



## The dyeing of supermicrofibre nylon with acid and vat dyes

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### ABSTRACT

5% omf dyeings of three 1:2 pre-metallised acid dyes displayed poor fastness to repeated washing at 60 °C on 0.05 dtex 'supermicrofibre' artificial nylon suede. Although fastness was improved by an aftertreatment with the full backtan, considerable dye loss still occurred during repeated washing, which was attributed to the large surface area of the microfibre substrate. In contrast, the wash fastness of 4% omf dyeings of three vat dyes, applied as both the alkali leuco and acid leuco variants, was substantially better than that of the acid dyes, despite the considerably higher colour strength of the vat dyeings. Although the rub fastness of the vat dyeings was higher than that of the acid dyeings, the level of rub fastness, especially wet, was only moderate. The colour and colour strength of the acid leuco dyeings were generally different to those of the alkali leuco dyeings.

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### 1. Introduction

Polyamide fibres are dyed predominantly using acid dyes, even though their wash fastness properties on such fibres leaves much to be desired [1]. Although 1:2 pre-metallised acid dyes generally display higher levels of wash fastness on nylon than their non-metallised counterparts, an aftertreatment of the dyed fibre with either a synthetic or a natural tanning is commonly used in order to achieve highest levels of wash fastness, especially in moderate to deep depths of shade [1]. The two-stage treatment of dyed nylon with tannic acid and potassium antimony tartrate constitutes the classic full backtan aftertreatment which is very effective in improving the wash fastness of acid dyes on nylon [1]. However, owing to many reasons, the use of the toxic antimony salt has been superseded in recent times by that of protease enzymes [2–4] and metal salts [5,6].

Polyamide microfibres are typically <1 dtex and enjoy manifold apparel applications owing to their soft handle, high lustre and excellent drapeability [1]. Although the polymer used in nylon microfibre is often the same as that employed in conventional dtex fibres and, therefore, microfibre can be dyed in a similar manner to its conventional counterpart, microfibre has a larger surface area than conventional decitex fibre per unit mass of substrate, resulting

in greater reflection of light from the microfibre surface which, in turn, results in higher amounts of dye being required to achieve the same visual depth of shade as that on conventional decitex fibres. For example, to produce the same depth of shade on 0.5 dtex microfibre as that obtained using 1% omf dye on 3.5 dtex conventional fibre, requires 2.65% omf dye [1]. This requirement to use higher amounts of dye on polyamide microfibre coupled with the inherently low/moderate wash fastness properties of acid dyes on nylon results in comparable depth dyeings on microfibre polyamide displaying lower wash fastness than on conventional decitex nylon fibres [1,7,8]. In addition, the larger surface area of microfibre results in a greater rate and extent of dye desorption during washing and, therefore, reduced levels of wash fastness [8]. This particular situation is exacerbated in the case of polyamide artificial suede [9], which was introduced some 40 or so years ago and which comprises 'supermicrofibre' of  $\sim 1.1 \times 10^{-4}$  to 0.33 dtex and which is considered to be the origin of textile microfibres [1]. Using the above example of a 1% omf depth of shade on 3.5 dtex fibre, in order to achieve the same depth of shade on 0.05 supermicrofibre suede would require the application of 8.7% omf dye.

Although vat dyes are widely used on cellulosic fibres on which they exhibit characteristically excellent light and wet fastness properties, this dye class is not routinely used on polyamide fibres (with the exception of their minor usage on nylon/cotton blends) owing to their low substantivity and the generally pale shades that accrue from their limited extent of diffusion within the substrate [1]. The dyes contain at least two conjugated carbonyl groups which,

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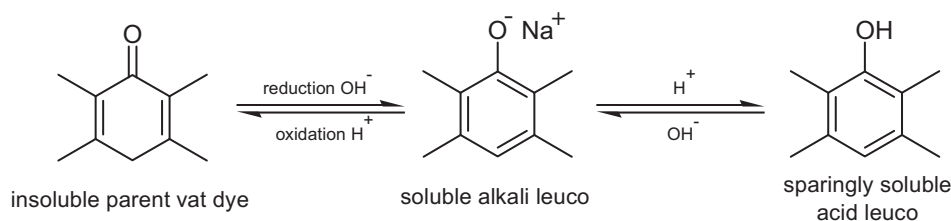


Fig. 1. Reduction/oxidation of vat dyes.

during their conventional application to cellulosic fibres, are converted by reduction under alkaline conditions to the corresponding, water soluble, 'alkali leuco' form which is applied to the substrate. At the end of dyeing, the alkali leuco form is oxidised, so as to regenerate the insoluble, parent vat dye *in situ* within the fibre (Fig. 1). However, the alkali leuco form is readily converted to the sparingly water-soluble 'acid leuco' variant (Fig. 1), which has been used to dye nylon fibres [10–12]. In effect, the acid leuco variant resembles a disperse dye in terms of its adsorption characteristics and, when oxidised at the end of dyeing, is converted into the insoluble parent vat dye *in situ* within the nylon substrate.

