

# USE OF T.A.E.D IN THE BLEACHING OF CELLULOSICS TO IMPROVE FIBRE QUALITY

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## Abstract

Over recent years interest in cotton blended fabrics has continued to grow. Enhanced or a combination of performance properties can be delivered to the textile such as durability, elasticity, drape or handle above that achieved by use of a single fibre alone. However, problems may be encountered with the fibre quality of certain cellulosic blends where one or more of the fibre(s) in the blend is susceptible to damage during conventional bleaching processes. For this reason, bleaching cannot be performed under harsh conditions and can sometimes require several bleaching stages to attain whiteness. If peroxide is used under low temperature conditions, performance can be improved by reaction with T.A.E.D which forms a strong oxidant which is a highly effective bleaching agent at low temperatures. Experiments have been undertaken to investigate the use of low temperature peroxide/T.A.E.D based bleaching systems for cotton/viscose and cotton/polyamide blends to improve fibre quality by attaining whiteness without compromising fibre strength.

## Introduction

### Background

A variety of materials are used within the Textile Industry to bleach a diverse range of textile fibres and fabrics. For environmental reasons, hydrogen peroxide is the ideal choice of bleaching reagent. However, in order to gain effective bleaching, high temperatures, high pH or extended reaction times are required. This presents drawbacks in terms of product fibre quality, long residence times required (hence, limiting production throughput), and gives difficulty in the processing of mixed fibre blends where high temperatures or high alkalinity cannot be tolerated. If low temperature conditions are necessary to gain effective bleaching, hypochlorite based processes are often used as a pre-treatment step before peroxide bleaching. However, two stage hypochlorite/peroxide bleaching increases the total processing time required for the bleached goods.

A means of overcoming these drawbacks and boosting the performance of peroxide can be achieved by converting hydrogen peroxide to a stronger oxidising agent which is highly effective at low temperatures. This can be accomplished by use of the bleach activator T.A.E.D (Tetra Acetyl Ethylene Diamine).

Within the Detergent Industry, the bleach activator T.A.E.D has been used for many years to provide effective bleaching, via the production of the peracetic anion, at temperatures and residence times for which peroxide alone would be ineffective.

Work has been undertaken to evaluate fibre quality benefits, in addition to other benefits (shortening of process time, replacement of two stage hypochlorite/peroxide bleaching processes) when T.A.E.D is used in combination with peroxide to bleach cotton blends.

### Chemistry of T.A.E.D

Under aqueous alkaline conditions, one mole of T.A.E.D reacts with two moles of perhydroxide anion to form two moles of peracetic anion and one mole of D.A.E.D (Di Acetyl Ethylene Diamine) Figure 1. It is the peracetic anion which delivers low temperature bleaching performance and additionally, biocidal activity.

Under alkaline conditions, the rate of peracid release is rapid and increases with increasing temperature and pH. Increasing the concentration of peroxide in the system also increases the rate of formation of peracid anion solution. Under alkaline conditions, it is usual to use an excess of peroxide over T.A.E.D to ensure sufficient peroxide is available to force the reaction to completion.

The experiments outlined in this paper were performed under alkaline conditions. Research at Warwick International<sup>(1)</sup> has demonstrated that T.A.E.D will also react under near neutral conditions to form a stronger oxidant believed to be peracetic acid. Under near neutral pH conditions (pH<8), at lower temperatures (60°C), the release of peracid is slower than seen under alkaline conditions but the peracid generated is more stable over a longer time period. These conditions are suited for processes where longer bleaching residence times are used or if fabrics are sensitive to high pH.

Both T.A.E.D and the reaction product D.A.E.D are non toxic, non sensitising and biodegrade to give carbon dioxide, water, nitrate and ammonia as end products. Ethylenediamine is not detected during biodegradation.

Additionally, T.A.E.D is a storage stable solid which is colourless, odourless, safe and easy to handle and can be delivered to the bleach bath in several ways. T.A.E.D is a solid and, thus, can be added directly to the bleach bath. Alternatively T.A.E.D may be delivered to the bleach bath as a solution by dissolving in hot water, or pre-reacting with peroxide prior to addition. To form stable pre-reacted solutions, the pH of the solution must be dropped after the maximum peracid release has been achieved. At such reduced pH, the pre-reacted solution exhibits excellent long term stability. To prepare a pre-reacted solution only a simple mixing vessel is required.

### **Use of T.A.E.D. in Textile Bleaching**

T.A.E.D can be used to bleach a variety of fibres used within the Textile Industry. Recent research has been undertaken at Warwick International to investigate the use of T.A.E.D to bleach mixed fibre blends susceptible to damage by conventional bleaching processes. In particular, work has focused on bleaching of cotton blends to explore potential fibre quality benefits of using peroxide/T.A.E.D based bleaching systems.

### **Results & Discussion**

Batch bleaching experiments were undertaken using a 48/52 cotton/viscose ribbed fabric and sport socks comprising 83/17 cotton/polyamide.

