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The aftertreatment of acid dyes on nylon 6,6 fibres: Part 2. Non-metallised acid dyes

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Abstract

Nylon 6,6 knitted fabric was dyed using three, levelling dyes and three milling dyes and the dyeings were aftertreated with a commercial syntan as well as a newly developed full backtan. When the dyed samples were subjected to five, consecutive ISO 105:C06/C2 wash tests, it was found that while the syntan was effective in improving wash fastness, the new backtanning process imparted greatest wash fastness improvement. © 2001 Elsevier Science Ltd. All rights reserved

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1. Introduction

The wash fastness of acid dyes on nylon 6,6 leaves much to be desired and an aftertreatment with either a synthetic or a natural tanning is commonly used to secure highest levels of wash fastness, especially in moderate to deep depths of shade [1]. Aftertreatment with a synthetic tanning agent (syntan) is generally considered to be less effective than aftertreatment with a natural tanning agent (the full backtan) in improving the wet fastness of acid dyes on nylon [1]. Nonetheless, the effectiveness of a commercial syntan in improving

The previous part of this paper [6] concerned the extent to which the wash fastness, of eight commercial 1:2 pre-metallised acid dyes on nylon 6,6 fabric, to repeated washing was improved by an aftertreatment with a commercial syntan, a

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the wash fastness of acid dyes on nylon 6,6 can be enhanced by the subsequent application of a selected, polymeric cationic agent to the syntanned fabric [2–5]. The use of the full backtan, which traditionally comprises treatment with tannic acid and the subsequent application of potassium antimony tartrate (tartar emetic) has, in recent years been superceded by that of synthetic tanning agents (syntans) owing to several disadvantages inherent in the two-stage process [1]. This paper comprises an investigation of a newly developed full backtan aftertreatment process for dyed nylon 6,6 that does not use tartar emetic.

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commercial syntan/cation system and a newly developed, one-bath, two-stage, full backtan aftertreatment that comprised tannic acid and a nonpotassium antimony tartrate complexing agent, Gallofix. It was found that while all three aftertreatments imparted improved wash fastness, the new full backtan system conferred greatest wash fastness improvement. In addition, backtanning imparted little shade change to the dyeings and the extent of this shade change was very similar to that given by the other two aftertreatments. The handle of the backtanned fabrics, while slightly harsher than that of the syntanned fabrics, was similar to that of dyeings which had been aftertreated with the syntan/cation system. The light fastness of the dyeings was little changed as a result of the backtanning process. In addition, the traditional full backtan aftertreatment that uses tannic acid and potassium antimony tartrate, is a commonly, two-stage, two-bath process in which the tannic acid is firstly applied to the dyed nylon fibre and the tartar emetic is subsequently applied from a fresh bath. Typically, these stages each take some 20–30 minutes at a temperature of between 70 and 90°C [1]. As the particular backtan aftertreatment method used in this work was a twostage, single-bath process lasting just 20 minutes at 70°C, the application profile of the new backtan process closely resembled that of a typical commercial syntan and it was therefore considered to offer potential time- and cost-savings over the traditional full backtan aftertreatment [6].

This part of the paper examines the effectiveness of the new full backtan aftertreatment, when compared to a commercial syntan, in improving the fastness to repeated washing of non-metallised acid dyes.

2. Experimental

2.1. Materials

The scoured, knitted, nylon 6,6 (78F68) fabric described earlier [6] was used. Commercial samples of the non-metallised acid dyes listed in Table 1 were generously supplied by Crompton & Knowles. A commercial sample of the syntan *Mesitol NBS/E* was kindly supplied by Bayer and

Table 1 Dyes used

Nylanthrene Red B-2BSA C.I. Acid Red 266 Levellin Nylanthrene Yellow B-4RK C.I. Acid Yellow 219 Levellin Nylanthrene Blue C-GLF C.I. Acid Blue 281 Milling	Commercial name	C.I. generic name	Dye type
Nylanthrene Red C-3BR C.I. Acid Red 151 Milling Nylanthrene Yellow C-3RL C.I. Acid Orange 67 Milling	Nylanthrene Red B-2BSA Nylanthrene Yellow B-4RK Nylanthrene Blue C-GLF Nylanthrene Red C-3BR	C.I. Acid Red 266 C.I. Acid Yellow 219 C.I. Acid Blue 281 C.I. Acid Red 151	Milling

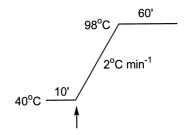
commercial samples of the tanning agent *Floctan 1* and of the complexing agent *Gallofix* were generously provided by Omnichem-Ajinmoto.