This paper concerns the dyeing of artificial nylon suede with both the alkali and acid leuco forms of vat dyes and compares the resulting dyeings with those obtained using 1:2 pre-metallised acid dyes which had been aftertreated with a modified full backtan, in terms of depth of shade and fastness to repeated washing.

## 2. Experimental

### 2.1. Materials

#### 2.1.1. Fabric

The artificial nylon suede used (0.06 dtex) was kindly supplied by Daewoo Co. Ltd., Seoul. The fabric was scoured in an aq. solution containing 2 g dm<sup>-3</sup> Na<sub>2</sub>CO<sub>3</sub> and 5 g dm<sup>-3</sup> non-ionic surfactant Lanapex R (ICI Surfactants) for 30 min at 60 °C. The scoured suede was rinsed thoroughly in tap water and allowed to dry in the open air.

#### 2.1.2. Dyes

The acid dyes used in this work, which were kindly supplied by Crompton & Knowles and the vat dyes used, which were kindly supplied by Town End Chemicals and Ciba–Geigy, are shown in Table 1. Each of the dyes was selected as being a typical representative of each dye class.

#### 2.1.3. Chemicals and auxiliaries

The commercial reducing agent *Rongalit C* was generously supplied by BASF and the non-ionic surfactant, *Sandozin NIE*, was kindly provided by Clariant; the dispersing agent *Matexil DA-N* was generously provided by Uniqema. Samples of the tannic acid *Floc-tan 1* and the tin sulfite derived fixing agent *Gallofix* were kindly supplied by Omnichem-Ajinmoto. All other reagents used were of standard laboratory grade.

Table 1

1:2 pre-metallised acid dyes and vat dyes used.

Commercial name	C.I. generic name
Neutrilan Bordeaux M-B	C.I. Acid Violet 90
Neutrilan Yellow S-2G	C.I. Acid Yellow 220
Neutrilan Navy M-BRX	C.I. Acid Blue 193
Cibanone Violet 2R	C.I. Vat Violet 1
Cibanone Navy BAMD	C.I. Vat Blue 18
Cibanone Olive B-01 MD	C.I. Vat Green 3

### 2.2. Dyeing

Samples of nylon suede were dyed in sealed, stainless steel dye pots of 300 cm<sup>3</sup> capacity, housed in a Zeltex Polycolor PC 1000 laboratory-scale dyeing machine, using a liquor ratio of 20:1. The dyeing methods used to apply the vat dyes and acid dyes are shown in Figs. 2–4.

### 2.3. Oxidation of vat dyeings

At the end of dyeing, the samples were rinsed for 10 min in tap water and then oxidised using a liquor ratio 20:1 (Fig. 5); the oxidised samples were then rinsed in running tap water for 10 min.

### 2.4. Reduction clearing of vat dyeings

The rinsed, oxidised dyeings were reduction cleared using a 20:1 liquor ratio (Fig. 6) and the samples were then rinsed in tap water and allowed to dry in the open air.

### 2.5. Modified full backtan aftertreatment of 1:2 pre-metallised dyeings

At the end of dyeing, the samples were rinsed for 10 min in tap water and then aftertreated using the one-bath, two-stage after-treatment process outlined in Fig. 7. The backtanned samples were then rinsed in tap water and allowed to dry in the open air.

### 2.6. Wash fastness

The dyed suede sample was subjected to five, consecutive ISO C06/C2 [13] wash tests. At the end of each wash test, the sample was rinsed thoroughly in tap water. A fresh sample of SDC multi-fibre strip was used to assess the extent of staining for each of the five wash tests. The extent of staining of the adjacent multifibre strip was expressed using the staining grey scale.

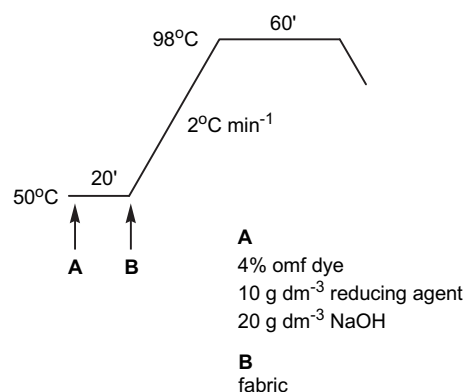


Fig. 2. Alkali leuco vat dyeing method.

