A NEW PROCESS FOR THE DESIZING, SCOURING AND LEACHING OF COTTON/ NYLON FABRICS WITH TETRAACETYLETHYLENEDIAMINE

K. S. HUANG, E. P. JAN and L. A. LU

Department of Polymer Materials, Kun Shan University of Technology, Yung Kang, Tainan, Republic of China (Taiwan)

Received February 9, 2005

The study explores the effect of tetraacetylethylenediamine (TAED) and NaBO₃ on cotton/nylon (C/N) fabrics treated by one-bath/two-steps method. The results showed that, the strength of some fabrics parts excepted, all other qualities were better than those of the fabrics treated by traditional methods. However, the strength of the treated fabrics deteriorated as the concentration of H₂O₂, TAED and NaBO₃ increased, although absorbability and whiteness ran counter to this pattern. Shrinkage also increased, but not greatly. As seen from FTIR and SEM analyses, the method developed in this study is capable of producing favorable pretreatment effects. By using a pretreatment solution composed, each of 0.2 g/L NaOH, 2 g/L H₂O₂ and 2 g/L of TAED and NaBO₃, in one-bath/two-steps process at < 70 °C for 30 min, the fabrics evidenced better results.

Keywords: fabrics, strength, scouring, shrinkage

INTRODUCTION

Under the leadership of Dupont, nylon fabrics are expected to become the main material in the fashion industry of the 21st century, while the most popular fabrics, nylon/cotton (N/C), will also continue to dominate the market. The popularity of N/C or C/N fabrics illustrates the importance of nylon fibers in today’s textile industry. The unique feel of Nylon 66 recommends it on the market, making it the best choice for N/C or fabrics.¹ However, since the cotton/nylon fabrics contain two different types of fibers of varying qualities, numerous problems may arise, such as cross-contamination, increased handling time and poor appearance. Such problems often occur during dyeing, while other problems, such as damage to fabric strength or uneven surface shrinkage, may appear in the pretreatment stage. These difficulties result in increased dyeing costs or defect rates. On the current competitive market, where speed, low cost and high quality are prerequisites for survival, these problems demonstrate that current technologies are in urgent need of upgrade.

Therefore, the development of low-cost and high-quality pretreatment technologies becomes a “must” of our days.

Current C/N pretreatment technologies can be roughly classified into two types: (1) room temperature long-term treatment and (2) high temperature short-term treatment. The former class has the advantage of causing less damage to the strength of the fabrics and less shrinkage, although processing often takes 24 h. It is clearly not suitable in a production system requiring speedy delivery. High temperature short-term treatment involves fabrics subjected to a temperature of 90–100 °C. It is therefore a time-effective and speedy process. However, since the fabrics are treated at high pH and temperature values, their strength is likely to be reduced to a greater than expected degree, while total shrinkage is also higher.

Considering these, the study aims at
K. S. Huang et al.

developing an energy-conserving and highly efficient pretreatment technology, inducing less damage to fabrics strength, combined with reduced surface shrinkage. This technology, developed through repeated laboratory experiments, now expects its industrial application.

Tetraacetylethylenediamine (TAED), an oxidative activator used in the cleaning industry for many years, has the advantage of being a non-toxic, non-allergic, non-multiple, and also biodegradable by-product. More than that, ethylenediamine does not damage the fabric during the decomposition process.

As we all now live in a society in which “green textile products” dominate the market, high-price brand names observe the environmental concept of “product continuity”, prominent in European and US markets. Therefore, the use of biodegradable chemicals in dyeing and treatment processes is becoming a major trend. Thus, TAED is deservedly receiving a lot of attention from part of the fabric industry.

EXPERIMENTAL

Materials

Sodium perborate tetrahydrate, sodium hydroxide, sodium silicate, hydrogen peroxide (all of reagent grade; Wako Pure Chemical Industry, Japan), tetraacetylethylenediamine (TAED, reagent grade; ACROS), nonionic surfactant (PO-50 A, NICCA, Taiwan), untreated C/N fabrics with the following specifications: cotton: 32/L, fill/nylon: 140/48, warp/fill 84×76 (provided by Chuan-Jing Enterprise, Taiwan) were used in the study.

