

# The development of a metal-free, tannic acid-based aftertreatment for nylon 6,6 dyed with acid dyes—part 2: further studies

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## Abstract

A single-bath, two-stage aftertreatment for nylon 6,6 dyed with acid dyes has been developed, in which an enzyme is used to complex tannic acid. The effectiveness of the tannic acid/enzyme aftertreatment in improving the fastness to repeated washing of five commercial acid dyes on nylon 6,6 was measured against that of a traditional full backtan aftertreatment. It was found that optimal conditions for application of the enzyme were found to be 70 °C at pH 6. The metal-free, tannic acid/enzyme aftertreatment offers a potentially more environmentally acceptable alternative to the antimony-based and tin-based systems

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**Keywords:** Nylon 6,6; Acid dyes; Full backtan; Tannic acid; Enzyme

## 1. Introduction

This paper concerns the development of a metal-free, tannic acid-based aftertreatment for acid dyed nylon, with the aim of providing a potentially more environmentally acceptable aftertreatment to the traditional full backtan. The previous part of this paper [1] showed that the effectiveness of a newly developed, tannic acid/enzyme aftertreatment in improving the fastness

to repeated washing of three commercial acid dyes on nylon 6,6, was comparable to that of four established aftertreatments which had been chosen as ‘references’ against which the developed tannic acid/enzyme system was compared.

This part of the paper concerns the determination of the optimum application conditions for application of the tannic acid/enzyme system. The precise mechanism by which the tannic acid/enzyme aftertreatment operates will be the focus of a subsequent part of this paper. As before [1], in recognition of the fact that different washing temperatures are employed in Northern Europe, the effectiveness of the tannic acid/enzyme system was determined at three washing temperatures, namely 40, 50 and 60 °C, using a repeated wash fastness testing protocol.

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## 2. Experimental

### 2.1. Materials

Scoured, knitted nylon 6.6 fabric (which was kindly supplied by Dupont (UK)) described earlier [1] was used. Five commercial acid dyes (Table 1) were used, each having been generously supplied by Yorkshire Chemicals. A commercial sample of *Textan 3* (tannic acid) was kindly provided by OmniChem-Ajinomoto and a commercial sample

of the enzyme *Savinase* was generously supplied by Novazyme.

All other chemicals used were laboratory grade reagents.

### 2.2. Dyeing

The dyes used (Table 1) were applied using the equipment described earlier [1] following the method shown in Fig. 1; the pH was adjusted using McIlvaine buffer [2]. The dyeings were rinsed thoroughly in tap water and allowed to dry in the open air.

### 2.3. Enzyme treatment

The aftertreatment method is shown in Fig. 2; the equipment described earlier [1] was used, the pH of application being adjusted using McIlvaine buffer [2]. At the end of treatment, the samples were removed, rinsed thoroughly in tap water and allowed to dry in the open air.

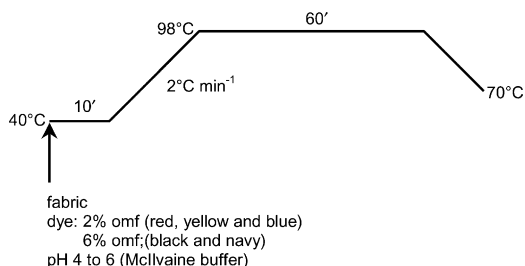


Fig. 1. Dyeing method.

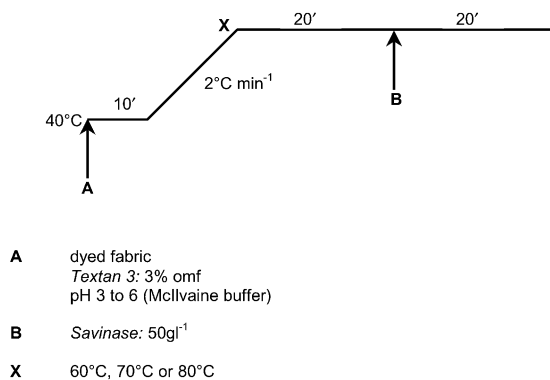


Fig. 2. Tannic acid/enzyme application method.

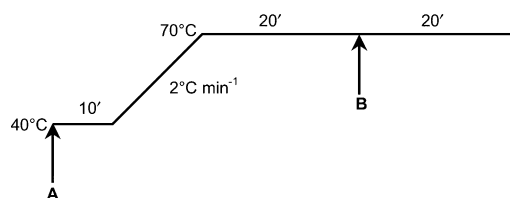


Fig. 3. Full backtan application method.

Table 1  
Dyes used

Commercial name	Type	C.I generic name
<i>Neutrilan Red K-2G</i>	Unsulphonated 1:2 pre-metallised	Acid Red 278
<i>Nylanthrene Yellow C-3RL</i>	Non-metallised acid	Acid Orange 67
<i>Nylanthrene Blue C-GLF</i>	Non-metallised acid	Acid Blue 281
<i>Nylanthrene Black C-DPL</i>	Non-metallised acid	Acid Black 172
<i>Neutrilan Blue S-BGR</i>	Monosulphonated 1:2 pre-metallised	Acid Blue 284

## 2.4. Full backtan aftertreatment

The aftertreatment method is given in Fig. 3; the equipment described earlier [1] was used, the application pH (pH 3) being adjusted using McIlvaine buffer [2]. The aftertreated samples were removed, rinsed thoroughly in tap water and allowed to air dry.

## 2.5. Colour measurement

All measurements were carried out using the equipment and procedures described earlier [1].

## 2.6. Wash-fastness

The wash fastness of the dyed samples to five, consecutive wash tests was determined at three temperatures (40, 50 and 60 °C), using the modified, ISO standard wash tests (ISO CO6/A2S (40 °C), ISO CO6/B2S (50 °C) and ISO CO6/C2S (60 °C) [3]) described earlier [1]. The extent of the staining of adjacent multifibre strip was expressed in the appropriate staining grey scale whereas the change in shade of the sample after washing was expressed in CIE  $L^*a^*b^*$   $\Delta E$  units.

## 2.7. Light fastness

The fastness of the dyeings to light was determined using the ISO B02 method [3].

## 3. Results and discussion

### 3.1. Background

As previously explained [1], three common washing temperatures were used in this work, namely 40, 50 and 60 °C, in cognisance on the fact that washing temperatures vary within Europe. In the first part of this paper [1], three commercial acid dyes were chosen for use on the basis that each displayed moderate fastness to repeated washing on the knitted nylon 6,6 fabric substrate employed. A decision was made in this part of the work to extend the number of dyes studied to five; the additional two dyes (*Nylanthrene Blue C-GLP* and *Neutrilan Navy S-BGR*) were selected because each displayed moderate fastness to the repeated wash testing system used. As the aim of this work was to develop an aftertreatment to improve the fastness to repeated washing, it seems appropriate firstly to establish the levels of fastness of the five dyes used.

In this context, Tables 2–4 show the shade change which the dyeings underwent when they were subjected to five, consecutive wash tests at the three washing temperatures. From the colorimetric and colour strength data presented, the moderate fastness of the five dyes to repeated washing is clearly evident, insofar as the dyeings underwent a reduction in colour strength  $f(k)$  due to the loss of dye during washing. From the  $f(k)$

Table 2  
Colorimetric data and wash fastness results for untreated dyeings washed at 40 °C<sup>a</sup>

Dye	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
Red K-2G <sup>#</sup>	0	42.2	44.3	17.6	47.7	21.7	91.0
	5	44.0	43.0	18.0	46.6	22.7	78.7
Yellow C-3RL <sup>#</sup>	0	69.9	28.8	71.2	76.8	68.0	66.9
	5	70.5	27.8	68.8	74.6	68.0	50.1
Blue C-GLF	0	42.3	−2.0	−37.9	38.0	267.0	62.7
	5	44.3	−2.9	−36.6	37.0	266.0	52.8
Black C-DPL <sup>#</sup>	0	24.9	−0.3	−3.9	3.9	265.3	180.7
	5	26.5	−0.3	−3.9	3.9	265.6	160.4
Navy S-BGR	0	15.3	2.7	−14.4	14.6	280.6	391.0
	5	15.9	2.7	−15.4	15.6	280.2	373.4

<sup>a</sup> Data from Ref. [1].

values shown, it is also apparent that the reduction in colour strength increased with increasing temperature of wash testing; this expected finding can be attributed to a corresponding increase in the extent of removal of dye from the dyed samples during wash testing. The colorimetric data presented in Tables 2–4 reveal that the shade changes observed for the three dyes were attributable to a loss of dye from the dyeings rather than to changes in the colour of the dyeings. The magnitude of the reduction in colour strength of the dyeings that occurred as a result of repeated washing is evident in Fig. 4 which shows the colour difference (DE) obtained between unwashed dyeings and dyeings which had been subjected to five, repeated wash tests. The effect of washing

temperature on the extent of the shade change that the dyeings underwent is clearly evident.

Table 5 shows the extent of staining of multi-fibre strip obtained as a result of the five, consecutive wash tests. As previously discussed [1], the low level of staining obtained for the adjacent acrylic, polyester and cotton components was anticipated in view of the inherent low substantivity of the three acid dyes towards such fibre types; also expected, from the viewpoint of the substantivity of the three dyes used, was the very high extent of staining obtained for the adjacent nylon 6,6 fibre and the moderate staining of the wool component. The results presented in Table 5 clearly show that the level of staining of the adjacent materials increased, markedly, with increase

Table 3

Colorimetric data and wash fastness results for untreated dyeings washed at 50 °C<sup>a</sup>

Dye	Number of washes	<i>L</i> <sup>*</sup>	<i>a</i> <sup>*</sup>	<i>b</i> <sup>*</sup>	<i>C</i> <sup>*</sup>	<i>h</i> <sup>o</sup>	<i>f(k)</i>
Red K-2G <sup>#</sup>	0	42.2	44.3	17.6	47.7	21.7	91.0
	5	44.5	42.9	18.1	46.5	22.8	76.6
Yellow C-3RL <sup>#</sup>	0	69.9	28.8	71.2	76.8	68.0	66.9
	5	71.4	26.9	66.7	71.9	68.0	48.9
Blue C-GLF	0	42.3	−2.0	−37.9	38.0	267.0	62.7
	5	46.0	−3.1	−36.1	36.2	265.1	47.4
Black C-DPL <sup>#</sup>	0	24.9	−0.3	−3.9	3.9	265.3	180.7
	5	26.7	−0.3	−3.9	3.9	266.1	156.0
Navy S-BGR	0	15.3	2.7	−14.4	14.6	280.6	391.0
	5	16.0	2.7	−15.4	15.7	280.0	367.7

<sup>a</sup> Data from Ref. [1].