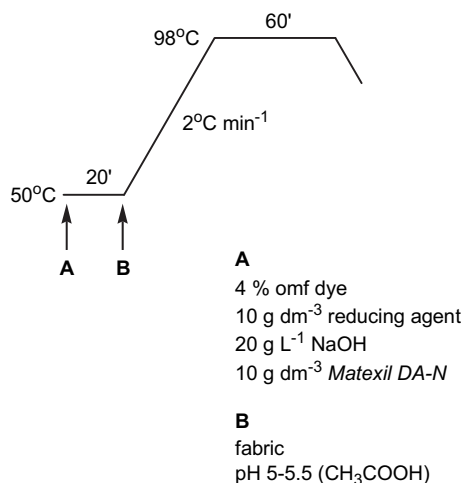


Fig. 3. Acid leuco vat dyeing method.

The extent to which repeated wash testing reduced the depth of shade of the dyeings was determined using Eq. (1) where the subscript 1 refers to the colour strength of the dyeing before washing and the subscript 2 refers to the colour strength of the dyeing after wash #1, #2, ... #5, respectively.

$$\% \text{ colour loss} = \left( \frac{fk_1 - fk_2}{fk_1} \right) \times 100 \quad (1)$$

## 2.7. Rub fastness

Dyed suede was subjected to the ISO X12 test method [13].

## 2.8. Colour measurement

All measurements were carried out using an X-rite Match-Rite spectrophotometer interfaced to a PC using D<sub>65</sub> light source and 10° standard observer, with specular component excluded and UV component included; an average of four readings was taken for each sample. The corresponding CIE colorimetric data and *fk* values were calculated from the reflectance data.

## 3. Results and discussion

### 3.1. Acid dyes

Table 2 shows the colorimetric data obtained for polyamide suede which had been dyed with 5% omf of each of the 1:2 pre-

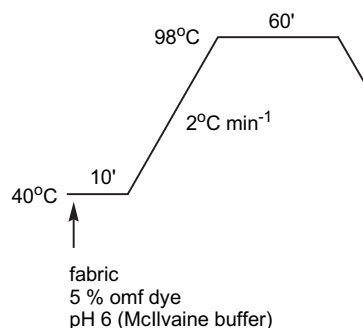


Fig. 4. Acid dye application method.

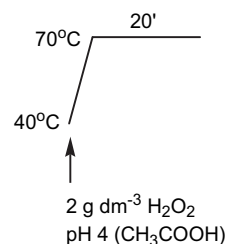


Fig. 5. Oxidation method.

metallised dyes acid dyes used; the effects of subjecting the dyeings to five repeated wash tests are also shown. In Table 2, results are included for dyeings which had received no full backtan after-treatment as well as dyeings which had been treated with the two-stage aftertreatment process depicted in Fig. 7.

### 3.1.1. Non-aftertreated dyeings

In the case of dyeings which had not been aftertreated with the full backtan, it is apparent that for each of the three dyes used, repeated washing reduced the depth of shade, as evidenced by an increase in lightness (*L\** value) but the colour (hue, *h* and chroma, *C\**) of the dyeings was little affected. As Fig. 8 shows, the colour strength (*fk*) of the dyeings decreased with increasing number of washes owing to desorption of dye having occurred during each of the five wash tests. Values of the corresponding % colour loss secured for the non-aftertreated dyeings, before and after repeated washing, are shown in Fig. 9 from which it is apparent that the extent of colour loss achieved for the first two or so wash tests was greater than that obtained for subsequent wash tests. This finding is also illustrated by the decreases in the corresponding *fk* values shown in Fig. 8 and can be attributed to surplus dye having not been removed from the dyeings at the end of dyeing; the finding (Fig. 9) that the % colour loss obtained for the dyeings decreased with increasing number of wash tests can be attributed to the first two or so washes having removed such surplus dye. It is apparent (Fig. 9) that whilst the three dyes varied in terms of the rate of colour loss that occurred over the five washes, there was relatively little difference in the final extent of colour loss, this being ~80% or so. Unsurprisingly, the vagrant dye that was removed from the dyeings during washing deposited on several of the adjacent multifibre strip materials (Table 2). It is clear that, for each of the three dyes used, the dyeings displayed poor fastness to five, consecutive wash fastness tests at 60 °C, as shown by the staining values achieved. The low levels of staining achieved in the cases of the adjacent acrylic, polyester, 2° acetate and cotton components was anticipated in view of the inherent low substantivity of 1:2 pre-metallised acid dyes towards such types of fibre. Also expected, in the context of the substantivity of the two dyes used, was the very high extent of staining obtained for the adjacent nylon 6,6 and wool

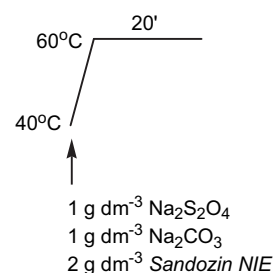


Fig. 6. Reduction clearing method.