2.2. Dyeing

Two percent omf dyeings were obtained using the equipment described earlier [6]; the dyeing method used is shown in Fig. 1. The pH was adjusted to 6, using a McIlvaine buffer [6], for the milling acid dyes while a pH of 4 was provided for the acid levelling dyes using 2 g l⁻¹ ammonium sulfate. At the end of dyeing, the dyed sample was removed, rinsed thoroughly in tap water and allowed to dry in the open air.

2.3. Syntan aftertreatment

Dyeings were aftertreated with the syntan *Mesitol NBS/E* (2% omf and also 4% omf) at pH 4 and 20:1 liquor ratio, using the equipment described earlier [6]; the method is shown in Fig. 2.



2% omf dye levelling dyes: pH 4 with 2 gl⁻¹ (NH₄)₂SO₄ milling dyes: pH 6 with McIlvaine buffer

Fig. 1. Dyeing methods.

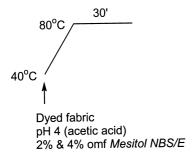


Fig. 2. Syntan aftertreatment.

2.4. Backtanning

The aftertreatment method is given in Fig. 3; 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 [6].

2.6. Wash fastness

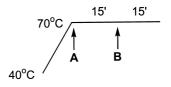
The samples were subjected to five, consecutive ISO 105:C06/C2 wash tests as described earlier [6]. The reduction in depth of shade that occurred as a result of washing was calculated using Eq. (1) where fk_u and fk_w are the weighted K/S functions of the unwashed and washed samples, respectively.

Reduction in colour strength

$$= \left(\frac{fk_{\rm u} - fk_{\rm w}}{fk_{\rm u}}\right) \times 100\tag{1}$$

3. Results and discussion

Owing to the variance of non-metallised acid dyes in terms of their application and fastness properties on nylon fibres, it is common for dye makers to classify their dye ranges. The maker of the dyes used in this work sub-divide their non-metallised acid dyes in to three groups, namely levelling, milling and super-milling, based on the



A dyed fabric pH 3/3.5 (acetic acid) 2% omf Floctan 1

B 4% omf Gallofix

Fig. 3. Full backtan aftertreatment.

application behaviour and fastness characteristics of the dyes on wool. Generally, the substantivity and wet fastness of the three types of dye increases in the order: *levelling < milling < super-milling* while the migration power of the dyes and the pH of application decrease in the order: *levelling < milling < super-milling*. In this work, it was decided to investigate the effectiveness of the newly developed full backtan after treatment in improving the wet fastness of levelling and milling types of non-metallised acid dye.

Fig. 4 shows the reduction in colour strength that occurred for dyeings of C.I Acid Blue 62, as a result of the five, consecutive ISOC06/C2 wash tests. It is apparent that the reduction in colour strength achieved for the non-aftertreated dyeing increased with increasing number of washes thus

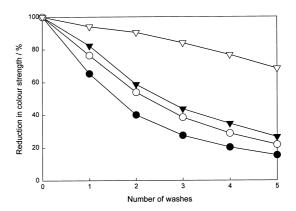


Fig. 4. Effect of repeated wash testing on colour strength of dyeings: C.I. Acid Blue 62. \bullet Nil aftertreatment; \bigcirc 2% omf syntan; \blacktriangledown 4% omf syntan; \bigtriangledown full backtan.

showing that dye loss occurred in a progressive manner. Clearly, aftertreatment with 2% omf syntan reduced the extent of colour loss that occurred during repeated washing; the effectiveness of the syntan in reducing colour loss was enhanced when 4% omf syntan was used. Fig. 4 also shows that aftertreatment with the newly developed full backtan resulted in much reduced colour loss. Figs. 5 and 6 show the reduction in colour strength that occurred as a result of the repeated washing of dyeings of the two remaining levelling dyes, C.I Acid Red 266 and C.I Acid Yellow 219, respectively. The results for the two dyes were similar to those obtained for C.I. Acid

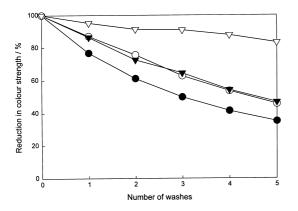


Fig. 5. Effect of repeated wash testing on colour strength of dyeings: C.I. Acid Red 266. \bullet Nil aftertreatment; \bigcirc 2% omf syntan; \blacktriangledown 4% omf syntan; \triangledown full backtan.

