EFFICIENT, BRIGHTER PEROXIDE BLEACHING OF WOOL/COTTON BLENDS USING TAED P.A. Duffield Woolmark (Europe) Ltd. Ilkley, West Yorkshire Great Britain A.J. Mathews and N.A. Turner Warwick International Group Ltd. Mostyn, Flintshire Great Britain

Abstract

Investigations on hydrogen peroxide bleaching blends of wool and cotton showed that whiteness levels were increased by the addition of tetra acetyl ethylene diamine (TAED). The enhancement was produced in batchwise and pad-batch processes, without adverse effects on tensile properties. Bleaching in an unbuffered bath with an alkaline stabiliser gave the best whiteness.

Introduction

Blends of wool and cotton have been of commercial importance for many years. Intimate blends of wool and cotton were used in khaki military shirtings and continue to be used, particularly in skirts, blouses and women's outerwear. Some cotton warp and wool weft union fabrics were more recently replaced by blends of cotton with synthetic fibres. However, there is a renewed interest in wool/cotton products, based on their comfort properties and consumer demand for natural fibres. Market research showed this demand to be strongest in the product areas of trousers and skirts.

The comfort and aesthetic benefits of wool/cotton blend products were noted in earlier papers and therefore need not be repeated here. It is clear however, that, despite the advantages of wool/cotton over other fibres and blends, consumer demand for these products was not fully met. The main reason for this situation is the technical difficulty of processing wool/cotton products, particularly preparation. dyeing and finishing. Efforts have therefore been made to develop simpler and more economic processing routes. This paper describes recent work on one of these aspects, namely bleaching. It relates to intimate wool/cotton blends of 30/70 and 55/45 composition, which are representative of commercially available products. Cotton rich blends, containing up to 40% wool, process efficiently on short staple machinery and, for this reason, most cotton manufacturers favour wool poor blends. However, on the worsted system wool rich blends perform more efficiently. It is anticipated that the methods discussed will be equally applicable to union (yarn) blends.

The textile industry is well aware of the different processing conditions for wool and cotton. In practice this often means that processes are selected according to the majority fibre in the blend. However, this approach invariably leads to a diminution of the properties contributed by the minority fibre. Therefore, for wool/cotton bleaching an alternative solution was necessary to maintain the aesthetic qualities of both fibres.

Oxidative bleaching methods for wool are invariably based on hydrogen peroxide⁽¹⁾. Increasingly this is also the case for cotton. It was therefore concluded that hydrogen peroxide could provide a basis for bleaching the blend. Cotton is often bleached at higher values of pH and temperature than are acceptable for wool. Therefore, it was essential for the blend to use a process that gave an acceptable bleaching effect under milder conditions than those adopted for 100% cotton. A J Mathews⁽²⁾ described the application of TAED (Tetra Acetyl Ethylene Diamine) for bleaching cellulose fibres at lower temperatures, within a shorter time or with reduced peroxide levels than Investigations were therefore conventional methods. carried out to examine the possibilities of using the same product in bleaching wool/cotton blends.

TAED has been widely used as a bleach activator, to enhance the bleaching effect of peroxy compounds in detergents. This is achieved by the formation of peracetic anion, which is a more effective bleaching species than hydrogen peroxide alone at temperatures less than 122°F. A description of the mechanism was given in S Scarborough's paper, presented at this conference.

Properties of Wool/Cotton Products

Cotton manufacturers will meet much of the demand for wool/cotton products ⁽²⁾. These companies may have little experience with wool and it is therefore pertinent to briefly describe some important aspects of wool and its processing.

Wool is a keratinous fibre, formed primarily of protein chains, crosslinked via disulphide (cystine) bridges. It is therefore not surprising that it can be degraded in strongly alkaline conditions by hydrolysis of amide bonds:

$$\begin{array}{ccc} & \text{Alkali} \\ \text{R}_1\text{-}\text{C-NH-R}_2 & \xrightarrow{} & \text{R}_1\text{-}\text{C-O^-} & + & \text{R}_2\text{-}\text{NH^+} \\ & & \parallel \\ & \text{O} & & \text{O} \end{array}$$

A possible further consequence of alkaline treatment at high temperature is a loss of fibre bulk and, consequently, handle. F Harrigan gives further information on the effects of alkali treatment on wool in his paper. Under alkaline, oxidising conditions other reactions may take place, which can lead to a further breakdown of the keratin structure:

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$\mathbf{R_1R_2CHCH_2SSCH_2CHR_3R_4}$

 $\begin{array}{c} \downarrow & \mbox{Alkali}/\\ \Psi & \mbox{Peroxide} \end{array}$ R_1R_2CHCH_2SO_3- + R_3R_4CHCH_2SO_3-

Alkali solubility is the test normally used to measure oxidative damage to wool, i.e. cleavage of disulphide crosslinks. It is therefore a convenient test for peroxide bleached wool. However, on a wool/cotton blend the method can be imprecise and physical tests must be used. Wool does not have as high tensile strength as cotton. Extensibility is of equal or greater importance in predicting processing efficiency. Therefore, for intimately blended products, both tensile strength and extensibility must be measured to obtain a meaningful estimate of performance.