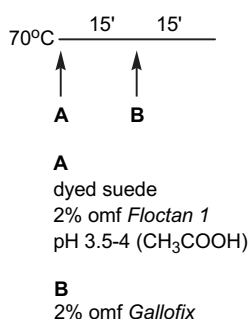


Fig. 7. Full backtan after treatment.

fibres, on which this type of dye is intended for use. It is evident (Table 2) that the fastness of the dyeings achieved for the first two or so wash tests was noticeably worse than that obtained for subsequent washes. This can be attributed to the first two or so washes having removed such surplus dye, as previously discussed for the % colour loss values shown in Fig. 9.

### 3.1.2. Aftertreated dyeings

Table 2 shows the colorimetric data obtained for the 5% omf dyeings which had been aftertreated with the full backtan using the method shown in Figs. 7 and 8 shows the corresponding *fk* values recorded for the dyeings both before and after repeated wash testing. Comparison of this data with that of the dyeings which had

not been aftertreated (Table 2 and Fig. 8) prior to wash testing, reveals that, for each of the three dyes, aftertreatment imparted little change to both the colour and colour strength of the dyeings. These findings are surprising in view of the discoloration that can be imparted by this particular aftertreatment [1,2] and may be due to the moderately deep and relatively dull shades employed in this work. In the two-stage aftertreatment employed herein, the gallotannin was firstly applied to the dyed nylon suede and the tanned fabric then treated with the metal salt from a fresh bath. In the case of polyamide fibres, it is believed [5,6] that the gallotannin behaves as a high *M<sub>r</sub>* acid, binding to the protonated amino end groups in the substrate and, that sequential treatment with the tin salt results in the formation of a large molecular size, low water-soluble complex that is situated at the surface of the dyed substrate and which provides a physical barrier to the diffusion of dye from the dyed fabric during washing. When the wash fastness data obtained for the dyeings which had been aftertreated are compared with those of the non-aftertreated dyeings (Table 2) it is clear that the full backtan improved fastness to repeated washing at 60 °C. The beneficial effect of aftertreatment on the extent of colour loss that occurred during washing is clearly seen in the data presented in Fig. 9. However, it is also apparent that the full backtan was less effective in improving the wash fastness of C.I. Acid Blue 193 than for either C.I. Acid Violet 90 or C.I. Acid Yellow 220 (Fig. 9). As aftertreatment resulted in reduced % colour loss for each of the five washes undertaken (Fig. 9), the corresponding colour strength of the aftertreated dyeings was higher than that of their non-aftertreated counterparts (Fig. 8).

Table 2

Colorimetric and wash fastness data for 5% omf acid dyeings.