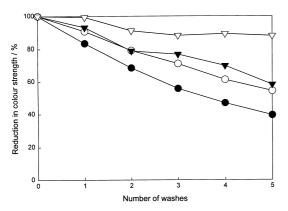


Fig. 6. Effect of repeated wash testing on colour strength of dyeings: C.I. Acid Yellow 219. \bullet Nil aftertreatment; \bigcirc 2% omf syntan; ∇ 4% omf syntan; ∇ full backtan.

Blue 62 in that while the syntan aftertreatment reduced the extent of dye loss, superior reduction in dye loss was imparted by the full backtan aftertreatment. However, inspection of Fig. 5 reveals that in the case of C.I Acid Red 266, there was little difference between the reduction in colour loss imparted by aftertreatment with 2% omf and 4% omf syntan.

The corresponding assessments of the extent of staining of the six fibres in the adjacent SDC multifibre strip fabric achieved for the dyeings after repeated washing are displayed in Table 2. It is evident that aftertreatment with 4% omf syntan was more effective than 2% omf syntan in lowering the extent of staining, while aftertreatment with the newly developed full backtan resulted in lowest staining of the adjacent materials. Colour loss in laundry translates to a change in the shade of a dyeing. Table 3 shows the colour difference (ΔE) between the unwashed dyeings and those which had been washed both once and five times. Unsurprisingly, significant colour loss was associated with greater shade changes. The effectiveness of the syntan, especially when applied at a level of 4% omf, in reducing shade change is obvious; however, the reduction in shade change

Table 2 Staining assessment for aftertreated levelling dyes (2% omf) on nylon 6.6^a

Aftertreatment	SA	С	N	P	A	W
C.I. Acid Blue 62						
Nil	2/3	4/5	1	5	5	1/2
2% omf Syntan	3/4	4/5	2-1/2	4	5	2
4% omf Syntan	3/4	5	2	5	5	2
Full backtan	4/5	5	3-2/3	5	5	3/4-3
C.I. Acid Red 266						
Nil	2	2	1	4	5	1/2 - 1
2% omf Syntan	2/3	2/3	1/2	4/5	5	2
4% omf Syntan	2/3	2/3	2	4/5	5	2
Full backtan	4/5	4/5	3-2/3	5	5	3/4-3
C.I. Acid Yellow 2	219					
Nil	2	2	1	5	5	1/2
2% omf Syntan	3	3-2/3	1/2	5	5	2
4% omf Syntan	3	3-2/3	2-1/2	5	5	2/3-2
Full backtan	4/5	4-3/4	3	5	5	4/5

^a SA, secondary acetate; C, cotton; N, nylon 6.6; P, polyester; A, acrylic; W, wool.

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	ΔE 1 Wash	ΔE 1 Wash			ΔE 5 Washes		
Aftertreatment	C.I. Acid Blue 62	C.I. Acid Red 266	C.I. Acid Yellow 219	C.I. Acid Blue 62	C.I. Acid Red 266	C.I. Acid Yellow 219	
Nil	6.80	3.63	4.68	28.53	13.54	17.45	
2% omf Syntan	4.55	1.90	2.53	22.79	10.24	12.02	
4% omf Syntan	3.61	1.81	1.77	19.85	9.93	10.80	
Full backtan	1.96	0.54	0.60	8.02	2.77	4.28	

Table 3 Colour change (ΔE CIE Lab) after one and five washes for levelling dyes

imparted by the full backtan aftertreatment is also evident.

With regards the three milling, non-metallised acid dyes, Figs. 7–9 show the reduction in colour strength that occurred for dyeings of C.I Acid Blue 281, C.I. Acid Red 151 and C.I. Acid Orange 67, respectively, as a result of the five, consecutive ISOC06/C2 wash tests. The results obtained are similar to those secured for the three levelling, non-metallised acid dyes. The extent of the reduction in colour strength achieved for the non-aftertreated dyeings, which increased with increasing number of washes, was reduced by aftertreatment. Generally, aftertreatment with 4% omf syntan was more effective in reducing colour loss than was 2% omf syntan; however, aftertreatment with the full backtan resulted in lowest colour loss for each of the three dyes used. Table 4 shows the staining of the six adjacent fibres in the SDC multifibre strip fabric for the three milling,