Table 4

Colorimetric data and wash fastness results for untreated dyeings washed at 60 °C<sup>a</sup>

Dye	Number of washes	<i>L</i> <sup>*</sup>	<i>a</i> <sup>*</sup>	<i>b</i> <sup>*</sup>	<i>C</i> <sup>*</sup>	<i>h</i> <sup>o</sup>	<i>f(k)</i>
Red K-2G <sup>#</sup>	0	42.2	44.3	17.6	47.7	21.7	91.0
	5	44.9	42.6	18.4	46.4	23.4	73.2
Yellow C-3RL <sup>#</sup>	0	69.9	28.8	71.2	76.8	68.0	66.9
	5	73.3	24.3	63.3	67.8	69.0	37.5
Blue C-GLF	0	42.0	−1.7	−37.8	37.9	267.5	63.5
	5	48.9	−4.0	−34.3	34.5	263.3	37.3
Black C-DPL <sup>#</sup>	0	24.9	−0.3	−3.9	3.9	265.3	180.7
	5	26.9	−0.3	−3.9	3.9	265.7	153.0
Navy S-BGR	0	15.3	2.7	−14.4	14.6	280.6	391.0
	5	16.3	2.6	−15.2	15.4	279.7	355.2

<sup>a</sup> Data from ref. [1].

in washing temperature; this can be attributed to a corresponding increase in the amount of dye removed from the dyeings as the temperature at which wash fastness testing was increased (Tables 2–4).

The results presented clearly show that each of the five dyes used displayed moderate fastness to repeated washing.

### 3.2. Full backtan

As the purpose of this work was to develop a metal-free, tannic acid-based aftertreatment as an

alternative to the traditional full backtan, it was decided to use the full backtan aftertreatment as a reference against which the effectiveness of the tannic acid/enzyme system would be judged. In this section of the work, dyeings were aftertreated with the full backtan and subjected to five, repeated wash tests at 40, 50 and 60 °C.

Tables 6–8 show the colorimetric data obtained for dyeings which had been aftertreated with the full backtan and subjected to repeated wash testing. Comparison of this data with that obtained for the untreated dyeings (Tables 2–4) reveals that aftertreatment markedly improved the wash

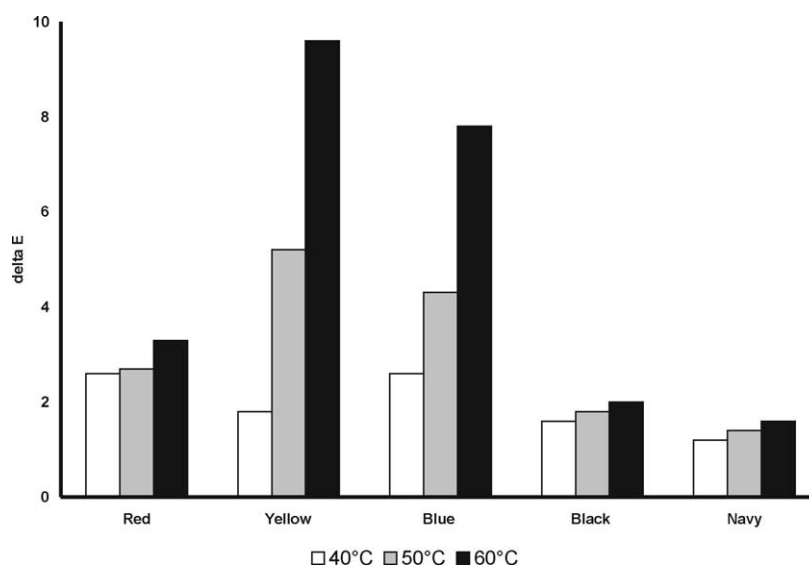


Fig. 4. Colour difference between unwashed and washed dyeings: untreated.

Table 5  
Staining of adjacent multifibre strip achieved for untreated dyeings<sup>a</sup>

Dye	Number of washes	Wool	Acrylic	Polyester	Nylon 6,6	Cotton	2° Acetate
Red K-2G <sup>#</sup>	1	<b>5</b> 5* (3)	<b>5</b> 5* (5)	<b>5</b> 5* (5)	<b>2/3</b> 1* (1)	<b>5</b> 5* (5)	<b>5</b> 5* (5)
	5	<b>5</b> 5* (4)	<b>5</b> 5* (5)	<b>5</b> 5* (5)	<b>3</b> 2* (1)	<b>5</b> 5* (5)	<b>5</b> 5* (5)
Yellow C-3RL <sup>#</sup>	1	<b>5</b> 2/3* (1/2)	<b>5</b> 4* (4)	<b>5</b> 4* (3)	<b>2/3</b> 1/2* (1)	<b>5</b> 4* (4)	<b>2/3</b> 1/2* (1)
	5	<b>5</b> 3/4* (2/3)	<b>5</b> 5* (5)	<b>5</b> 4/5* (4)	<b>3</b> 2/3* (2)	<b>5</b> 4/5* (4/5)	<b>3</b> 2/3* (2)
Blue C-GLF	1	<b>4</b> 1/2* (1)	<b>5</b> 5* (5)	<b>4/5</b> 4/5* (3)	<b>1/2</b> 1* (1)	<b>3/4</b> 2/3* (2)	<b>2/3</b> 1/2* (1)
	5	<b>4/5</b> 2/3* (1/2)	<b>5</b> 5* (5)	<b>4/5</b> 5* (3/4)	<b>2</b> 1/2* (1)	<b>4</b> 3* (2/3)	<b>3</b> 2/3* (2)
Black C-DPL <sup>#</sup>	1	<b>4/5</b> 3/4* (3)	<b>5</b> 3* (2/3)	<b>5</b> 4/5* (4/5)	<b>1/2</b> 1/2* (1)	<b>5</b> 5* (4/5)	<b>5</b> 5* (5)
	5	<b>5</b> 4* (4)	<b>5</b> 4/5* (4)	<b>5</b> 5* (5)	<b>2</b> 2/3* (2)	<b>5</b> 5* (5)	<b>5</b> 5* (5)
Navy S-BGR	1	<b>5</b> 3* (2)	<b>5</b> 4/5* (4)	<b>5</b> 5* (4/5)	<b>1/2</b> 1* (1)	<b>5</b> 5* (4/5)	<b>5</b> 5* (5)
	5	<b>5</b> 4* (3)	<b>5</b> 5* (5)	<b>5</b> 5* (5)	<b>2/3</b> 2* (1/2)	<b>5</b> 5* (5)	<b>5</b> 5* (5)

<sup>a</sup> Bold = 40 °C; \* = 50 °C; ( ) = 60 °C; # data from Ref. [1].

Table 6

Colorimetric data for full backtanned dyeings washed at 40 °C<sup>a</sup>

Dye	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	<i>f(k)</i>
Red K-2G <sup>#</sup>	0	41.0	43.1	16.4	46.1	20.8	97.7
	5	41.2	43.7	16.4	46.7	20.6	97.2
Yellow C-3RL <sup>#</sup>	0	68.2	27.9	71.3	76.5	68.6	76.0
	5	69.0	27.3	70.0	75.1	68.7	74.6
Blue C-GLF	0	41.4	−2.2	−37.4	37.5	266.6	63.9
	5	42.0	−2.7	−36.6	36.7	265.8	61.3
Black C-DPL <sup>#</sup>	0	23.4	−0.4	−4.0	4.0	264.3	202.5
	5	23.7	−0.4	−3.6	3.6	263.7	200.8
Navy S-BGR	0	14.4	2.3	−13.6	13.8	279.6	397.9
	5	14.7	2.4	−14.2	14.4	279.6	396.7

<sup>a</sup> Data from Ref. [1].

Table 7

Colorimetric data and wash fastness results for full backtanned dyeings washed at 50 °C<sup>a</sup>

Dye	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	<i>f(k)</i>
Red K-2G <sup>#</sup>	0	41.0	43.1	16.4	46.1	20.8	97.7
	5	41.3	43.9	16.3	46.8	20.4	96.9
Yellow C-3RL <sup>#</sup>	0	68.2	27.9	71.3	76.5	68.6	76.0
	5	69.3	26.7	68.8	73.8	68.8	74.0
Blue C-GLF	0	41.4	−2.2	−37.4	37.5	266.6	63.9
	5	42.3	−3.1	−35.8	35.9	265.1	59.7
Black C-DPL <sup>#</sup>	0	23.4	−0.4	−4.0	4.0	264.3	202.5
	5	23.9	−0.4	−3.5	3.6	263.5	199.8
Navy S-BGR	0	14.4	2.3	−13.6	13.8	279.6	397.9
	5	14.9	2.1	−14.3	14.5	278.4	394.6

<sup>a</sup> Data from Ref. [1].

Table 8

Colorimetric data and wash fastness results for full backtanned dyeings washed at 60 °C<sup>a</sup>

Dye	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	<i>f(k)</i>
Red K-2G <sup>#</sup>	0	41.0	43.1	16.4	46.1	20.8	97.7
	5	41.4	44.1	16.2	47.0	20.2	96.0
Yellow C-3RL <sup>#</sup>	0	68.2	27.9	71.3	76.5	68.6	76.0
	5	70.0	26.0	67.2	72.1	68.8	73.3
Blue C-GLF	0	41.4	−2.2	−37.4	37.5	266.6	63.9
	5	43.4	−4.0	−34.0	34.2	263.3	58.8
Black C-DPL <sup>#</sup>	0	23.4	−0.4	−4.0	4.0	264.3	202.5
	5	24.0	−0.5	−3.4	3.4	261.6	198.7
Navy S-BGR	0	14.4	2.3	13.6	13.8	279.6	397.9
	5	15.1	2.1	−14.4	14.6	278.3	392.7

<sup>a</sup> Data from Ref. [1].

fastness of each of the dyes to repeated wash fastness, in terms of change in shade of the dyeings. Also, it is clear that aftertreatment increased the colour strength and flattened the shade of the dyeings; in the cases of the red and yellow dyeings, aftertreatment also imparted a yellow colour. As previously discussed [1], these findings can be attributed to the well known fact that the full backtan aftertreatment can alter the shade of dyeings [4]. Fig. 5 shows the colour difference obtained for the full backtan treated dyeings after five repeated wash tests. Comparison of Fig. 5 with that obtained for the untreated dyeings

(Fig. 4) shows that aftertreatment reduced the shade change of the dyeings in terms of the colour difference obtained after repeated wash testing at the three temperatures used. The full backtan was especially effective in reducing the extent of staining of the adjacent multifibre strip during repeated wash testing, as evidenced by a comparison of the staining data achieved for the untreated dyeings (Table 5) with those obtained for the backtanned dyeings (Table 9).

Thus, aftertreatment with the full backtan markedly improved the fastness of the five dyes to repeated washing in terms of both change in shade

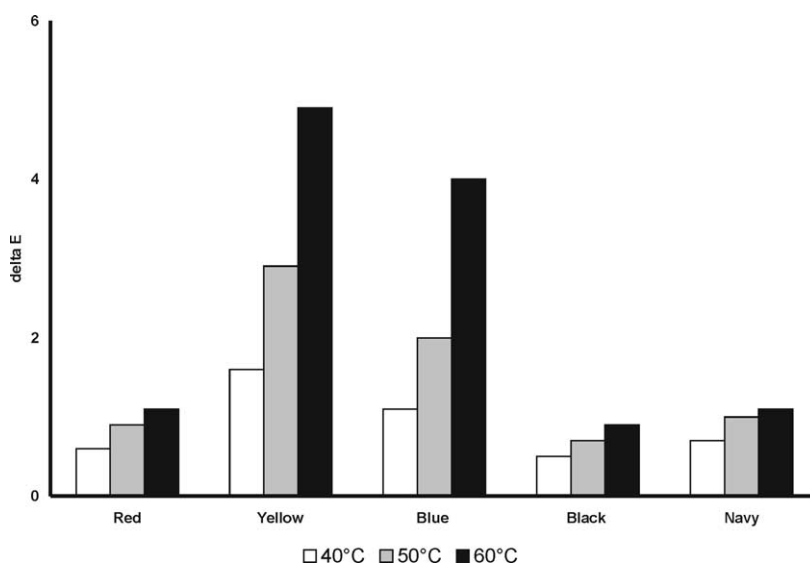


Fig. 5. Colour difference between unwashed and washed dyeings: full backtan.