Dye	After treatment	No. of washes	<i>L</i> <sup>*</sup>	<i>a</i> <sup>*</sup>	<i>b</i> <sup>*</sup>	<i>C</i> <sup>*</sup>	<i>h<sub>o</sub></i>	<i>fk</i>	Staining of adjacent					
									Acetate	Cotton	Nylon	PET	Acrylic	Wool
C.I. Acid Violet 90	nil	0	38.93	40.26	−3.67	40.42	354.79	75.5	—	—	—	—	—	—
		1	50.63	35.01	−5.56	35.45	350.98	30.2	5	4/5	2/3	4/5	5	2/3
		2	56.34	32.01	−5.54	32.49	350.18	19.8	5	5	3/4	5	5	4
		3	60.96	29.21	−5.42	29.71	349.48	14.0	5	5	4	5	5	4/5
		4	64.23	26.73	−5.25	27.24	348.89	10.9	5	5	4/5	5	5	4/5
	full backtan	5	66.97	24.98	−4.99	25.47	348.70	8.7	5	5	4/5	5	5	5
		0	38.12	40.31	−3.94	40.50	354.41	80.1	—	—	—	—	—	—
		1	42.79	36.74	−1.74	36.78	357.30	55.7	5	4	3	4/5	5	3
		2	46.00	36.58	−2.26	36.65	356.47	44.1	5	5	3/4	5	5	4
		3	46.55	35.07	−1.67	35.11	357.24	42.1	5	5	4/5	5	5	4/5
		4	49.81	34.16	−2.57	34.25	355.70	33.0	5	5	4/5	5	5	4/5
		5	50.31	33.06	−1.66	33.11	357.12	32.0	5	5	4/5	5	5	5
		0	28.42	0.73	−15.42	15.44	272.72	129.0	—	—	—	—	—	—
		1	34.74	−0.02	−15.53	15.53	269.94	81.6	5	4	1/2	4/5	4/5	2/3
		2	38.79	−0.31	−14.60	14.61	268.77	61.5	5	4/5	2	5	5	2/3
		3	42.08	−0.50	−14.11	14.12	267.98	49.3	5	5	2	5	5	3/4
		4	44.33	−0.67	−13.77	13.78	267.20	42.5	5	5	3/4	5	5	4
		5	45.99	−0.62	−13.49	13.50	267.35	43.3	5	5	3/4	5	5	4/5
		0	27.97	0.20	−15.48	15.48	270.75	134.6	—	—	—	—	—	—
		1	34.15	−0.21	−15.42	15.42	269.22	85.3	5	4	2	4/5	4/5	3
C.I. Acid Blue 193	nil	1	34.74	−0.02	−15.53	15.53	269.94	81.6	5	4	1/2	4/5	4/5	2/3
		2	38.79	−0.31	−14.60	14.61	268.77	61.5	5	4/5	2	5	5	2/3
		3	42.08	−0.50	−14.11	14.12	267.98	49.3	5	5	2	5	5	3/4
		4	44.33	−0.67	−13.77	13.78	267.20	42.5	5	5	3/4	5	5	4
		5	45.99	−0.62	−13.49	13.50	267.35	43.3	5	5	3/4	5	5	4/5
	full backtan	0	27.97	0.20	−15.48	15.48	270.75	134.6	—	—	—	—	—	—
		1	34.15	−0.21	−15.42	15.42	269.22	85.3	5	4	2	4/5	4/5	3
		2	38.33	−0.56	−14.41	14.42	267.78	63.8	5	5	3	5	5	3/4
		3	40.21	−0.71	−14.04	14.05	267.12	56.2	5	5	3	5	5	3/4
		4	42.52	−0.94	−13.48	13.51	266.02	48.2	5	5	3/4	5	5	4
		5	44.23	−0.99	−13.11	13.35	265.74	43.0	5	5	3/4	5	5	4/5
		0	63.79	18.65	65.04	67.66	74.00	82.4	—	—	—	—	—	—
		1	68.36	15.31	59.75	61.68	75.63	46.4	4	4	2/3	4/5	4/5	2
		2	71.97	12.70	55.99	57.41	77.22	31.0	4/5	4/5	3	5	5	2/3
		3	73.96	10.67	51.70	52.79	78.34	23.0	5	5	3/4	5	5	3
		4	75.14	9.52	48.74	49.66	78.05	19.1	5	5	4	5	5	4
	full backtan	5	76.66	8.89	47.95	48.77	79.49	16.7	5	5	4	5	5	4
		0	64.18	17.40	63.71	66.05	74.72	75.5	—	—	—	—	—	—
		1	66.75	16.81	64.01	66.19	75.28	62.5	4/5	4/5	2/3	4/5	5	2/3
		2	68.23	14.60	60.49	62.23	76.43	48.3	5	4/5	3/4	5	5	3
		3	69.61	14.61	61.04	62.76	76.54	44.7	5	5	3/4	5	5	3
		4	69.98	14.03	58.89	60.54	76.60	39.9	5	5	4	5	5	4
C.I. Acid Yellow 220	nil	5	71.67	12.88	57.98	59.39	77.47	34.2	5	5	4/5	5	5	4
		0	63.79	18.65	65.04	67.66	74.00	82.4	—	—	—	—	—	—
		1	68.36	15.31	59.75	61.68	75.63	46.4	4	4	2/3	4/5	4/5	2
		2	71.97	12.70	55.99	57.41	77.22	31.0	4/5	4/5	3	5	5	2/3
		3	73.96	10.67	51.70	52.79	78.34	23.0	5	5	3/4	5	5	3
	full backtan	4	75.14	9.52	48.74	49.66	78.05	19.1	5	5	4	5	5	4
		5	76.66	8.89	47.95	48.77	79.49	16.7	5	5	4	5	5	4
		0	64.18	17.40	63.71	66.05	74.72	75.5	—	—	—	—	—	—
		1	66.75	16.81	64.01	66.19	75.28	62.5	4/5	4/5	2/3	4/5	5	2/3
		2	68.23	14.60	60.49	62.23	76.43	48.3	5	4/5	3/4	5	5	3

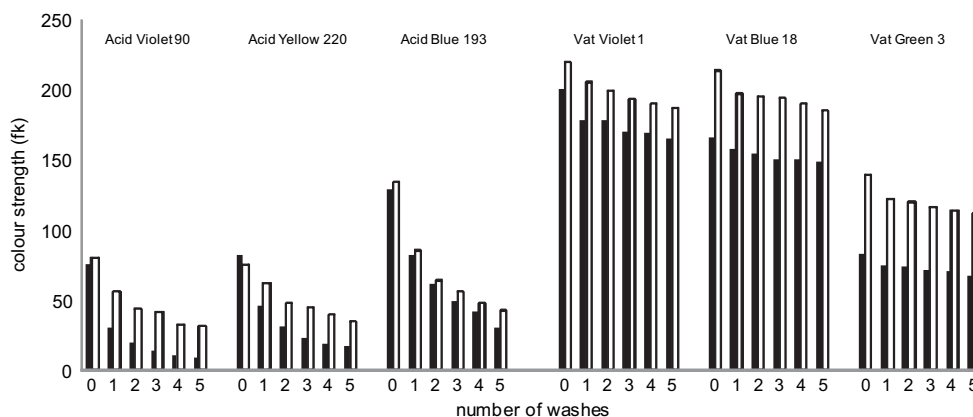


Fig. 8. Colour strength of acid and vat dyes before and after repeated ISOC6/C2 wash tests. Acid dyes: ■ nil after treatment □ full backtan Vat dyes: ■ alkali leuco □ acid leuco.