Methods

Conventional method

A 25×20 cm fabric sample, placed in a pretreatment solution composed of NaOH (adjusted pH ~10.5), 1 g/L Na2SiO3, 5 g/L H2O2 and 1 g/L nonionic surfactant, was then treated in a 1:30 (v/v) ratio at 90 ºC for 30 min. After washing and drying, the fabric was placed into a plastic bag for testing. The pH values of the sample were tested both before and after the treatment.

Method of study

The sample was pretreated according to the following curve, the pH values of each solution cotton/nylon fabrics being tested both before and after the treatment.

The processing procedure involves the following steps:

1) Mix 0.05 g/L of NaOH, a suitable amount of H2O2 (0–6 g/L) and 1 g/L of nonionic surfactant, in a 1:30 (v/v) ratio into the first stage pretreatment solutions. Test pH values prior to the treatment.

2) Place 20×25 cm samples in steel bottles containing the pre-prepared solutions. Increase the temperature by 1.5 ºC/min. When temperature reaches 70 ºC, maintain the solution at this temperature for 15 min. Add NaBO3 (0–4 g/L) and TAED (0–4 g/L) and keep it constant for other 15 min.

3) Lower the temperature to 40 ºC and then remove the fabric. Test the pH values of the residual solution in each of the steel bottles.

4) Dry the fabrics after washing and place them into sealed bags for testing.

Measurements

Compare the color variations of the treated samples to blank fabrics, to contrast the whiteness of the test samples. Use a Hunterlab K/S 45/O-L computer full-color spectrometer to derive the chromatic aberration values (K/S) of the fabric, based on the Kubelka–Munck formula. A larger K/S value indicates a lower level of whiteness: K/S = (1–R)2 /2R, where R is the reflectance, K is the absorption rate and S is the scattering coefficient.

Fabric absorbability (absorbance ability) can be also utilized to determine the effect of the pretreatment, i.e. the level of hydrophobes removed from the fabric. Higher absorbability or a shorter length of time for the disappearance of the water drops indicates better pretreatment conditions. The water drops are released from a certain height onto the surface of the fabric and the time taken for their disappearance is measured, the data thus obtained being then used to indicate the absorbability of the fabrics.
For shrinkage and strength measurements, an Alphaten 400 and an accelerated machine are used, as based on the AATCC TM 187-2001 and CNS 2111 testing methods. Changes in the functional groups of fibers and surfaces of the yarn are analyzed on a Bio-Rad Digilab FTS-40 Fourier spectrum analyzer and a Jeol 5610 scanning electron microscope.

RESULTS AND DISCUSSION

Effect of the pretreatment time on the pH value of the solutions

As shown in Figure 1 (indicating the first 20 min), when pretreatment solutions come into contact and react with fabric impurities in the first 5 minutes, the pH values of the solutions are lowered to 11 from the initial value of 12, as a result of the saponification occurring between the alkaline substances from the pretreatment solutions and fabric impurities.

Figure 1: Changes in pH values under various pretreatment times (NaOH: 0.05g/L, Na2SiO3: 0.2g/L, H2O2: 2g/L; nonionic surface agent: 0.2g/L, TAED: 1g/L; NaBO3: 1g/L, 70° × 30'; -♦- without fabrics; -■- fabrics in the pretreatment solution)

This causes a reduction in the alkaline solution and, consequently, the pH values of the pretreatment solutions are slightly lowered. In the second stage (after 20 min), when extra-chemicals (NaBO3 and TAED) are added, the pH values drop significantly.

This is due to the fact that NaBO3 reacts with water, producing H2O2, which again reacts with TAED and produces peracetic acid – as shown in formulae (1) and (2). Changes in the pH values of the treatment solutions without samples are also illustrated in Figure 1.

In the 20 min prior to processing, the pH values remain at 12 until the second stage. In the second stage (after 20 min), when extra chemicals (TAED and NaBO3) have been added to the solutions, the pH values follow a descending trend till the pH reaches a value of 9, the reasons being the same as for the solutions with C/N fabrics. Nonetheless, the final pH values of the two experiments are still slightly different. The pH value of the solutions with fabric is ~7, and of 8 without fabric.

Two conclusions can be drawn from this: the former is that the addition of TAED/NaBO3 lowers the pH values of the solutions, while the latter is that the presence of the fabric samples has a certain effect on the pH values of the pretreatment solutions.