Table 9  
Staining of adjacent multifibre strip achieved for backtanned dyeings<sup>a</sup>

Dye	Number of washes	Wool	Acrylic	Polyester	Nylon 6,6	Cotton	2° Acetate
Red K-2G <sup>#</sup>	1	5 5* (5)	5 5* (5)	5 5* (5)	4/5 4/5* (2/3)	5 5* (5)	5 5* (5)
	5	5 5* (4/5)	5 5* (5)	5 5* (5)	5 4* (2)	5 5* (5)	5 5* (5)
Yellow C-3RL <sup>#</sup>	1	5 5* (4/5)	5 5* (5)	5 5* (5)	5 5* (4)	5 5* (5)	5 5* (5)
	5	5 5* (4/5)	5 5* (5)	5 5* (5)	5 4/5* (3)	5 5* (5)	5 5* (5)
Blue C-GLF	1	5 4/5* (3)	5 5* (5)	5 5* (4/5)	4 4* (2)	5 5* (3)	5 4/5* (2/3)
	5	5 4* (4)	5 5* (5)	5 5* (5)	4 3/4* (1/2)	5 5* (3)	5 5* (2)
Black C-DPL <sup>#</sup>	1	5 5* (4/5)	5 5* (5)	5 5* (5)	4/5 4/5* (3/4)	5 5* (5)	5 5* (5)
	5	5 5* (4/5)	5 5* (5)	5 5* (5)	5 4/5* (3/4)	5 5* (5)	5 5* (4/5)
Navy S-BGR	1	5 4/5* (4/5)	5 5* (5)	5 5* (5)	5 4* (2/3)	5 5* (4/5)	5 5* (5)
	5	5 4/5* (4/5)	5 5* (5)	5 5* (5)	5 4/5* (3/4)	5 5* (5)	5 5* (4/5)

<sup>a</sup> Legend as for Fig. 5; \* data from Ref. [1].

of dyeings and staining of adjacent multifibre strip.

### 3.3. Tannic acid/enzyme aftertreatment

Initial findings [1] revealed that when the potassium antimonyl tartrate component in the full backtan was replaced by the enzyme *Savinase*, the tannic acid/enzyme aftertreatment was comparable to that of four 'reference' aftertreatments in improving the repeated wash fastness of three acid dyes. In the first part of the work [1], as no references were found pertaining to the use of an enzyme in conjunction with tannic acid for the improvement of the fastness of acid dyes on nylon, a decision was made to apply the enzyme using the same application conditions (ie pH, temperature, time, etc.) that had been used for the full backtan. However, it was decided that an investigation should be made of the effects of temperature and pH of application of the enzyme on the efficacy of

the aftertreatment. Consequently, in this part of the work, the application of *Savinase* was carried out at four pH values (pH 3, 4, 5 and 6) and three temperatures (60, 70 and 80 °C) using the method shown in Fig. 3.

### 3.4. Effect of pH

In this section of the work, the enzyme was applied at pH 3, 4, 5 and 6 at 70 °C and the dyeings were subjected to five, repeated wash tests at 40, 50 and 60 °C.

Fig. 6 shows the extent to which the tannic acid/enzyme system reduced the shade change of the dyeings in terms of the colour difference obtained after repeated wash testing at each of the three washing temperatures. It is apparent that, as expected, the extent of the change in shade obtained for each dye increased with increasing wash temperature. Also, for each of the five dyes used, the extent of the shade change obtained

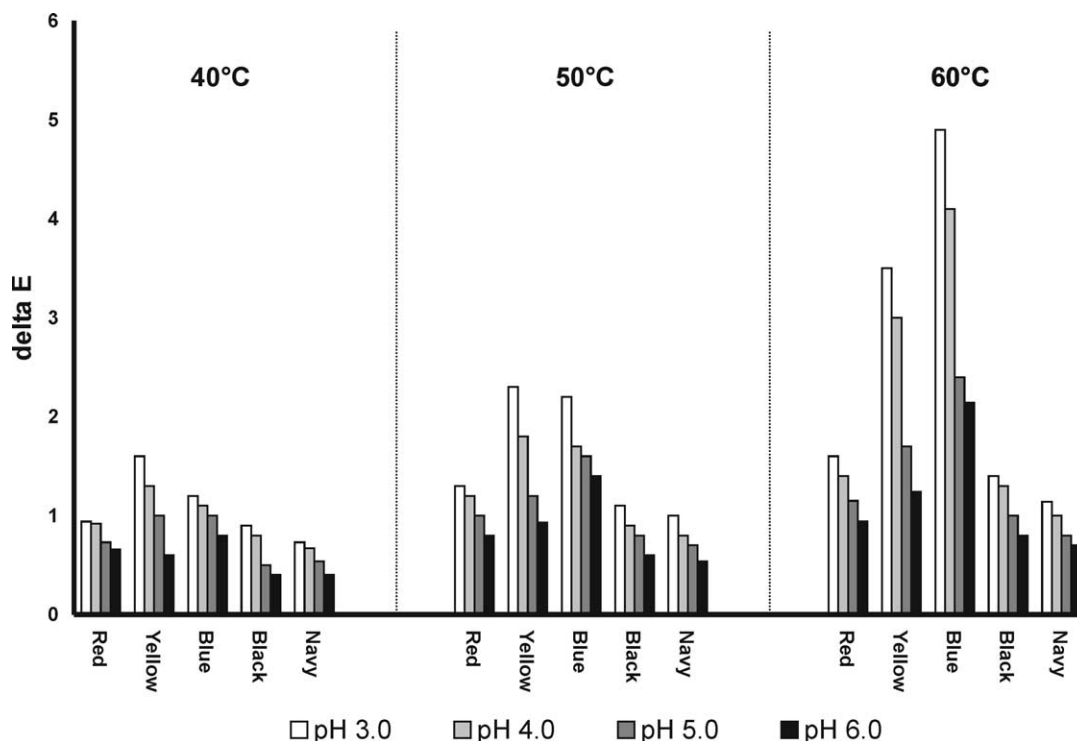


Fig. 6. Effect of pH of application of enzyme on the colour difference obtained for tannic acid/enzyme system at different washing temperatures.

Table 10

Effect of pH of aftertreatment on the wet fastness: *Neutrilan Red K-2G* washed at 40 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	42.0	43.9	17.2	47.1	21.4	91.4
	5	42.2	43.3	17.0	46.5	21.4	90.9
5.0	0	42.0	43.8	17.1	47.0	21.3	91.2
	5	42.3	43.2	16.8	46.3	21.2	90.5
4.0	0	41.4	43.6	16.7	46.7	21.0	92.2
	5	41.8	42.8	16.5	45.9	21.1	90.9
3.0	0	41.2	43.5	16.6	46.6	20.9	92.8
	5	41.8	42.8	16.4	45.8	21.0	91.3

Table 11

Effect of pH of aftertreatment on the wet fastness: *Nylanthrene Yellow C-3RL* washed at 40 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	68.9	28.6	74.6	79.9	68.4	68.9
	5	69.3	28.2	74.4	79.6	69.2	68.5
5.0	0	68.8	28.5	74.4	79.7	69.0	69.3
	5	69.4	28.0	73.8	78.9	69.2	68.6
4.0	0	68.5	28.3	73.9	79.1	69.0	70.1
	5	69.4	28.1	73.0	78.2	68.9	68.9
3.0	0	68.2	28.0	73.6	78.7	69.2	70.5
	5	69.2	27.7	72.4	77.5	69.1	69.1

Table 12

Effect of pH of aftertreatment on the wet fastness: *Nylanthrene Blue C-GLF* washed at 40 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	41.9	−2.0	−38.1	38.2	267.0	62.9
	5	42.4	−2.3	−37.5	37.6	266.5	62.0
5.0	0	41.8	−1.9	−38.2	38.3	267.2	63.2
	5	42.3	−2.3	−37.4	37.5	266.5	62.1
4.0	0	41.6	−1.7	−38.4	38.4	267.5	63.5
	5	42.2	−2.2	−37.6	37.7	266.7	62.2
3.0	0	41.5	−1.6	−38.5	38.5	267.5	63.7
	5	42.1	−2.3	−37.7	37.8	266.5	62.2

Table 13

Effect of pH of aftertreatment on the wet fastness: *Nylanthrene Black C-DPL* washed at 40 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	24.5	−0.3	−3.8	3.8	265.5	191.2
	5	24.9	−0.3	−3.7	3.7	265.4	190.6
5.0	0	24.3	−0.3	−3.7	3.7	265.4	192.0
	5	24.8	−0.3	−3.6	3.6	265.2	190.9
4.0	0	24.0	−0.2	−3.7	3.7	266.9	194.9
	5	24.8	−0.2	−3.6	3.6	266.8	193.1
3.0	0	23.7	−0.2	−3.6	3.6	266.8	197.2
	5	24.6	−0.2	−3.5	3.5	266.7	195.0

Table 14

Effect of pH of aftertreatment on the wet fastness: *Nuetrilan Navy S-BGR* washed at 40 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	15.1	2.6	−14.1	14.3	280.5	393.1
	5	15.2	2.6	−14.5	14.7	280.2	392.6
5.0	0	14.9	2.5	−14.0	14.2	280.1	393.9
	5	15.1	2.5	−14.5	14.7	279.8	393.0
4.0	0	14.6	2.3	−13.8	14.0	279.4	395.0
	5	14.9	2.3	−14.4	14.6	279.1	393.7
3.0	0	14.5	2.3	−13.6	13.8	279.6	395.6
	5	14.9	2.2	−14.2	14.4	278.8	394.1

Table 15

Effect of pH of enzyme application on wash fastness: *Neutrilan Red K-2G* washed at 50 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	42.0	43.9	17.2	47.1	21.4	91.4
	5	42.3	43.2	17.0	46.4	21.5	90.7
5.0	0	42.0	43.8	17.1	47.0	21.3	91.2
	5	42.4	43.0	16.7	46.1	21.2	90.2
4.0	0	41.4	43.6	16.7	46.7	21.0	92.2
	5	41.9	42.6	16.4	45.7	21.1	90.1
3.0	0	41.2	43.5	16.6	46.6	20.9	92.8
	5	42.1	42.6	16.3	45.6	21.0	90.2