Conventional Wool Bleaching Processes

Batchwise wool bleaching with hydrogen peroxide is conventionally carried out at pH 5.5-10.0 and temperatures of 104-160°F. Higher pH values normally require lower processing temperatures to minimise the deterioration of fibre properties. Hydrogen peroxide (35% w/w) levels of 15 to 30 ml/l are typically used, depending on the degree of whiteness required. Acid bleaching processes tend to produce whites with slightly redder hues than alkaline bleaching. This can be overcome with a subsequent reductive bleach, which, as on cotton, produces higher whiteness levels.

Many wool bleaching methods are available but one of the most effective, in terms of colour and minimising fibre damage, was based on a peroxide stabiliser developed for cotton. It was therefore concluded that it could also provide the basis of an efficient system for wool/cotton blends. The product is Perowhite BAC (Hydrolabs), also available outside the USA as Stabicol BAC (Allied Colloids).

Wool is seldom bleached by continuous processes, except in fibre scouring, which yields only small improvements in colour. Pad-batch methods, although not widely used, give good whiteness with low levels of fibre damage. They are mainly applied to wool tops or yarn and, unlike cotton are seldom used for bleaching fabric.

Experimental

Materials

24/2 Nec 55/45 wool/cotton yarn 20/2 Nec 30/70 wool/cotton yarn

These yarns were also used in woven fabric for pad-batch processes.

TAED was used as the proprietary product, Warwick T200 (Warwick International), which is specifically formulated for textile applications.

Hydrogen peroxide was 50% w/w.

Methods

The selection of processing route, batch, pad-batch or continuous will depend on the machinery available. Blends of wool and cotton are normally dyed by batch or pad-batch methods. Therefore, the bleaching investigations also concentrated on these two routes. Continuous methods are not so successful on the blend as they are on 100% cotton and were not included, although they may form the subject of future work.

Preliminary batch bleaching experiments were carried out, as described by Mathews⁽²⁾. Subsequent evaluations used a laboratory liquor-circulating machine at a liquor/goods ratio of 12:1.

The following procedure was adopted:

Set the bath cold with Perowhite BAC plus pH buffer, if required, followed by additional chemicals and then hydrogen peroxide.

Raise the bath temperature to $122^{\circ}F$ and add Warwick T200 (1.0 g/l, unless otherwise specified), if required.

Raise the bath temperature to the required value and run for 60 minutes, unless otherwise indicated.

The samples were rinsed twice and then acid soured with acetic acid to approximately pH 5.5.

Pad-batch evaluations were carried out by padding fabric with the indicated solutions, to approximately 80% wet pickup and then batching for 24 hours. The fabrics were then rinsed and acid soured.

Whiteness measurements were made, as previously described ⁽²⁾.

<u>Results</u>

Batch Processing

Preliminary work was carried out in a bleaching bath buffered to pH9.0 at 158°F. These conditions have been shown to maintain good physical properties of blended yarns. Samples were bleached for times up to 90 minutes to assess the potential for reducing process times with TAED. The results in figure 1 show, as anticipated, that increased bleaching time leads to increased whiteness. The addition of TAED also generates significant improvements in whiteness, even at reduced hydrogen peroxide levels, compared to the standard.

The reason for buffering the bleach bath to pH 9.0 was to overcome the fall in pH, which resulted from the formation of peracetic species. The initial bath, without buffer, was

around pH10.0-10.5. However, it was quickly discovered that, at liquor to goods ratios of 10-12:1, the bleach bath fell to around pH 9.0 during the process, thus obviating the need for a buffer system. Bleaching without buffer also gave increased levels of whiteness at all hydrogen peroxide concentrations studied (figures 2 and 3).

These figures also showed that the levels of whiteness achieved on the wool rich blend were lower than those on the cotton rich blend. The higher proportion of cream/yellow pigment in the wool fibres undoubtedly causes this effect. Equally, the benefit gained from addition of TAED was greater on the cotton rich blend.

Based on the above observations, a very simple formulation was adopted for further work:

Perowhite BAC	3.0 g/l
Hydrogen peroxide (50%)	x g/l
Warwick T200	y g/l

Investigations to establish the most suitable concentration for Warwick T200 showed that 1 g/l was close to the optimum value, under the conditions evaluated. On the 30/70 blend, increasing the concentration to 2 g/l yielded no improvement in whiteness (figure 4). On the wool rich blend the higher level of T200 led to a lower whiteness value, that was equivalent to bleaching without the product.

A bleaching temperature of $158^{\circ}F$ was selected on the basis of preliminary laboratory experiments and previous experience with alkaline treatments on wool. Trials on both yarns confirmed that a lower temperature of $140^{\circ}F$ yielded lower whiteness values, with no benefits in tensile properties. $158^{\circ}F$ was therefore adopted as the most appropriate value for batchwise processing. Measurements of yarn tensile properties were used to assess the levels of damage incurred during bleaching (figures 5 and 6). Although both 30/70 and 55/45 wool/cotton yarns were tested, the results were similar and are therefore given only for the latter blend. It was anticipated that, being wool rich, it was potentially more susceptible to damage.

