (50%) : 3% owf
Stabilizer AWNI : 0.5% owf
Temperature : 95°C
Time : 1.5 h
pH : 10-11
Fabric : Grey (40s Ne interlocked)
Machine used : Winch

2.2.8 Dyeing
The samples obtained after bleaching using sodium silicate and non-silicate stabilizers AWNI and NS were dyed with four different reactive colours and evaluated on a computer color matching system (Macbeth Color-Eye 3100). The reactive dyes used were: Ichofix Orange HE2R (Dyechem), Serifix Red HE 8B (Serene), Ichofix Golden Yellow HER (Dyechem) and Serifix Navy Blue HR (Serene).

3 Results and Discussion
The results of the experiments with recipe I and recipe II are given in Tables 1 and 2 respectively. It is observed that for sodium silicate stabilized bleaching, the whiteness index of the bleached fabric increases with an increase in the peroxide concentration. However, at concentrations of 3% owf and above the WI is almost constant. It is clear from Table 1 that on increasing the peroxide concentration from 3% owf to 4% owf, the WI increases by just about 1%, whereas the loss in yarn strength of the bleached fabric almost doubles. Thus, considering the whiteness and the strength loss, around 3% (owf) H$_2$O$_2$ is the optimum concentration of peroxide for bleaching the scoured knitted fabric.

Using the data given in Table 1, the best fit equations relating WI and %loss in yarn strength to H$_2$O$_2$ (% owf) used initially were found out using the SYSTAT software. From these modeled equations, the respective curves were plotted against the H$_2$O$_2$ concentration used. These curves are shown in Figs 1 and 2 along with their respective equations.

It is evident from Table 1 that with non-silicate stabilizer NS, the whiteness equivalent to that with silicate is obtained but the %loss in yarn strength is reduced to almost half.

The absorbency in all these cases was less than one second. The peroxide consumption during bleaching is almost equal to peroxide used initially. Fig. 3 shows the curve for peroxide consumed versus peroxide used initially.

<table>
<thead>
<tr>
<th>Stabilizer</th>
<th>H$_2$O$_2$ (50%) % owf</th>
<th>H$_2$O$_2$ consumed % owf</th>
<th>WI (Hunter whiteness index)</th>
<th>Yarn tenacity gf/tex</th>
<th>Yarn strength loss, %</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Recipe I</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sodium silicate</td>
<td>0.5</td>
<td>0.46</td>
<td>72.19</td>
<td>12.14</td>
<td>10.8</td>
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<td></td>
<td>1.0</td>
<td>0.84</td>
<td>74.23</td>
<td>11.95</td>
<td>12.2</td>
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<tr>
<td></td>
<td>1.5</td>
<td>1.33</td>
<td>75.58</td>
<td>11.36</td>
<td>16.5</td>
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<tr>
<td><strong>Recipe II</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stabilizer NS</td>
<td>5.0</td>
<td>4.6</td>
<td>74.60</td>
<td>11.83</td>
<td>12.7</td>
</tr>
<tr>
<td>Sodium silicate</td>
<td>5.0</td>
<td>4.6</td>
<td>76.39</td>
<td>11.15</td>
<td>18.1</td>
</tr>
</tbody>
</table>

Fig. 1—Hunter whiteness vs initial peroxide concentration for sodium silicate stabilized bleaching in open system
Regression equation: $Y = 59.158 + 26.353X - 15.704X^2 + 3.976X^3 - 0.35X^4$, where $Y$ = Hunter whiteness, and $X$ = Concentration of H$_2$O$_2$ (% owf).
For the equation, $R = 0.998$, $R^2 = 0.996$, Std. error = 0.632, and F-ratio = 166.179
Table 2 shows that the whiteness of the knitted fabric is better in closed system compared to that in open system (Table 1) at the same peroxide concentration. This is owing to better circulation of bleaching liquor and hence better penetration in the closed system. This is also supported by the higher loss of yarn strength in a closed system compared to that in open system for the same peroxide concentration.

From the results of the combined scouring and bleaching process (Table 3), it may be seen that the whiteness is almost same for all recipes with peroxide concentration at or above 3% owf. For scourex process employing sodium silicate as stabilizer, a concentration of 3% (owf) H$_2$O$_2$ is optimum above which the whiteness index is constant.

In some mills which follow the conventional bleaching, 6% (owf) H$_2$O$_2$ is used. From our study, it is indicated that in this process (recipe VI) the WI is 74.44 whereas the non-silicate stabilizer (recipe V) gives the same whiteness (WI=75.07) at 3% (owf) H$_2$O$_2$ (Table 3). Recipe III using a combination of silicate and non-silicate stabilizer gives almost the same whiteness (WI=74.65) at 2.1% (owf) H$_2$O$_2$. This shows that non-silicate stabilizer gives the same results at a lower peroxide concentration and hence can be used as a substitute without any adverse effect on the fabric quality.