Table 16

Effect of pH of enzyme application on wash fastness: *Nylanthrene Yellow C-3RL* washed at 50 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	68.9	28.6	74.6	79.9	68.4	68.9
	5	69.5	28.1	74.1	79.3	69.2	68.2
5.0	0	68.8	28.5	74.4	79.7	69.0	69.3
	5	69.6	27.9	73.7	78.8	69.3	68.3
4.0	0	68.5	28.3	73.9	79.1	69.0	70.1
	5	69.7	28.1	72.6	77.9	68.8	68.4
3.0	0	68.2	28.0	73.6	78.7	69.2	70.5
	5	69.8	27.6	71.6	77.0	69.0	68.3

Table 17

Effect of pH of enzyme application on wash fastness: *Nylanthrene Blue C-GLF* washed at 50 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	41.9	−2.0	−38.1	38.2	267.0	62.9
	5	42.6	−2.6	−37.1	37.2	266.0	61.5
5.0	0	41.8	−1.9	−38.2	38.3	267.2	63.2
	5	42.7	−2.8	−37.3	37.4	266.5	61.0
4.0	0	41.6	−1.7	−38.4	38.4	267.5	63.5
	5	42.6	−2.7	−37.4	37.5	265.9	60.8
3.0	0	41.5	−1.6	−38.5	38.5	267.5	63.7
	5	42.6	−2.6	−36.9	37.0	266.0	60.3

Table 18

Effect of pH of enzyme application on wash fastness: *Nylanthrene Black C-DPL* washed at 50 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	24.5	−0.3	−3.8	3.8	265.5	191.2
	5	25.1	−0.3	−3.7	3.7	265.4	189.7
5.0	0	24.3	−0.3	−3.7	3.7	265.4	192.0
	5	25.1	−0.3	−3.5	3.5	265.1	189.9
4.0	0	24.0	−0.2	−3.7	3.7	266.9	194.9
	5	24.9	−0.2	−3.5	3.5	266.7	192.5
3.0	0	23.7	−0.2	−3.6	3.6	266.8	197.2
	5	24.8	−0.2	−3.4	3.4	266.6	193.9

Table 19

Effect of pH of enzyme application on wash fastness: *Neutrilan Navy S-BGR* washed at 50 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	15.1	2.6	−14.1	14.3	280.5	393.1
	5	15.3	2.6	−14.6	14.8	280.1	392.0
5.0	0	14.9	2.5	−14.0	14.2	280.1	393.9
	5	15.1	2.5	−14.7	14.9	279.6	392.4
4.0	0	14.6	2.3	−13.8	14.0	279.4	395.0
	5	15.0	2.2	−14.5	14.7	278.6	393.0
3.0	0	14.5	2.3	−13.6	13.8	279.6	395.6
	5	15.1	2.2	−14.4	14.6	278.7	392.8

Table 20

Effect of pH of enzyme application on wash fastness: *Neutrilan Red K-2G* washed at 60 °C

PH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	42.0	43.9	17.2	47.1	21.4	91.4
	5	42.4	43.1	16.9	46.3	21.4	90.3
5.0	0	42.0	43.8	17.1	47.0	21.3	91.2
	5	42.6	42.9	16.7	46.0	21.3	89.7
4.0	0	41.4	43.6	16.7	46.7	21.0	92.2
	5	42.2	42.5	16.4	45.6	21.1	89.6
3.0	0	41.2	43.5	16.6	46.6	20.9	92.8
	5	42.3	42.4	16.3	45.4	21.0	89.8

Table 21

Effect of pH of enzyme application on wash fastness: *Nylanthrene Yellow C-3RL* washed at 60 °C

PH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	68.9	28.6	74.6	79.9	68.4	68.9
	5	69.8	28.1	73.9	79.1	69.2	67.8
5.0	0	68.8	28.5	74.4	79.7	69.0	69.3
	5	69.9	27.8	73.3	78.4	69.2	67.8
4.0	0	68.5	28.3	73.9	79.1	69.0	70.1
	5	69.9	28.0	71.9	77.2	68.7	67.6
3.0	0	68.2	28.0	73.6	78.7	69.2	70.5
	5	70.4	27.3	71.0	76.1	69.0	67.5

Table 22

Effect of pH of enzyme application on wash fastness: *Nylanthrene Blue C-GLF* washed at 60 °C

PH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	41.9	−2.0	−38.1	38.2	267.0	62.9
	5	43.0	−2.9	−36.5	36.6	265.5	60.0
5.0	0	41.8	−1.9	−38.2	38.3	267.2	63.2
	5	43.3	−3.1	−36.8	36.9	265.2	59.7
4.0	0	41.6	−1.7	−38.4	38.4	267.5	63.5
	5	43.4	−3.7	−36.3	36.5	264.2	58.5
3.0	0	41.5	−1.6	−38.5	38.5	267.5	63.7
	5	44.1	−4.4	−35.4	35.7	262.9	58.0

Table 23

Effect of pH of enzyme application on wash fastness: *Nylanthrene Black C-DPL* washed at 60 °C

PH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	24.5	−0.3	−3.8	3.8	265.5	191.2
	5	25.3	−0.3	−3.6	3.6	265.2	188.4
5.0	0	24.3	−0.3	−3.7	3.7	265.4	192.0
	5	25.3	−0.3	−3.4	3.4	265.0	188.3
4.0	0	24.0	−0.2	−3.7	3.7	266.9	194.9
	5	25.3	−0.3	−3.5	3.5	265.1	190.5
3.0	0	23.7	−0.2	−3.6	3.6	266.8	197.2
	5	25.1	−0.2	−3.3	3.3	266.5	192.1

Table 24

Effect of pH of enzyme application on wash fastness: *Neutrilan Navy S-BGR* washed at 60 °C

pH	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
6.0	0	15.1	2.6	−14.1	14.3	280.5	393.1
	5	15.4	2.5	−14.7	14.9	279.6	391.4
5.0	0	14.9	2.5	−14.0	14.2	280.1	393.9
	5	15.3	2.4	−14.7	14.9	279.3	391.6
4.0	0	14.6	2.3	−13.8	14.0	279.4	395.0
	5	15.2	2.2	−14.6	14.8	278.6	391.7
3.0	0	14.5	2.3	−13.6	13.8	279.6	395.6
	5	15.2	2.2	−14.5	14.7	278.6	391.8

decreased with increasing pH of application of the enzyme. The corresponding colorimetric data are shown in Tables 10–24. It is evident that when the enzyme was applied at each of the four pH values used (3, 4, 5 and 6), aftertreatment flattened the shade of dyeings and, in the cases of red and yellow dyeings, imparted a yellow-brown colour. These findings can, as proposed earlier [1], be

attributed to the tannic acid component of the aftertreatment having altered the shade of dyeings.

The extent of the staining of adjacent multifibre strip that occurred during repeated wash testing at each of the three wash temperatures is shown in Tables 25–27. As expected, the degree of staining increased with increasing washing temperature and, of the six component fibres, nylon 6,6 was

Table 25

Effect of pH of enzyme application on staining obtained for washing at 40 °C<sup>a</sup>

pH	Number of washes	Wool	Acrylic	Polyester	Nylon 6.6	Cotton	2° Acetate
6	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [4/5] {4/5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
5	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4/5) [4/5] {4/5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
4	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4/5) [4/5] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
3	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4) [4/5] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}

<sup>a</sup> Bold = Neutrilan Red K-2G; no brackets = Nylanthrene Yellow C-3RL; ( ) = Nylanthrene Blue C-GLF; [ ] = Nylanthrene Black C-DPL; { } = Neutrilan Navy S-BGR.

Table 26

Effect of pH of enzyme application on the staining obtained for washing at 50 °C<sup>a</sup>

pH	Number of washes	Wool	Acrylic	Polyester	Nylon 6.6	Cotton	2° Acetate
6	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4/5) [4/5] {4/5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
5	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4/5) [4/5] {4/5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
4	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4/5) [4] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}
3	1	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5* (4) [4] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}
	5	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 5 (4/5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}

<sup>a</sup> Legend as for Table 25.

Table 27

Effect of pH of enzyme application on the staining obtained for washing at 60 °C<sup>a</sup>

pH	Number of washes	Wool	Acrylic	Polyester	Nylon 6.6	Cotton	2° Acetate
6	1	<b>5</b> 4/5* (4) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>4/5</b> 4/5* (4) [4] {3}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}
	5	<b>5</b> 4/5* (4) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}	<b>4/5</b> 4/5* (2/3) [4/5] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}
5	1	<b>4/5</b> 4/5* (3/4) [4/5] {4/5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (5) [4/5] {5}	<b>4</b> 4/5* (3) [3/4] {2/3}	<b>5</b> 5* (5) [4/5] {5}	<b>5</b> 5* (4/5) [5] {5}
	5	<b>5</b> 4/5* (3/4) [5] {5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}	<b>4/5</b> 4* (2) [4/5] {3/4}	<b>5</b> 5* (4/5) [5] {5}	<b>5</b> 4/5* (4) [5] {5}
4	1	<b>4/5</b> 4/5* (3) [4] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [4/5] {5}	<b>3/4</b> 4/5* (2/3) [3] {2}	<b>5</b> 4/5* (4) [4] {5}	<b>5</b> 4* (4) [5] {5}
	5	<b>5</b> 4* (2) [5] {4/5}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4/5) [5] {5}	<b>3/4</b> 3* (1/2) [4] {2/3}	<b>5</b> 5* (4) [5] {5}	<b>5</b> 4/5* (3) [5] {5}
3	1	<b>3/4</b> 4* (2/3) [4] {3/4}	<b>5</b> 5* (5) [5] {4/5}	<b>5</b> 5* (4/5) [4/5] {5}	<b>3</b> 3* (2) [3] {2}	<b>4</b> 4* (3) [4] {4}	<b>5</b> 4* (2) [5] {5}
	5	<b>5</b> 4* (1/2) [5] {4}	<b>5</b> 5* (5) [5] {5}	<b>5</b> 5* (4) [4/5] {5}	<b>3/4</b> 2/3* (1/2) [4] {2/3}	<b>5</b> 5* (2/3) [5] {5}	<b>5</b> 3/4* (1/2) [5] {5}

<sup>a</sup> Legend as for Table 25.

stained most heavily. When the staining results shown in Tables 25 to 27 are compared to those obtained for the untreated dyeings (Table 5), it is clear that the tannic acid/enzyme system was very effective in reducing the extent of staining at each wash temperature. It is evident that the level of staining of the multifibre components was lowest when the enzyme had been applied at pH 6.

Thus, the findings suggest that the lowest extents of shade change and staining were achieved when the enzyme had been applied at pH 6.