#### **Cotton/Viscose Blend (48/52 mix)**

A low temperature bleaching process using T.A.E.D was investigated to improve the fabric strength of a 48/52 cotton/viscose blend. When bleached to high whiteness, the blend is susceptible to warp breakout problems when bleached using a conventional hypochlorite based process. (Warp is 100% viscose and weft 100% cotton.) Bleaching is required to remove the seed husks prior to dyeing to pale shades and for whites.

#### **Experimental**

Samples of cotton/viscose 48/52 blend (initial CIE Whiteness 42.24) were bleached using a wash wheel at 60°C for 75 minutes. The batch bleaching process combined scouring and bleaching steps in a single stage as shown in Figure 2. The liquor:goods ratio used was 10:1. The T.A.E.D based bleach formulation employed is shown in Table 1.

The bleach liquor was prepared by adding the chemicals to water at 20°C/68°F in the following sequence: stabiliser, sodium hydroxide and peroxide (3.5g/l, 50%w/w). This solution was rapidly heated to 60°C/140°F and the fabric was scoured for 15 minutes. After addition of the remaining peroxide (1g/l, 50% w/w), the pH was adjusted to pH 10 using citric acid and T.A.E.D was added as the last component. The fabric was bleached for 1 hour at 60°C/140°F. After bleaching, fabric was rinsed then washed in a dilute citric acid solution and air dried. The CIE Whiteness was determined using D65 illumination at 10 degrees using a Spectroflash Spectrophotometer. Wet and dry tensile strength was measured in the weft direction. T.A.E.D was added as a granulate, Warwick T200™.

Results in Table 2 clearly show that the target brightness achieved by the hypochlorite process can be matched at 60°C/140°F using a T.A.E.D/peroxide based bleaching system. Alternatively the whiteness can be significantly improved using T.A.E.D if the temperature of the bleaching process is increased to 70°C/158°F. In addition, OBA's can also be readily added directly to the bleach bath to achieve

full whites. Complete seed husk removal was observed on the fabric after bleaching.

The wet and dry tensile strengths of the fabric bleached at 60°C/140°F are shown in Figure 3. The results clearly demonstrate that minimal loss of tensile strength has been incurred by bleaching the cotton/viscose using a peroxide/T.A.E.D based bleaching system. In contrast, the fabric bleached using hypochlorite based process has shown greater than 50% loss in tensile strength (wet and dry).

Hence, the use of low temperature peroxide/T.A.E.D bleaching can greatly improve the fabric strength and can match or boost the brightness of cotton/viscose blends compared to that achieved by commercial hypochlorite based processes.

#### **Cotton/Polyamide (83/17 mix)**

Current commercial processes for bleaching sport socks often involve use of a hypochlorite stage followed by a peroxide bleach stage. Such a two stage hypochlorite/peroxide process can be used to achieve acceptable whiteness. It is important that a low temperature bleach process is used

for processing of socks with coloured markings, to prevent colour bleed. The use of a T.A.E.D/peroxide based batch bleaching process has been investigated to bleach two different types of socks comprising 83% cotton/17% polyamide, one of which contains red pre-dyed markings.

#### **Experimental**

Samples of socks with red dyed markings (initial CIE Whiteness 5.22) and all white socks (initial CIE Whiteness 6.71) were bleached using the wash wheel apparatus which was programmed to hold the temperature at 70°C/158°F for 75 minutes. The type of bleach process used was similar to that detailed for cotton/viscose (Figure 2) and involved a combined scour and bleach process (15 minutes scour at pH~12 followed by a 1 hour bleach at pH~10). In addition, an OBA was added to the bleach bath at the start of the combined scour/bleach process. The bleach formulation used is shown in Table 3.

Results given in Table 4 show that the brightness achieved using the T.A.E.D based bleaching formulation is clearly improved over the socks bleached in a commercial hypochlorite/peroxide process. All the socks bleached using T.A.E.D gave a CIE Whiteness (UV excluded) equivalent to ~67 CIE units. For the socks with red coloured markings and the all white socks, the base bleaching (UV excluded) was approximately 10 units higher than the hypochlorite/peroxide bleached socks. For both the red marked socks and the white socks, results for T.A.E.D based bleach performance (including the OBA) exceeded results obtained by the hypochlorite/peroxide process. Additionally, no evidence of colour bleeding on the socks was observed.

Hence, use of a single stage T.A.E.D based bleaching process for pre-dyed sport socks can be used to boost the whiteness over that achieved by conventional two stage hypochlorite/peroxide bleaching processes. Thus, T.A.E.D provides an alternative processing route.

### Other Application Areas

The use of T.A.E.D is also being investigated for batch bleaching of cellulose such as cotton fibre and fabric, cotton/polyester, wool/cotton and regenerated cellulose. The potential fibre quality benefits in terms of whiteness and strength depend to a certain extent on the substrate being bleached. Additional benefits may include cost and energy savings, by bleaching at lower temperatures and pH, and reduced residence times. T.A.E.D can also be used for bleaching cotton fibre and fabric in continuous processes.

### Summary

Use of T.A.E.D to activate peroxide provides a safe, flexible and effective method of bleaching textiles.