![Fig. 2](image-url)  
**Fig. 2**—Loss in yarn tenacity vs initial peroxide concentration for sodium silicate stabilized bleaching in open system  
Regression equation: $Y = 6.139X - 0.779X^4 + 0.408X^3 - 0.051X^2$, where $Y =$ % loss in yarn tenacity, and $X =$ Concentration of H$_2$O$_2$ (% owf)  
For the equation, $R = 0.997$, $R^2 = 0.999$, Std. error = 2.316 and F-ratio = 120.43

![Fig. 3](image-url)  
**Fig. 3**—Peroxide consumed vs peroxide used initially for sodium silicate stabilized bleaching in open system  
Regression equation: $Y = 0.982X$, where $Y =$ Peroxide consumed (% owf), and $X =$ Peroxide used initially (% owf)  
For the equation, $R = 1.0$, $R^2 = 1.0$, Std. error = 0.042, and F-ratio = 31730.039

<table>
<thead>
<tr>
<th>Process (Recipe)</th>
<th>H$_2$O$_2$ (50%) % owf</th>
<th>Hunter whiteness index</th>
<th>Yarn tenacity gf/tex</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rapid bleaching (recipe III)</td>
<td>2.1</td>
<td>74.65</td>
<td>13.52</td>
</tr>
<tr>
<td>Scourex (recipe IVA)</td>
<td>1.0</td>
<td>72.67</td>
<td>13.68</td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>73.98</td>
<td>13.77</td>
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<td></td>
<td>3.0</td>
<td>75.10</td>
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<td></td>
<td>4.0</td>
<td>73.60</td>
<td>13.65</td>
</tr>
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<td></td>
<td>5.0</td>
<td>75.38</td>
<td>13.61</td>
</tr>
<tr>
<td></td>
<td>6.0</td>
<td>75.13</td>
<td>12.48</td>
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<td>Modern bleaching (recipe V)</td>
<td>3.0</td>
<td>75.07</td>
<td>12.72</td>
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<tr>
<td>Conventional bleaching (recipe VI)</td>
<td>6.0</td>
<td>74.44</td>
<td>14.98</td>
</tr>
<tr>
<td>Grey (standard)</td>
<td>—</td>
<td>48.80</td>
<td>8.6</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th>Stabilizer</th>
<th>% H$_2$O$_2$ used</th>
<th>Hunter whiteness index</th>
<th>Yarn tenacity gf/tex</th>
</tr>
</thead>
<tbody>
<tr>
<td>AWNI (Clariant)</td>
<td>3</td>
<td>72.26</td>
<td>13.75</td>
</tr>
<tr>
<td>Grey (standard)</td>
<td>—</td>
<td>50.57</td>
<td>9.71</td>
</tr>
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</table>
Based on the above studies, an industrial trial was taken on Winch with a non-silicate stabilizer (recipe VII) and results are given in Table 4. It is observed that the yarn tenacity is almost same to that obtained in the laboratory. This is indicative of the industrial trial being commensurate with the laboratory experiments. The whiteness index data also support the above fact.

Colour evaluation data of samples dyed after bleaching with different peroxide stabilizers are given in Table 5. The sample bleached using sodium silicate as stabilizer was taken as the standard. The data show that the samples bleached using non-silicate stabilizers AWNI and NS have invariably lighter shade with all the dyes. However, the degree of lightness varies for different dyes. The $Da^*$ and $Db^*$ values are less than 1, except in a few cases. The total colour difference $DE$ in all these cases is greater than 1, indicating a considerable difference in colour of these samples compared to the standard. This difference is mostly due to the depth of the shade as shown by the data. It is only in case of Orange HE2R and Golden Yellow HER that the tonal variations also add significantly to $DE$ value.

Table 6 shows the $K/S$ values for the dyed fabrics. It is seen that the $K/S$ values for the samples bleached in a non-silicate stabilized system are lower compared to those for the samples bleached in sodium silicate stabilized system. This indicates that the former are lighter in shade compared to the latter.

Table 7 shows the relative unlevelness index (RUI) values for the dyed samples. It is seen that the levelness is excellent for the samples bleached by employing either sodium silicate as stabilizer or non-silicate stabilizers. Thus, it can be said that the dyeing uniformity is excellent in all the cases.

The bending length data of samples bleached using silicate and non-silicate stabilizers (Table 8) show that the bending length is smaller for the samples bleached by employing non-silicate stabilizer compared to the one bleached with silicate stabilizer. The same trend is observed in the case of dyed fabric. Thus, it can be said that the system employing non-silicate stabilizers give a softer handle to the fabric, which can be assigned to the elimination of precipitate deposition as in the case of silicates.

4 Conclusions

The optimum peroxide concentration is 3% owf for two-stage process as well for single-stage process like scourex or modern bleaching process. A comparison of these processes shows that non-
silicate stabilizers can be used in place of silicate without any adverse effect on the uniformity and levelness of dyeing. The bending length data show that the stiffness of the samples bleached in non-silicate stabilized system is lower compared to that in silicate stabilized system, thereby indicating that the problem arising due to silicate deposition is avoided. Hence, non-silicate stabilizers can effectively substitute silicate and maintain the fabric quality by avoiding the problems of precipitation as faced by silicates. The results of industrial trial support the findings of the laboratory experiments.

Acknowledgement

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References