### 3.4.1. Effect of temperature

In this section of the work, the enzyme was applied at pH 6 at 60, 70 and 80 °C and the dyeings

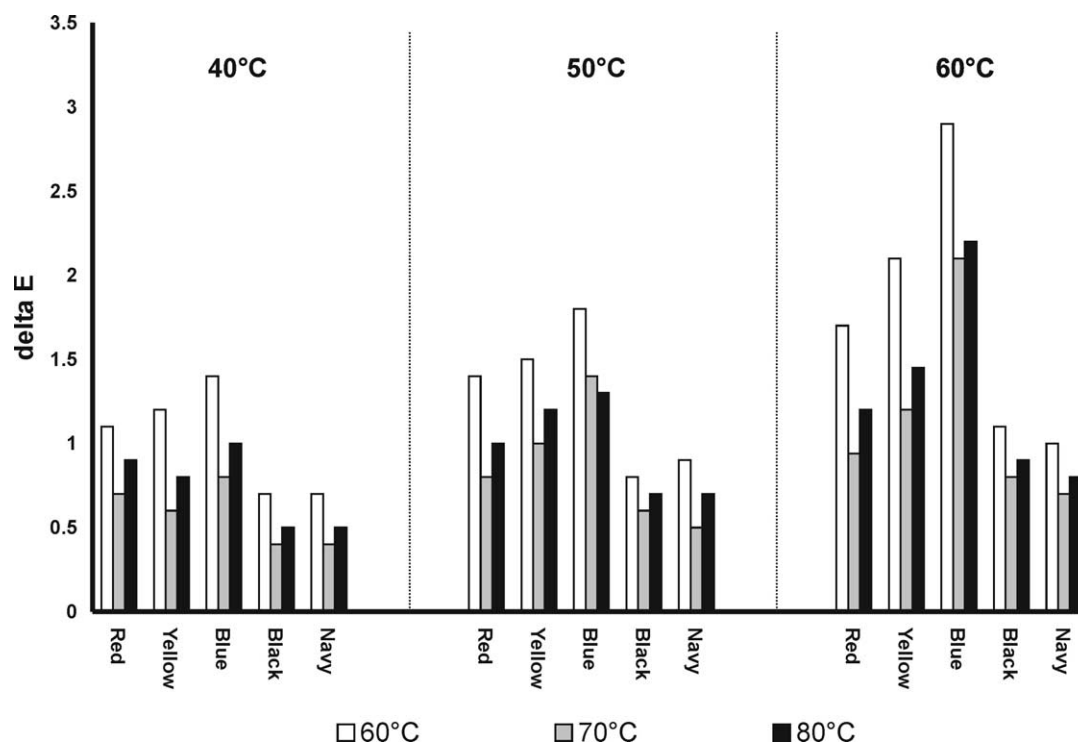


Fig. 7. Effect of temperature of application of enzyme (60, 70 and 80 °C) on the colour difference obtained for tannic acid/enzyme.

Table 28

Effect of temperature of enzyme application on wash fastness: *Neutrilan Red K-2G* washed at 40 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	42.0	44.0	17.3	47.3	21.5	91.3
	5	42.7	43.3	16.9	46.5	21.3	87.8
70	0	42.0	43.9	17.2	47.1	21.4	91.4
	5	42.2	43.3	17.0	46.5	21.4	90.9
80	0	41.7	43.5	16.8	46.6	21.1	92.5
	5	42.0	42.7	16.7	45.9	21.4	91.8

Table 29

Effect of temperature of enzyme application on wash fastness: *Nylanthrene Yellow C-3RL* washed at 40 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	69.0	28.6	74.0	79.3	68.9	68.2
	5	69.8	28.1	73.2	78.4	69.0	67.2
70	0	68.9	28.6	74.6	79.9	68.4	68.9
	5	69.3	28.2	74.4	79.6	69.2	68.5
80	0	68.7	28.4	74.3	79.5	69.1	69.6
	5	69.2	27.9	73.9	79.0	69.3	68.9

Table 30

Effect of temperature of enzyme application on wash fastness: *Nylanthrene Blue C-GLF* washed at 40 °C

Temperature/°C	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
60	0	42.1	−2.0	−38.0	38.1	267.0	62.8
	5	42.9	−2.5	−37.0	37.1	266.1	61.3
70	0	41.9	−2.0	−38.1	38.2	267.0	62.9
	5	42.4	−2.3	−37.5	37.6	266.5	62.0
80	0	41.7	−1.8	−38.4	38.4	267.3	63.4
	5	42.3	−2.1	−37.7	37.8	266.8	62.2

Table 31

Effect of temperature of enzyme application on wash fastness: *Nylanthrene Black C-DPL* washed at 40 °C

Temperature/°C	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
60	0	24.7	−0.3	−3.8	3.8	265.5	189.4
	5	25.4	−0.3	−3.9	3.9	265.6	187.2
70	0	24.5	−0.3	−3.8	3.8	265.5	191.2
	5	24.9	−0.3	−3.7	3.7	265.4	190.6
80	0	24.3	−0.3	−3.6	3.6	265.2	192.3
	5	24.8	−0.3	−3.5	3.5	265.1	191.5

Table 32

Effect of temperature of enzyme application on wash fastness: *Neutrilan Navy S-BGR* washed at 40 °C

Temperature/°C	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
60	0	15.2	2.6	−14.3	14.5	280.3	390.2
	5	15.5	2.6	−14.9	15.1	279.9	388.8
70	0	15.1	2.6	−14.1	14.3	280.5	393.1
	5	15.2	2.6	−14.5	14.7	280.2	392.6
80	0	14.8	2.5	−13.9	14.1	280.2	394.9
	5	15.0	2.5	−14.4	14.6	279.8	394.0

Table 33

Effect of temperature of enzyme application on wash fastness: *Neutrilan Red K-2G* washed at 50 °C

Temperature/°C	Number of washes	$L^*$	$a^*$	$b^*$	$C^*$	$h^\circ$	$f(k)$
60	0	42.0	44.0	17.3	47.3	21.5	91.3
	5	43.0	43.2	16.8	46.4	21.2	86.9
70	0	42.0	43.9	17.2	47.1	21.4	91.4
	5	42.3	43.2	17.0	46.4	21.5	90.7
80	0	41.7	43.5	16.8	46.6	21.1	92.5
	5	42.2	42.6	16.7	45.8	21.4	91.2

Table 34

Effect of temperature of enzyme application on wash fastness: *Nylanthrene Yellow C-3RL* washed at 50 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	69.0	28.6	74.0	79.3	68.9	68.2
	5	70.0	28.0	73.0	78.2	69.0	66.8
70	0	68.9	28.6	74.6	79.9	68.4	68.9
	5	69.5	28.1	74.1	79.3	69.2	68.2
80	0	68.7	28.4	74.3	79.5	69.1	69.6
	5	69.4	27.7	73.7	78.7	69.4	68.3

Table 35

Effect of temperature of enzyme application on wash fastness: *Nylanthrene Blue C-GLF* washed at 50 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	42.1	−2.0	−38.0	38.1	267.0	62.8
	5	43.2	−2.7	−36.8	36.9	265.8	60.5
70	0	41.9	−2.0	−38.1	38.2	267.0	62.9
	5	42.6	−2.6	−37.1	37.2	266.0	61.5
80	0	41.7	−1.8	−38.4	38.4	267.3	63.4
	5	42.4	−2.4	−37.5	37.6	266.3	61.6

Table 36

Effect of temperature of enzyme application on wash fastness: *Nylanthrene Black C-DPL* washed at 50 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	24.7	−0.3	−3.8	3.8	265.5	189.4
	5	25.5	−0.4	−3.9	3.9	264.1	186.9
70	0	24.5	−0.3	−3.8	3.8	265.5	191.2
	5	25.1	−0.3	−3.7	3.7	265.4	189.7
80	0	24.3	−0.3	−3.6	3.6	265.2	192.3
	5	25.0	−0.3	−3.5	3.5	265.1	190.3

Table 37

Effect of temperature of enzyme application on wash fastness: *Neutrilan Navy S-BGR* washed at 50 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	15.2	2.6	−14.3	14.5	280.3	390.2
	5	15.7	2.5	−15.0	15.2	279.5	387.7
70	0	15.1	2.6	−14.1	14.3	280.5	393.1
	5	15.3	2.6	−14.6	14.8	280.1	392.0
80	0	14.8	2.5	−13.9	14.1	280.2	394.9
	5	15.1	2.4	−14.5	14.7	279.4	392.9

Table 38

Effect of temperature of aftertreatment on the wet fastness: *Neutrilan Red K-2G* washed at 60 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	42.0	44.0	17.3	47.3	21.5	91.3
	5	43.3	43.0	16.8	46.2	21.3	85.7
70	0	42.0	43.9	17.2	47.1	21.4	91.4
	5	42.4	43.1	16.9	46.3	21.4	90.3
80	0	41.7	43.5	16.8	46.6	21.1	92.5
	5	42.4	42.5	16.6	45.6	21.3	90.6

Table 39

Effect of temperature of aftertreatment on the wet fastness: *Nylanthrene Yellow C-3RL* washed at 60 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	69.0	28.6	74.0	79.3	68.9	68.2
	5	70.1	27.8	72.4	77.6	69.1	65.4
70	0	68.9	28.6	74.6	79.9	68.4	68.9
	5	69.8	28.1	73.9	79.1	69.2	67.8
80	0	68.7	28.4	74.3	79.5	69.1	69.6
	5	69.6	27.7	73.4	78.5	69.3	67.9

Table 40

Effect of temperature of aftertreatment on the wet fastness: *Nylanthrene Blue C-GLF* washed at 60 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	42.1	−0.2	−38.0	38.1	267.0	62.8
	5	43.8	−3.2	−36.0	36.1	264.9	59.2
70	0	41.9	−2.0	−38.1	38.2	267.0	62.9
	5	43.0	−2.9	−36.5	36.6	265.5	60.0
80	0	41.7	−1.8	−38.4	38.4	267.3	63.4
	5	42.8	−2.8	−36.8	36.9	265.7	59.8

Table 41

Effect of temperature of aftertreatment on the wet fastness: *Nylanthrene Black C-DPL* washed at 60 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	24.7	−0.3	−3.8	3.8	265.5	189.4
	5	25.8	−0.4	−3.9	3.9	264.1	185.7
70	0	24.5	−0.3	−3.8	3.8	265.5	191.2
	5	25.3	−0.3	−3.6	3.6	265.2	188.4
80	0	24.3	−0.3	−3.6	3.6	265.2	192.3
	5	25.2	−0.3	−3.4	3.4	265.0	189.1

Table 42

Effect of temperature of aftertreatment on the wet fastness: *Nuetrilan Navy S-BGR* washed at 60 °C

Temperature/°C	Number of washes	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> <sup>o</sup>	<i>f(k)</i>
60	0	15.2	2.6	−14.3	14.5	280.3	390.2
	5	15.8	2.5	−15.1	15.3	279.4	386.9
70	0	15.1	2.6	−14.1	14.3	280.5	393.1
	5	15.4	2.5	−14.7	14.9	279.6	391.4
80	0	14.8	2.5	−13.9	14.1	280.2	394.9
	5	15.2	2.4	−14.6	14.8	279.3	391.8

Table 43

Effect of temperature of aftertreatment on the staining obtained for washing at 40 °C<sup>a</sup>

Temperature/°C	Number of washes	Wool	Acrylic	Polyester	Nylon 6.6	Cotton	2° Acetate
60	1	5 5* (4) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	3/4 3/4* (3) [3/4] {3/4}	5 5* (4/5) [5] {5}	5 4/5* (4) [5] {5}
	5	5 5* (4) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	3/4 3/4* (3) [3/4] {3/4}	5 5* (5) [5] {5}	5 5* (4/5) [5] {5}
70	1	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) 4/5 [4/5]	5 5* (5) [5] {5}	5 5* (5) [5] {5}
	5	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}
80	1	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (4/5) [4/5] {4/5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}
	5	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}

<sup>a</sup> Legend as for Table 25.