The observed improvement in the wash fastness of each of the three acid dyes imparted by the full backtan can be attributed to the surface 'skin' of the large  $M_n$  low water-soluble gallotannin/tin complex having restricted dye diffusion out of the dyed artificial suede material during washing, as proposed in the case of acid dyes on polyamide fibres [5,6]. However, in the case of such conventional nylon fibre, the full backtan aftertreatment imparts significant improvements in both loss of colour and the extent of staining of adjacent fibres during repeated washing [5]. Fig. 9 reveals that the polyamide suede which had been dyed and aftertreated suffered considerable colour loss as a result of repeated washing whilst Table 2 shows that noticeable staining of adjacent nylon and wool materials occurred even after five washes, in the case of the after-treated dyeings. The relatively large % colour loss and high levels of staining observed for the aftertreated dyeings can be attributed to the previously discussed, characteristically very high surface area of the supermicrofibre (0.05 dtex) used coupled with the inherently low/moderate fastness of acid dyes on nylon fibres.

Table 3 shows the rub fastness of nylon suede which had been dyed with the three acid dyes, from which it is clear that although the rub fastness was quite poor, aftertreatment improved fastness to a small extent.

### 3.2. Vat dyes

As previously mentioned, although vat dyes are widely used to dye cellulosic fibres, many dyes display only low substantivity towards nylon fibres and generally pale shades accrue from their limited diffusion within the substrate [1]. However, the acid leuco

form of some vat dyes has been shown to furnish deep shades of high overall fastness on polyamide fibres [10–12]. In this work both the alkali leuco and acid leuco forms of vat dye were employed.

The application methods used for the alkali leuco (Fig. 2) and the acid leuco (Fig. 3) forms of the dyes were devised as the result of previous work in the authors' laboratory [14], that included the three vat dyes listed in Table 1, together with the nylon suede and the equipment described in the experimental section above.

Table 4 shows the colorimetric data and Fig. 8 the corresponding colour strength values, obtained for 4% omf dyeings of the three vat dyes both before and after repeated washing; results are shown for the alkali and acid leuco application methods used (Figs. 2 and 3, respectively). It is apparent (Fig. 8) that the colour strength of the acid leuco dyeings were higher than the corresponding alkali leuco dyeings and, also, that the higher colour strength of the acid leuco form persisted over the five repeated washes, even though fk values decreased gradually as the number of washes increased. In the cases of C.I. Vat Violet 1 and C.I. Vat Blue 18, the colour of the acid leuco dyeings differed to that of the alkali leuco dyeings, both before and after wash testing; much smaller difference in colour were obtained between the acid and alkali leuco dyeings of C.I. Vat Green 3. Although the reasons for the differences in both colour and colour strength observed for the acid leuco and alkali leuco dyeings of the three vat dyes are unknown as yet, results obtained using a relatively large number of vat dyes and a wide range of vatting, dyeing and clearing conditions suggest [15] that dye aggregation is a major, but not determining factor in terms of colour and that the higher colour

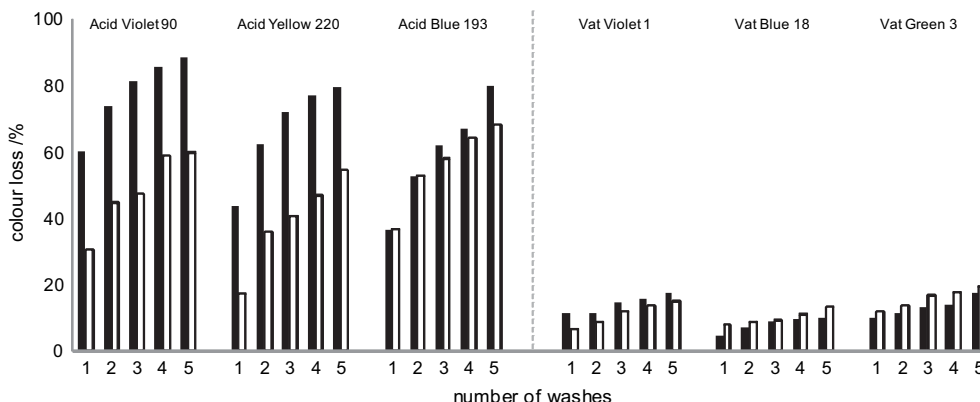


Fig. 9. % colour loss achieved for acid and vat dyes after repeated ISOC6/C2 wash tests. Acid dyes: ■ nil after treatment □ full backtan Vat dyes: ■ alkali leuco □ acid leuco.