4NaBO3 + 5 H2O → Na2B4O7 + 2NaOH + 4 H2O2
H2O2 → HOO− + H+ HOO−

(H3COC)2N-CH2-CH2-N(COCH3)2 → TAED
H3C-COO-O− + H3CONH(CH2)2NHCOCCH3
Peracetic acid

Effect of H2O2 and TAED concentrations on fabrics properties

Whiteness

Figure 2 plots the effect of TAED and H2O2 concentrations on the whiteness of the fabrics at fixed NaBO3 concentrations (1 g/L). As shown in Figure 2, higher H2O2 concentrations result in better whiteness. In addition, when the concentrations of H2O2, added during the first stage, are identical, higher TAED concentrations also result in superior whiteness, as the NaBO3 added during the second stage reacts with water, producing H2O2. The TAED again reacts with H2O2 and produces peracetic acid. However, as the concentrations of TAED and NaBO3 increase, more peracetic acid and more H2O2 are also produced. Since both the peracetic acid and H2O2 are oxidants, the colors in the fabrics are oxidized and removed from the fibers. Therefore, when the
concentration of oxidants increases, the bleaching effect is improved.

Figure 2: Effect of H₂O₂ concentration on the whiteness of the treated fabrics (NaBO₃: 1 g/L, 70°×30'; raw fabric: 73.84; conventional fabrics, whiteness = 75.50)

Figure 3: Effect of H₂O₂ concentration on the strength of the treated fabrics (NaBO₃: 1 g/L, 70°×30'; raw fabrics: 44.84)

Strength

Figure 3 indicates that fabrics strength reveals a descending tendency as the concentrations of TAED and H₂O₂ increase in the first stage. The peroxides react not only with the colors but also with the fibers. When the fibers are oxidized, strength is consequently lowered.

Oxidation of cellulose is a heterogeneous reaction, i.e. the oxidants do not necessarily have to react with the fibers as a whole. Usually, this type of oxidation is related to the “reachability” of the fibers, i.e. oxidation occurs rapidly in the non-crystallized areas of the fibers and slowly in the more orderly crystallized areas. However, lowering of fiber strength during pretreatment is not caused exclusively by oxidation.

The original first- and second-class methyl groups can be oxidized as well, which causes increase of the carbonyl groups, thus rendering cellulose more sensitive to alkalinity. The cellulose then becomes more prone to hydrolysis which, in turn, causes damage to fibers strength.¹⁰

Therefore, when the NaBO₃ added during the second stage reacts with water, producing H₂O₂ and alkaline substances, TAED again reacts with H₂O₂ and produces peracetic acid.

As already mentioned, chemicals such as H₂O₂ and peracetic acid are all oxidants reacting with the fibers in oxidative reactions. The presence of alkaline substances may also cause hydrolysis in fibers; therefore, when the concentration of NaBO₃ and TAED increases, the concentrations of peracetic acid, H₂O₂ and alkaline substances increase as well and, therefore, the strength of fibers is easily reduced (Tables 1 and 2).

Table 1
Effect of TAED or H₂O₂ concentration¹¹ on the physical properties of the treated fabrics

<table>
<thead>
<tr>
<th>Properties</th>
<th>TAED (g/L)</th>
<th>H₂O₂ (g/L)</th>
<th>Whiteness Index</th>
<th>Tensile strength retention (%)</th>
<th>Water drop disappearance time (sec)</th>
<th>Shrinkage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
<td>76.05</td>
<td>95.05</td>
<td>108.1</td>
<td>2.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>76.20</td>
<td>93.15</td>
<td>90.2</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>76.24</td>
<td>92.01</td>
<td>58.7</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>76.35</td>
<td>91.61</td>
<td>46.3</td>
<td>4.5</td>
<td></td>
</tr>
</tbody>
</table>

Table 1
### Table 2

Effect of TAED or H$_2$O$_2$ concentration$^1$ on the physical properties of the treated fabrics