Table 44

Effect of temperature of aftertreatment on the staining obtained for washing at 50 °C<sup>a</sup>

Temperature/°C	Number of washes	Wool	Acrylic	Polyester	Nylon 6.6	Cotton	2° Acetate
60	1	4/5 4* (3) [4] {4}	5 5* (5) [4/5] {5}	5 5* (5) [5] {5}	2/3 2/3* (2) [2/3] {2/3}	5 5* (4) [5] {5}	5 4* (4) [5] {5}
	5	5 4/5* (3/4) [4/5] {4}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	2/3 2/3* (2) [3] {2/3}	5 5* (4/5) [5] {5}	5 4/5* (4) [5] {5}
70	1	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	4/5 5* (4/5) [4/5] {4/5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}
	5	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}
80	1	5 5* (4/5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	4/5 4/5* (4/5) [4/5] {4/5}	5 5* (5) [5] {5}	5 4/5* (4/5) [5] {5}
	5	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	5 5* (4/5) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}

<sup>a</sup> Legend as for Table 25.

Table 45

Effect of temperature of aftertreatment on the staining obtained for washing at 60 °C<sup>a</sup>

Temperature/°C	Number of washes	Wool	Acrylic	Polyester	Nylon 6.6	Cotton	2° Acetate
60 °C	1	4 3* (2/3) [3/4] {3/4}	5 5* (5) [4] {5}	5 4* (4) [5] {5}	2/3 2* (1/2) [2/3] {2}	5 4/5* (3) [5] {5}	5 3/4* (3) [5] {5}
	5	4/5 3* (2/3) [4] {3/4}	5 5* (5) [4/5] {5}	5 4/5* (4) [5] {5}	2/3 2/3* (2) [2/3] {2/3}	5 5* (3/4) [5] {5}	5 5* (3) [5] {5}
70 °C	1	5 4/5* (4) [5] {5}	5 5* (5) [5] {5}	5 5* (5) [5] {5}	4/5 4/5* (4) [4] {3}	5 5* (5) [5] {5}	5 5* (4/5) [5] {5}
	5	5 4/5* (4) [5] {5}	5 5* (5) [5] {5}	5 5* (4/5) [5] {5}	4/5 4/5* (2/3) [4/5] {4}	5 5* (5) [5] {5}	5 5* (4/5) [5] {5}
80 °C	1	4/5 4* (3/4) [4] {4}	5 5* (5) [5] {5}	5 4/5* (4) [5] {5}	4/5 4* (3/4) [4/5] {4}	5 5* (4/5) [5] {5}	5 4/5* (4/5) [5] {5}
	5	5 4/5* (4) [4/5] {4}	5 5* (5) [5] {5}	5 5* (4) [5] {5}	4/5 4/5 (2/3) [4] {4}	5 5* (4/5) [5] {5}	5 5* (4/5) [5] {5}

<sup>a</sup> Legend as for Table 25.

Table 46  
Light fastness

	pH	Red K-2G	Yellow C-3RL	Blue C-GLF	Black C-DPL	Navy S-BGR
Untreated	—	5/6	7	4	5/6	6
Full backtan	3.0	5/6	7	4/5	5/6	6
Tannic acid/enzyme	6.0	5/6	7	4/5	5/6	6
	5.0	5/6	7	4	5/6	6
	4.0	5/6	7	4	5/6	6
	3.0	5/6	7	4/5	5/6	6

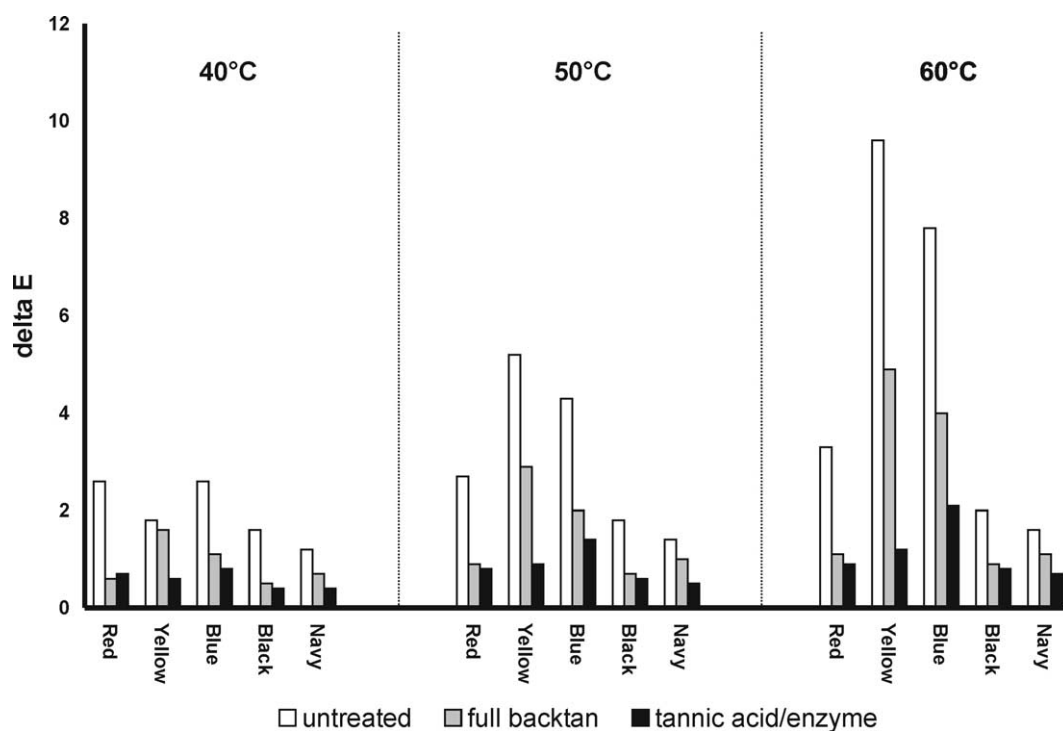


Fig. 8. Comparison of colour difference obtained for untreated dyeings as well as dyeings aftertreated with the full backtan and tannic acid/enzyme at pH 6 and 70 °C.

were subjected to five, repeated wash tests at 40, 50 and 60 °C.

Fig. 7 shows the extent to which the tannic acid/enzyme system reduced the shade change of the dyeings in terms of the colour difference obtained after repeated wash testing at each of the three washing temperatures employed. As expected, the extent of the change in shade obtained for each dye increased with increasing wash temperature. For each of the five dyes used, the extent of the shade change obtained was generally lowest when

the enzyme had been applied at 70 °C. The corresponding colorimetric results (Tables 28–42) reveal that aftertreatment flattened the shade of dyeings and, in the cases of red and yellow dyeings, imparted a yellow colour. These findings can, as proposed earlier [1], be attributed to the tannic acid component of the aftertreatment having altered the shade of dyeings.

In the context of the staining of adjacent multi-fibre strip that occurred during repeated wash testing, Tables 43–45 show that the level of staining of

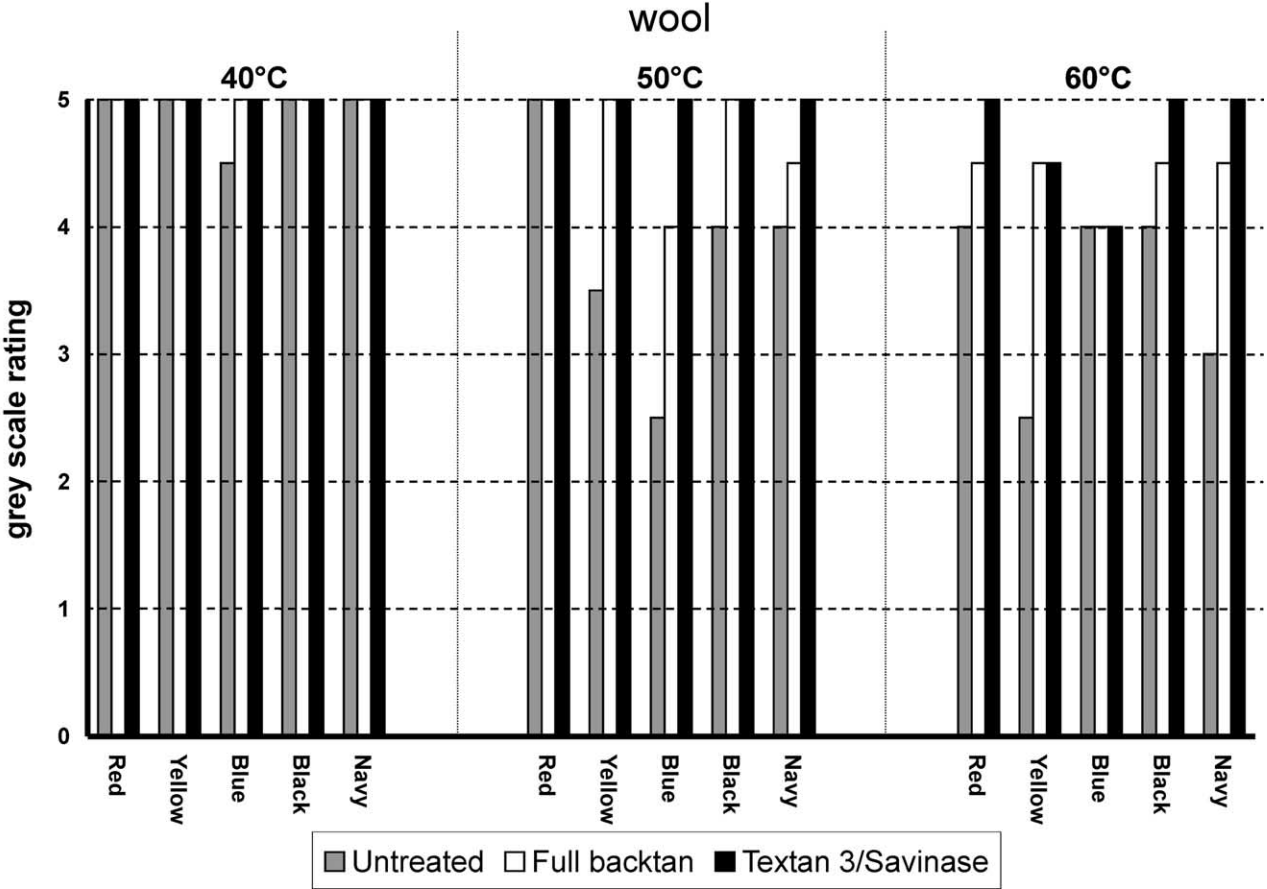


Fig. 9. Comparison of the staining obtained for untreated dyeings, and dyeings aftertreated with the full backtan and tannic acid/enzyme (pH 6; 70 °C).