**Table 3**

Rub fastness of 5% omf acid dyeings.

Dye	After treatment	Dry	Wet
C.I. Acid Violet 90	none	3/4	1
C.I. Acid Blue 193		3/4	1
C.I. Acid Yellow 220		3/4	1
C.I. Acid Violet 90	full backtan	3/4	1/2
C.I. Acid Blue 193		3/4	1/2
C.I. Acid Yellow 220		3/4	2

strengths that are generally achieved in the case of acid leuco dyeing, can be attributed to the lower solubility and, thus, higher substantivity, of the  $-OH$  derivative compared to that of the  $-O^-$  form of the dyes during application. A subsequent paper will be published which addresses these issues.

It is clear that both the alkali leuco and the acid leuco dyeings underwent only small % colour loss during repeated washing (Fig. 9) and that there was relatively small differences in the extent of colour loss displayed between the acid leuco and alkali leuco dyeings. The very low % colour loss values secured are reflected in the corresponding wash fastness results (Table 4), which reveal that both the acid leuco and alkali leuco dyeings displayed very good fastness to repeated washing. Although staining of the adjacent nylon component was observed for each of the three dyes, slight staining of adjacent diacetate fibre was obtained only in the case of C.I. Vat Green 3.

**Table 5**

Rub fastness of 4% omf vat dyeings.

Dye	Application method	Dry	Wet
C.I. Vat Violet 1	alkali leuco	4	2
C.I. Vat Blue 18		4/5	2/3
C.I. Vat Green 3		4/5	2/3
C.I. Vat Violet 1	acid leuco	3/4	2
C.I. Vat Blue 18		4/5	2/3
C.I. Vat Green 3		4/5	2/3

### 3.3. Comparison of acid and vat dyeings

The fastness results shown in Tables 2 and 4 clearly show that the vat dyeings (both acid and alkali leuco forms) displayed higher fastness than both the untreated and aftertreated acid dyes, even though the vat dyes were of much higher colour strength (Fig. 8); in addition, the vat dyeings exhibited considerably lower % colour loss over the repeated washing tests (Fig. 9). As the same scale was used for the y-axis in Figs. 8 and 9, the greater depth of shade and much lower loss of dye that were obtained for the vat dyeings is clearly visible.

Table 5 shows that the dry rub fastness of the vat dyeings was reasonably acceptable whereas wet rub fastness was <3. Nevertheless, the rub fastness ratings secured for the vat dyeings were much higher than those of the acid dyeings (Table 3) despite the considerably greater colour strength of the vat dyeings.

**Table 4**

Colorimetric and wash fastness data for 4% omf vat dyeings.