Work has demonstrated that T.A.E.D can be used in conjunction with peroxide to improve the fibre quality of cellulosic blends in terms of improved whiteness and reduced fibre strength loss.

In addition, use of T.A.E.D/peroxide provides a reduction in bleaching time by elimination of a bleaching step and provides an alternative bleaching formulation to hypochlorite for processes at lower temperatures.

References

Warwick International Group Limited Patent : WO 9418298

Table 1. Bleach formulation used for cotton/viscose fabric.

| Formulation                  | Level (g/l)                    |
|------------------------------|--------------------------------|
| Peroxide (50% w/w)           | 4.5 (3.5 scour & 1 for bleach) |
| T.A.E.D (as 100%)            | 1.4                            |
| Stabiliser JLP (ex Clariant) | 2                              |
| Sodium hydroxide (as 47%)    | 4                              |
| Citric acid                  | q.s.                           |

Table 2. 48/52 cotton/viscose results for bleach performance.

| Process                       | CIE Whiteness |
|-------------------------------|---------------|
| Control Hypochlorite          | 71.41         |
| T.A.E.D/Peroxide @ 60°C/140°F | 72.51         |
| T.A.E.D/Peroxide @ 70°C/158°F | 75.14         |

Table 3. Bleach formulation used for cotton/polyamide socks.

| Formulation                  | Level (g/l)                    |
|------------------------------|--------------------------------|
| Peroxide (50% w/w)           | 4.5 (3.5 scour & 1 for bleach) |
| T.A.E.D (as 100%)            | 1.4                            |
| Stabiliser JLP (ex Clariant) | 2                              |
| Sodium hydroxide (as 47%)    | 4                              |
| Uvitex NFB (ex Ciba Geigy)   | 1.2% o.w.f.                    |
| Citric acid                  | q.s.                           |

Table 4. 83/17 cotton/polyamide results for bleach performance.

| Sport sock (type)            | Bleach Process                | CIE Whiteness |               |
|------------------------------|-------------------------------|---------------|---------------|
|                              |                               | U.V. Included | U.V. Excluded |
| sport socks with red marking | hypochlorite/peroxide         | 125.22        | 54.38         |
|                              | T.A.E.D/peroxide @ 70°C/158°F | 133.99        | 66.86         |
| sport socks (all white)      | hypochlorite/peroxide         | 126.68        | 57.2          |
|                              | T.A.E.D/peroxide @ 70°C/158°F | 130.73        | 67.27         |

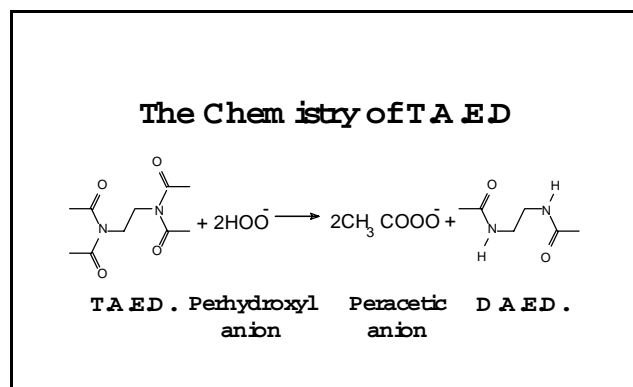


Figure 1. Reaction of T.A.E.D. with peroxide under alkaline conditions.

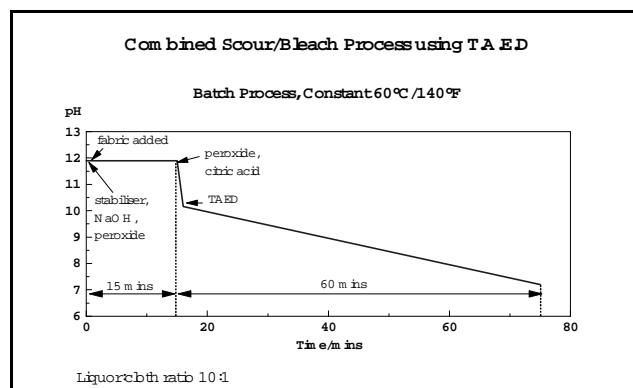


Figure 2. Batch Bleaching Process using T.A.E.D and Peroxide

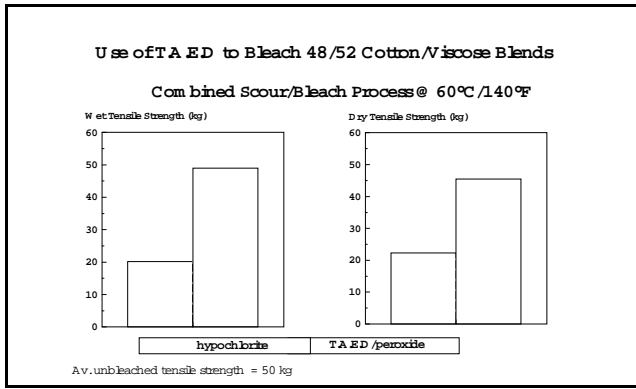


Figure 3. Tensile Strength Results for 48/52 Cotton/Viscose Blends