<table>
<thead>
<tr>
<th>TAED (g/L)</th>
<th>H$_2$O$_2$ (g/L)</th>
<th>Whiteness Index</th>
<th>Strength (kg)</th>
<th>Water drop disappearance time (sec)</th>
<th>Shrinkage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>76.52</td>
<td>94.60</td>
<td>101.1</td>
<td>3.5</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>76.64</td>
<td>92.89</td>
<td>92.4</td>
<td>4.5</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>76.80</td>
<td>92.02</td>
<td>61.7</td>
<td>5.0</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>76.98</td>
<td>91.48</td>
<td>43.3</td>
<td>6.0</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
<td>76.62</td>
<td>92.84</td>
<td>73.6</td>
<td>4.0</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>76.69</td>
<td>91.44</td>
<td>60.2</td>
<td>5.0</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>76.78</td>
<td>91.10</td>
<td>54.4</td>
<td>6.5</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>76.11</td>
<td>89.67</td>
<td>44.5</td>
<td>6.5</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
<td>76.64</td>
<td>90.19</td>
<td>68.2</td>
<td>4.0</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>76.77</td>
<td>89.88</td>
<td>45.1</td>
<td>5.5</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>76.90</td>
<td>89.70</td>
<td>40.5</td>
<td>7.0</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>77.15</td>
<td>87.31</td>
<td>30.0</td>
<td>6.5</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
<td>76.70</td>
<td>87.82</td>
<td>50.2</td>
<td>4.0</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>76.77</td>
<td>86.44</td>
<td>39.1</td>
<td>5.5</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>76.89</td>
<td>85.39</td>
<td>37.4</td>
<td>7.0</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>77.72</td>
<td>84.84</td>
<td>20.4</td>
<td>7.0</td>
</tr>
<tr>
<td>Conventional method$^2$</td>
<td></td>
<td>75.50</td>
<td>89.85</td>
<td>85.9</td>
<td>10.0</td>
</tr>
<tr>
<td>Raw fabric</td>
<td></td>
<td>73.84</td>
<td>44.84</td>
<td>Over 300</td>
<td>-</td>
</tr>
</tbody>
</table>

$^1$NaBO$_3$: 2 g/L, 70 $^\circ$C x 30'; $^2$NaOH: 0.05 g/L, nonionic surface activator: 1 g/L, Na$_2$SiO$_3$: 1 g/L, H$_2$O$_2$: 5 g/L, 90 $^\circ$C x 30"
Absorbability

Figure 4 clearly shows that the water drop disappearance time decreases as the concentrations of TAED and H₂O₂ increase, which indicates an increase in fabrics absorbability. The reason is that, when the original fabrics are scoured, the alkalinity of the pretreatment solution reacts through saponification with the impurities and sizing on the fabrics.

This causes sizing and impurities removal, so that the water molecules can easily enter the fibers. Therefore, the water drop disappearance time of desized and scoured fabrics is superior to that of the original fabrics. As for the two-step/one-bath treatment, as the concentration of H₂O₂, added in the first stage, increases, the absorbability of the fabrics also increases, which is probably due to the fact that more sizing and fibers have already been decomposed. Moreover, when the chemicals (NaBO₃ and TAED) added in the second stage react, alkaline substances are produced.

On the one hand, alkaline substances expand the hollowness of the fibers to increase their absorbability and, on the other, they react with first and second stage impurities, to upgrade fiber absorbability.

Compared to the traditional treatment, less alkaline substances are added into the process, therefore, less sizing and impurities are removed and, thus, a lower absorbability is achieved.

Shrinkage

The phenomenon of shrinkage, occurring during fabric dyeing and processing, has two main causes.

1) After sizing, the warps of the fabric are restricted, as they interlock with the fills. Therefore, when the fabric is scoured bleached, sizing is removed, on the one hand, and the fibers expand, on the other. The net effect is warp decrease, and yarn shrinkage, consequently, occurs.

2) Polyfibers have good heat-molding properties. Therefore, when the fabrics are heated, the fibers instinctively contract. If serious shrinkage occurs during scour bleaching, unevenness will appear on the surface of the C/N fabrics, affecting the follow-up processes.

As seen from Figure 5, the fabrics treated by this method have smaller fill shrinkage (nylon part) than those subjected to traditional treatments, which is mainly due to the fact that traditional treatments are conducted at a higher temperature; therefore, the nylon fibers in the fabrics evidence more noticeable heat shrinkage. In the treatment applied in the present study, shrinkages of the fabrics are smaller than that of the fabrics treated at lower concentrations of H₂O₂. For example, during such treatments, H₂O₂ is not added or it is added only at 2 g/L. The fabrics treated with this solution experience shrinkage of 4% or less, as due to the fact that, at higher concentrations of H₂O₂, the warp fibers have greater possibilities of undergoing oxidization. This probably causes a restraint between the warps (cellulose) and fills (nylon) to be lowered on a greater scale, thus inducing higher shrinkage of the nylon fibers.