Dye	After treatment	No. of washes	L*	a*	b*	C*	ho	Staining of adjacent					
								Acetate	Cotton	Nylon	PET	Acrylic	Wool
C.I. Vat Violet 1	alkali leuco	0	21.3	19.4	−24.9	31.5	307.9	—	—	—	—	—	—
		1	22.7	19.5	−24.6	31.4	308.3	5	5	4/5	5	5	5
		2	22.6	19.6	−24.8	31.7	308.3	5	5	5	5	5	5
		3	23.1	19.6	−24.9	31.7	308.3	5	5	5	5	5	5
		4	23.2	19.7	−25.2	31.9	308.1	5	5	5	5	5	5
	acid leuco	5	23.5	19.9	−25.3	32.2	308.2	5	5	5	5	5	5
		0	20.2	15.7	−22.1	27.1	305.4	—	—	—	—	—	—
		1	20.9	15.9	−22.2	27.3	305.6	5	5	3/4	5	5	5
		2	21.3	15.9	−22.0	27.2	305.9	5	5	4	5	5	5
		3	21.6	16.4	−22.7	28.0	305.8	5	5	4/5	5	5	5
	alkali leuco	4	21.8	16.5	−22.6	27.9	306.1	5	5	4/5	5	5	5
		5	21.9	16.3	−22.3	27.6	306.2	5	5	4/5	5	5	5
		0	24.8	5.7	−15.6	16.6	290.2	—	—	—	—	—	—
		1	25.4	5.6	−15.3	16.2	289.9	5	5	3	5	5	5
		2	25.7	5.3	−15.1	15.9	289.5	5	5	4	5	5	5
	acid leuco	3	25.9	5.6	−15.4	16.3	289.9	5	5	4	5	5	5
		4	26.1	5.6	−15.5	16.5	289.7	5	5	4/5	5	5	5
		5	26.1	5.7	−15.6	16.6	289.9	5	5	4/5	5	5	5
		0	21.9	5.3	−18.5	19.3	286.0	—	—	—	—	—	—
		1	22.9	5.1	−18.3	19.0	285.6	5	5	4	5	5	5
C.I. Vat Blue 18	alkali leuco	2	23.1	5.2	−18.6	19.3	285.5	5	5	4/5	5	5	5
		3	23.1	5.3	−18.6	19.3	285.7	5	5	4/5	5	5	5
		4	23.4	5.3	−18.6	19.3	285.8	5	5	4/5	5	5	5
		5	23.7	5.2	−18.5	19.2	285.6	5	5	5	5	5	5
		0	38.5	−12.1	5.1	13.1	156.8	—	—	—	—	—	—
	acid leuco	1	40.1	−12.5	5.4	13.7	156.5	4	5	5	5	5	5
		2	40.3	−12.3	5.3	13.4	156.7	4	5	5	5	5	5
		3	40.7	−12.7	5.5	13.8	156.4	4	5	5	5	5	5
		4	40.7	−12.6	5.4	13.8	156.6	4	5	5	5	5	5
		5	41.4	−12.7	5.5	13.9	156.4	4	5	5	5	5	5
	alkali leuco	0	31.1	−12.3	4.5	13.1	159.7	—	—	—	—	—	—
		1	33.1	−12.9	4.4	13.6	161.1	4	4	4	5	5	5
		2	33.4	−13.1	4.4	13.8	161.2	4/5	5	4/5	5	5	5
		3	33.9	−13.2	4.4	13.9	161.3	4/5	5	4/5	5	5	5
		4	34.1	−13.3	4.4	14.0	161.3	4/5	5	4/5	5	5	5
	acid leuco	5	34.4	−13.4	4.5	14.1	161.4	5	5	5	5	5	5
		0	38.5	−12.1	5.1	13.1	156.8	—	—	—	—	—	—
		1	40.1	−12.5	5.4	13.7	156.5	4	5	5	5	5	5
		2	40.3	−12.3	5.3	13.4	156.7	4	5	5	5	5	5
		3	40.7	−12.7	5.5	13.8	156.4	4	5	5	5	5	5

The reasons for the observed much higher colour strength of the vat dyeings and their higher levels of wash and rub fastness are yet to be resolved. It may be that the extent of dye exhaustion was greater in the case of the vat dyes or it the dye content of the vat dye samples may have been greater than that of the acid dye samples used. These and other explanations require investigation.

#### 4. Conclusions

Artificial nylon suede poses problems in terms of wash fastness of acid dyes because of the very high surface area of the component supermicrofibres. The need to apply higher amounts of dye to achieve a given depth of shade on the supermicrofibre material coupled with the dye's inherent propensity to desorb from the dyed suede because of the high fibre surface area, results in characteristic poor wash fastness of acid dyes on artificial suede materials. The fastness of nylon suede which had been dyed with 5% omf of the three 1:2 pre-metallised acid dyes was markedly improved by an aftertreatment with the full backtan. However, although the full backtanned acid dyeings exhibited reduced dye loss during repeated washing at 60 °C, a considerable amount of colour loss still occurred during repeated wash testing, which can be attributed to the large surface area of the substrate. Also, despite the fact that the extent of staining of adjacent multifibre strip was reduced by aftertreatment with the full backtan, the level of staining still left much to be desired.

In the case of vat dyeing, the colour strength of the 4% omf acid leuco dyeings was higher than that of the 4% omf alkali leuco dyeings; the higher colour strength of the acid leuco dyeings persisted throughout repeated washing. Both the alkali leuco and the acid leuco dyeings displayed significantly lower colour loss compared with that obtained for the acid dyeings, despite both types of vat dyeings being of considerably greater colour strength than their acid dye counterparts. In addition, the alkali leuco dyeings did not impart any staining to the adjacent nylon and wool materials and the acid leuco dyeings imparted little staining to the adjacent nylon component during washing. When compared with

the extent of staining achieved for acid dyed suede, both the alkali leuco and the acid leuco dyeings exhibited markedly lower staining of the adjacent multifibre strip. Although the rub fastness of the vat dyeings was higher than that of the acid dyeings, the level of rub fastness, especially wet, was only moderate.

The results show that deep dyeings of high fastness to repeated washing at 60 °C and of good dry and moderate wet rub fastness can be obtained on artificial nylon suede. However, research, using a large number of vat dyes, is on-going, to characterise more fully the colorimetric and fastness (e.g. light, perspiration, etc.) properties of acid leuco dyeings on nylon fibres of varying dtex. In addition, work is in hand to attempt to identify the reasons for the observed differences between the dyeing of polyamide fibres with the acid leuco and the alkali leuco forms of vat dyes.

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