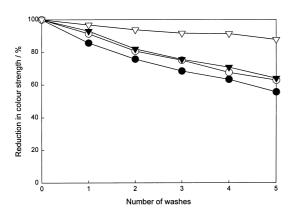


Fig. 7. Effect of repeated wash testing on colour strength of dyeings: C.I. Acid Blue 281. \bullet Nil aftertreatment; \bigcirc 2% omf syntan; \blacktriangledown 4% omf syntan; \triangledown full backtan.

non-metallised acid dyes used. Aftertreatment with 4% omf syntan was more effective than 2% omf syntan in reducing the extent of staining,

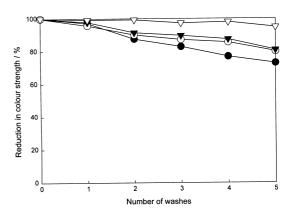


Fig. 8. Effect of repeated wash testing on colour strength of dyeings: C.I. Acid Red 151. \bullet Nil aftertreatment; \bigcirc 2% omf syntan; \blacktriangledown 4% omf syntan; \triangledown full backtan.

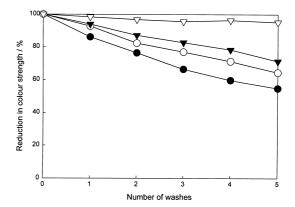


Fig. 9. Effect of repeated wash testing on colour strength of dyeings: C.I. Acid Orange 67. \bullet Nil aftertreatment; \bigcirc 2% omf syntan; \blacktriangledown 4% omf syntan; \triangledown full backtan.

Table 4 Staining assessment for aftertreated milling dyes (2% omf) on nylon 6.6

Aftertreatment	SA	С	N	P	A	W
C.I. Acid Blue 281						•
Nil	2/3	4	1/2-1	4	5	2
2% omf Syntan	3/4	4	2/3–2	4/5	5	2/3
4% omf Syntan	3/4	4	3-2/3	4/5	5	3-2/3
Full backtan	4/5	5	3	5	5	4/5
C.I. Acid Red 151						
Nil	2	2	1	3	5	1/2
2% omf Syntan	2/3	2/3-2	2-1/2	4	5	2
4% omf Syntan	2/3	2/3-2	2-1/2	4	5	2
Full backtan	4	3/4–3	3	4/5	5	3/4
C.I. Acid Orange 67						
Nil	2/3	4	1/2-1	4	5	2
2% omf Syntan	3/4	4/5	2/3	4/5	5	3-2/3
4% omf Syntan	3/4	5	3–2/3	4/5	5	3/4–3
Full backtan	5	5	5	5	5	5

Table 5 Colour change (ΔE CIE Lab) after one and five washes for milling dyes

	ΔE 1 Wash	ΔE 1 Wash			ΔE 5 Washes			
Aftertreatment	C.I. Acid Blue 281	C.I. Acid Red 151	C.I. Acid Orange 67	C.I. Acid Blue 281	C.I. Acid Red 151	C.I. Acid Orange 67		
Nil	2.16	1.70	2.07	7.70	5.89	8.35		
2% omf Syntan	1.57	1.10	0.85	5.40	3.81	6.93		
4% omf Syntan	1.58	1.24	0.62	6.01	3.60	4.82		
Full backtan	1.42	0.26	0.29	2.96	0.82	2.04		

while aftertreatment with the full backtan resulted in lowest staining of the adjacent multifibre strip materials. In terms of the colour difference between the unwashed dyeings and those which had been washed both once and five times, Table 5 demonstrates the effectiveness of the syntan in reducing shade change; however, the greater reduction in shade change imparted by the full backtan aftertreatment is also evident.

A comparison of the colour strength reductions achieved for the three levelling acid dyes (Figs. 4–6) and for the three milling acid dyes (Figs. 7–9) clearly shows the difference in the level of wet fastness displayed by the two types of non-metallised acid dye. The overall superior wet fastness of the milling acid dyes is evident from the extent of colour loss achieved for the respective

non-aftertreated dyeings. Also, it is clear that with the exception of C.I Acid Blue 62, the level of colour loss achieved after five wash tests was 90% or greater as a result of an aftertreatment with the full backtan.

4. Conclusions

The newly developed, one-bath, two-stage, full backtan aftertreatment, which does not use pot-assium antimony tartrate, improved the fastness of each of the six non-metallised acid dyes. The extent of the fastness improvement secured, in terms of both reduction in colour strength and staining of adjacent multifibre, was much greater than that achieved using the commercial syntan.

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