Tables 1 and 2 show that the fabrics treated with higher concentrations of NaBO₃ have higher shrinkage values, while the rates are still lower than those of the fabrics treated by traditional methods. This is mainly due to the fact that higher concentrations of NaBO₃ in the solution produce higher
concentrations of sodium hydroxide, which induces a superior scour-bleaching effect. Higher concentrations of sodium hydroxide also increase fiber expansion; therefore, higher shrinkage occurs.

**FTIR analysis**

Figure 6 plots the FTIR analysis of all fabrics. As seen from the original C/N fabric (Fig. 6a), 1051 cm\(^{-1}\) is the absorption peak of >CH–OH, 1725 cm\(^{-1}\) is the absorption peak of the sizing material, poly(methylacrylate) (PMA) or the >C=O group in the oil, 1661 and 1630 cm\(^{-1}\) are the absorption peaks of the >C=O group in the nylon fiber amide group, 1585 cm\(^{-1}\) is the absorption peak of >NH and 3297 and 3466 cm\(^{-1}\) are obviously the absorption peaks of –OH and –NH\(_2\). FTIR spectra for fabrics treated by the traditional method are given in Fig. 6b. The original peaks at 1725 cm\(^{-1}\) disappeared, which indicates that the sizing material (PMA) has been removed. Moreover, the original peaks at 1051 and 1661 cm\(^{-1}\) have nearly disappeared, while another sharp absorption peak is formed at 1634 cm\(^{-1}\). This indicates that the –OH of C2 – C3 in the fibers have been oxidized to the –CHO groups, which can also be verified from the broader absorption peaks at 3297 and 3466 cm\(^{-1}\), replaced by a sharp peak at 3293 cm\(^{-1}\).

For the fabrics treated with a moderate regime (H\(_2\)O\(_2\) 0 g/L, NaBO\(_3\) 1 g/L, TAED 0.5 g/L) used in the study, the FTIR spectra plotted in Figure 6c appear somewhat similar to the IR of the original fabrics; also, the absorption peaks of the amide groups become less sharp, indicating that there are still very small portions of unresolved sizing and oil, and also that cellulose has not been oxidized.

However, if the fabrics are treated under a harsh regime (H\(_2\)O\(_2\) 6 g/L, NaBO\(_3\) 4 g/L, TAED 4 g/L), the IRs are similar to those plotted in Figure 6d. It is obvious that this figure is almost identical to that illustrating the traditional treatment. The conclusion to be reached is therefore that fabrics treated by traditional methods or under a harsh regime (as in the present study) have greater chances of being oxidized, oxidation causing more significant damages to the fibers.

**SEM analysis**

Figure 7, plotting the SEMs of the original fabrics and of the fabrics treated under the harsh regime of this study, shows...
the occurrence of sizing-like substances in-between yarns or on the surface of the original fabric. This is the sizing material. In Figure 7b, the gaps between yarns or surface appear smoother, without inter-sticking, which indicates that the sizing materials have been completely removed from the fabrics.

![Figure 7: SEM of C/N fabrics (a - raw fabric; b - treated fabric (same criteria as in Fig. 6d))](image)

**CONCLUSIONS**

This study explores the effect of TAED and NaBO₃ on C/N fabrics, when treated by the two-steps/one-bath process method. The results of the experiment are summarized as follows:

- Addition of TAED and NaBO₃ lowers the pH values of the pretreatment solutions to almost neutral values.
- FTIR and SEM analyses showed that the method developed in this research can actually induce a favorable effect on the treated fabrics.
- Under an adequate regime, the fabrics treated by this method have properties superior to those treated by traditional methods, with the exception of strength, in part of the treated fabrics.
- Strength of the treated fabrics decreases as the concentrations of H₂O₂, TAED and NaBO₃ increase, although absorbability and whiteness run counter to this pattern. Shrinkage also increases, but not to a considerable extent.
- Fabrics treated with a pretreatment solution composed, each, of 0.2 g/L NaOH, 2 g/L H₂O₂ and 2 g/L of TAED and NaBO₃, in a two-sectional meta-bath treatment, at < 70 °C for 30 min, possess a superior scour – bleaching effect.

**REFERENCES**