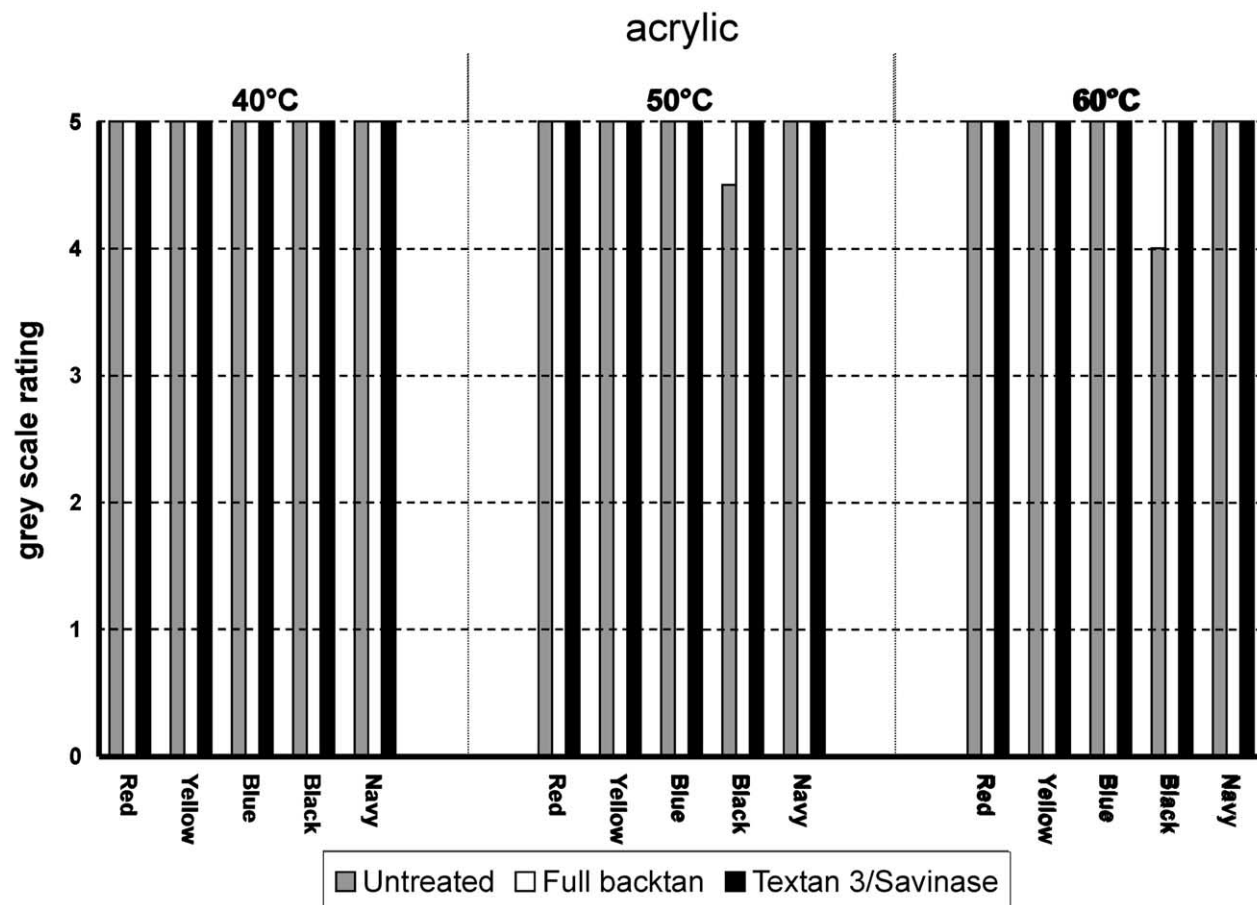


Fig. 10. Comparison of the staining obtained for untreated dyeings, and dyeings aftertreated with the full backtan and tannic acid/enzyme (pH 6; 70 °C).

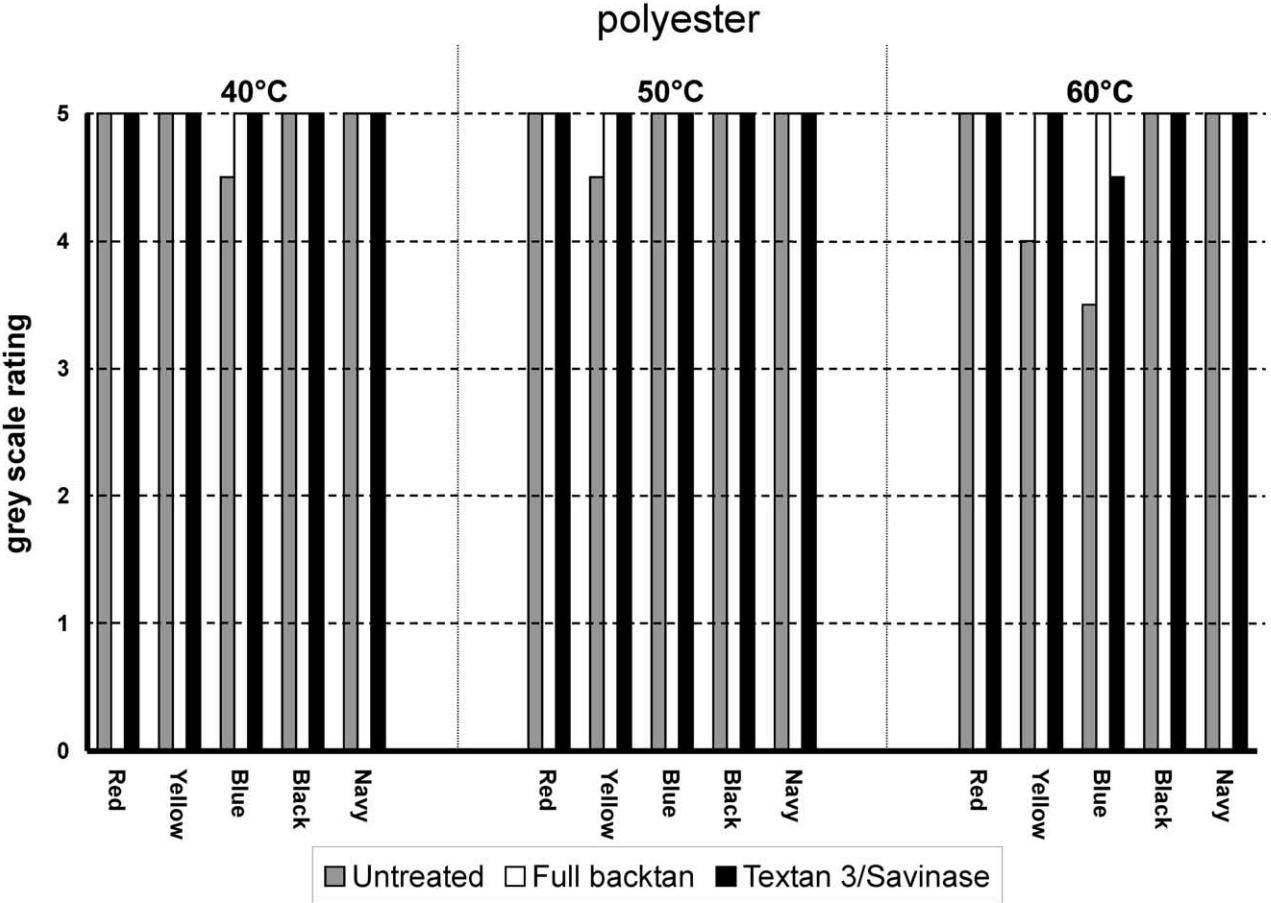


Fig. 11. Comparison of the staining obtained for untreated dyeings, and dyeings aftertreated with the full backtan and tannic acid/enzyme (pH 6; 70 °C).

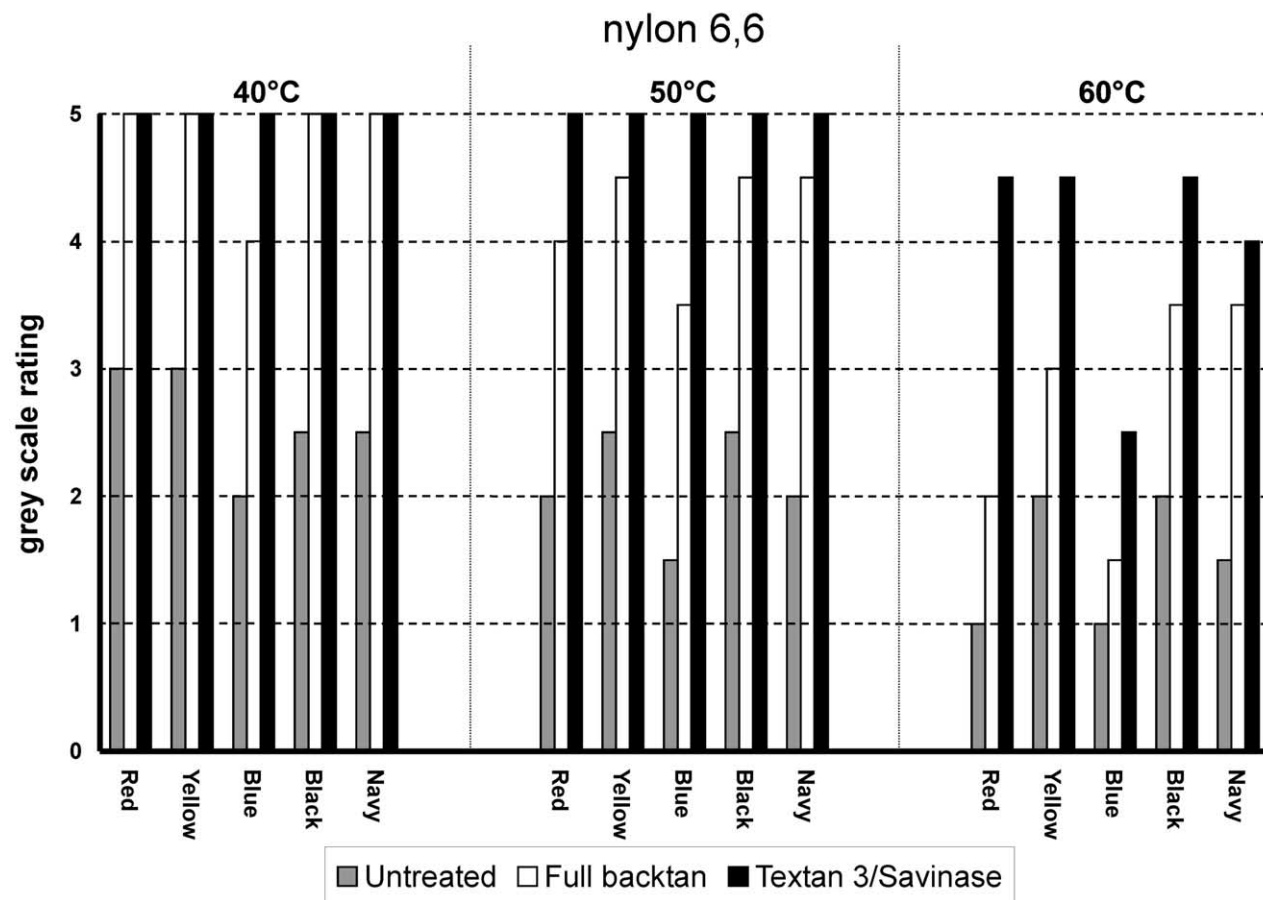


Fig. 12. Comparison of the staining obtained for untreated dyeings, and dyeings aftertreated with the full backtan and tannic acid/enzyme (pH 6; 70 °C).