There was little effect of any processing parameters on tensile properties. Unusually, yarn extensibility was increased as a result of bleaching.

Pad-Batch Processing

The effect of processing parameters on whiteness was examined in a series of trials on fabrics woven from yarns of both blends. Hydrogen peroxide concentration and pH were changed and bleachings conducted with and without TAED. Results for the 30/70 blend, with pad liquors buffered to pH 9.0 are shown in figure 7.

The data show no consistent trend in the effect of peroxide concentration. However, there is a suggestion that whiteness increases with decreasing peroxide levels, down to 30g/1. TAED clearly has a beneficial effect on colour

and 0.5g/l gave whiteness values similar to 1.0g/l. With a pad liquor buffered to pH 10.0, whiteness values followed a similar pattern to those illustrated but were approximately five to six units higher. This increase in whiteness at higher pH is similar to the effect obtained from long liquor (batch) processing. The effect of TAED was greater on the 30/70 blend than on the 55/45 blend, again similar to long liquor bleaching.

Tensile tests showed the bleached samples to be slightly stronger and more extensible than the equivalent unbleached fabric. This unusual increase in tensile performance properties is probably caused by the bleached samples being fully relaxed, having been wet processed without tension. This is in contrast to the unbleached, scoured fabric control, which was processed on a commercial scale and tenter dried.

Discussion

The magnitudes of advantages from application of TAED in wool/cotton bleaching depend on the fibre blend ratio and the bleach bath pH. In batchwise processes pH buffers were unnecessary to produce the best whiteness values. Thus a very simple bath formulation may be used. At the higher pH levels, in unbuffered liquors, the greatest benefits arose on cotton rich products. It is unclear why this should be the case, since wool is the yellower of the two fibres and therefore has the greatest potential for bleaching.

The work on batchwise processing was reflected in padbatch bleaching, where better whiteness was produced also with pad liquors at pH 10.0, rather than pH 9.0. There were also greater improvements in colour with TAED on the cotton rich blend.

TAED offers manufacturers the option in their bleaching processes, either to increase product whiteness or reduce processing costs. Cost reductions can be achieved through reduced hydrogen peroxide additions or shorter processing times. These benefits may be obtained without impairment of tensile properties. Under the batchwise bleaching conditions adopted, 100% wool normally undergoes chemical modification that impairs its physical properties. On the wool/cotton blends examined this damage was not It is possible that the cotton component apparent. contributed most of the strength and any reduction in wool strength was masked. However, this does not account for the good extensibility figures, particularly on the wool rich blend. It must therefore be concluded that cotton inhibits alkaline damage to wool, an observation made by other workers.

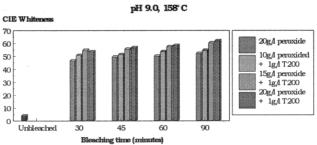
This investigation was carried out on a relatively small scale. Further work is needed to confirm the results and economic advantages on a commercial scale. Additionally, there are obvious areas for further evaluation. These include the combination of bleaching and scouring processes and the application of the technology to blends of wool with other cellulosic fibres.

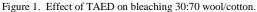
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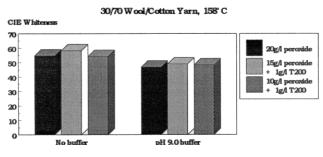


Figure 2. Effect of pH 9.0 Buffer on whiteness.

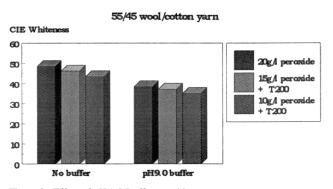


Figure 3. Effect of pH 9.0 Buffer on whiteness.

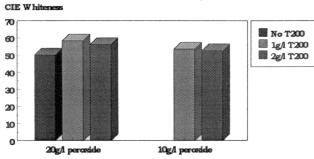
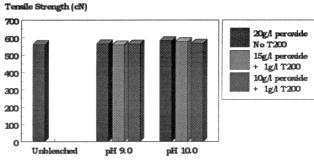


Figure 4. Effect of TAED concentration on whitenes.



55/45 Wool/Cotton Yarn

Figure 5. Effect of bleaching parameters on yarn strength.

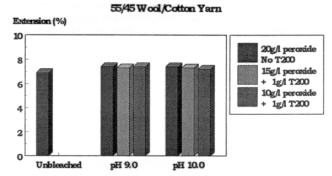
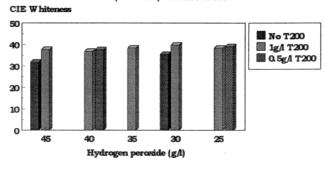


Figure 6. Effect of bleaching parameters on yarn extensibility.



30/70 Wool/Cotton Fabric

Figure 7. Effect of bad-patch bleaching parameters on whitness.

30/70 Wool/Cotton Yarn, pH 9.0