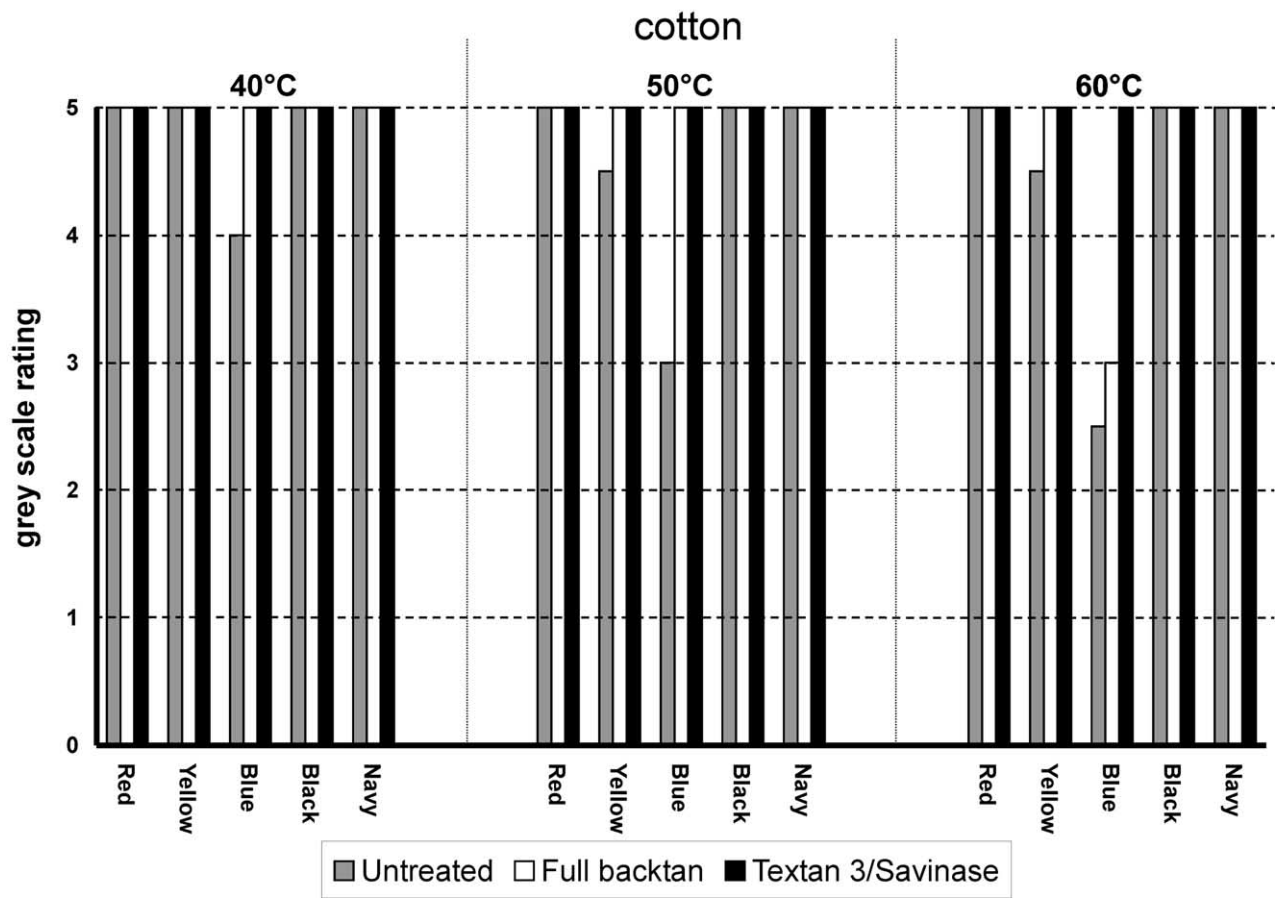


Fig. 13. Comparison of the staining obtained for untreated dyeings, and dyeings aftertreated with the full backtan and tannic acid/enzyme (pH 6; 70 °C).

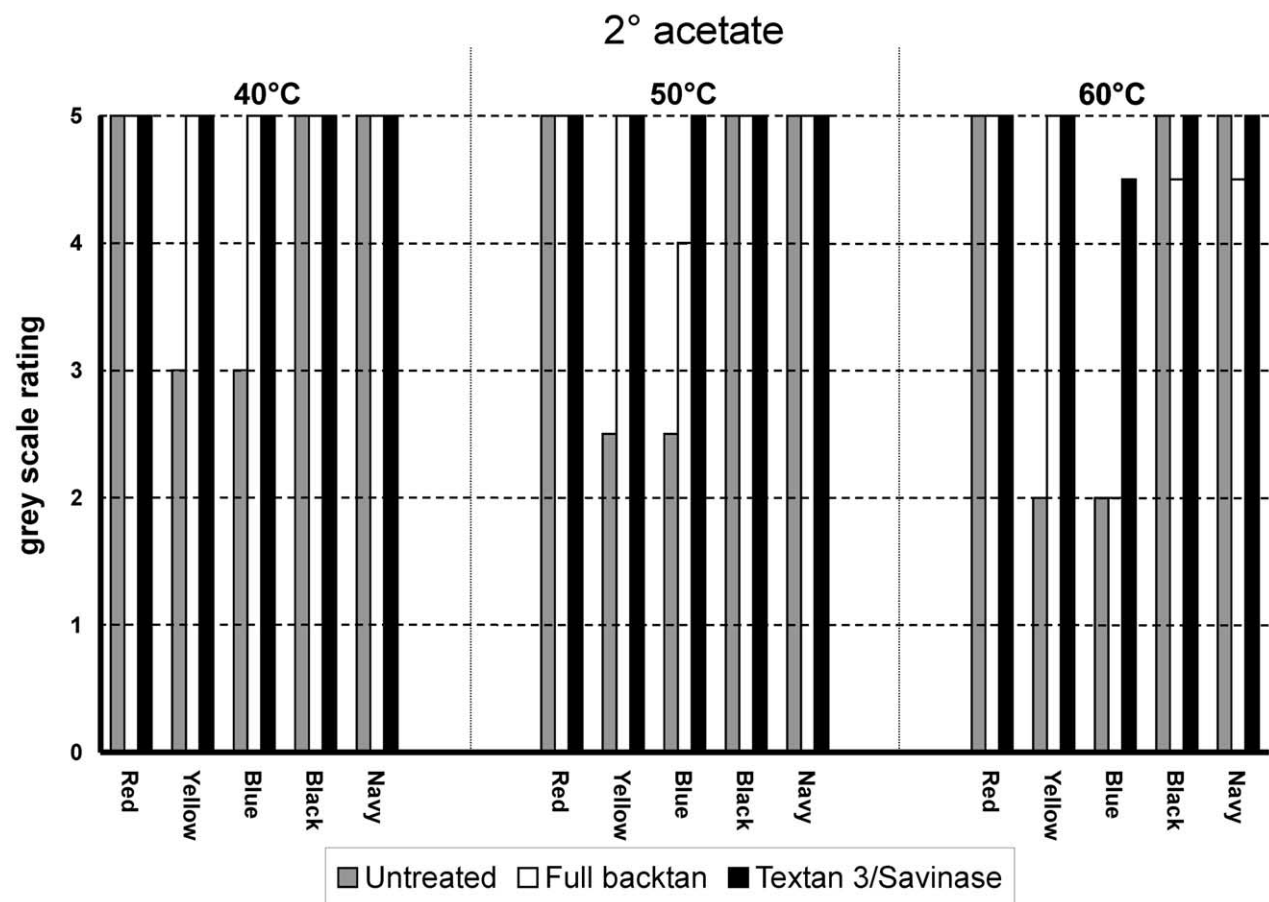


Fig. 14. Comparison of the staining obtained for untreated dyeings, and dyeings aftertreated with the full backtan and tannic acid/enzyme (pH 6; 70 °C).

the multifibre strip components was higher when the enzyme had been applied at 60 °C than at the other two application temperatures and that applying the enzyme at 70 °C and 80 °C produced similar levels of staining. When the staining results shown in [Tables 43 to 45](#) are compared to those obtained for the untreated dyeings ([Table 5](#)), it is apparent that the tannic acid/enzyme system markedly reduced the extent of staining of the adjacent components, when compared with the results obtained for the non-aftertreated dyeings.

Thus, in view of the findings that the lowest extent of shade change was achieved for an application temperature of 70 °C and that virtually identical levels of staining of multifibre strip were achieved when the enzyme had been applied at 70 °C and 80 °C, it seems reasonable to propose that the optimum temperature for application of the enzyme is 70 °C.

### 3.5. *Light fastness*

[Table 46](#) shows that aftertreatment of the dyeings, with either the full backtan or the tannic acid/enzyme system, neither improved nor reduced the light fastness of the dyeings.

### 3.6. *Handle*

Each of the aftertreatments used were judged to have impaired the handle of the dyeings to the same extent.

### 3.7. *Comparison with full backtan*

As mentioned, the purpose of this work was to develop a metal-free, tannic acid-based aftertreatment as an alternative to the traditional full backtan. In this context, it follows that a comparison needs to be made between the results obtained using the tannic acid/enzyme system when it had been applied using the conditions which gave optimum fastness improvement (i.e. pH 6 and 70 °C) and the results obtained using the full backtan, in terms of the extents of shade change and staining of adjacent materials that occurred during repeated washing.

[Fig. 8](#) shows the colour difference obtained as a result of five repeated wash tests at the three washing temperatures used, for the untreated dyeings and dyeings which had been aftertreated with the full backtan as well as the tannic acid/enzyme at pH 6 and 70 °C. The effectiveness of both the full backtan and the tannic acid/enzyme aftertreatments is evident. Also, it is clear that the tannic acid/enzyme aftertreatment was more effective than its full backtan counterpart in reducing the extent of the shade change. While the nature and magnitude of any shade change that a dyed sample undergoes during washing contribute to the assessment of wash fastness, the extent of any staining of adjacent materials by vagrant dye during washing is an equally important element of wash fastness. In this context, [Figs. 9–14](#) show the extent of staining that was obtained for the six components of the multifibre strip. While both the full backtan and the tannic acid/enzyme pH 6 aftertreatments were effective in reducing the level of staining, the greater effectiveness of the tannic acid/enzyme pH 6 aftertreatment is clearly evident, especially in the case of wash testing at 60 °C.

## 4. **Conclusions**

Optimal conditions for applying the enzyme, in the two-stage, single bath aftertreatment method were found to be 70 °C at pH 6. When applied under these particular conditions, the effectiveness of the tannic acid/enzyme aftertreatment in improving the fastness to repeated washing of five commercial acid dyes on nylon 6,6 was superior to that of a traditional full backtan aftertreatment. The metal-free, tannic acid/enzyme aftertreatment offers a potentially more environmentally acceptable alternative to the antimony-based and tin-based systems.

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