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Synthesis and properties of novel aziridinyl azo dyes from 2-aminothiophenes—Part 2: Application of some disperse dyes to polyester fibres

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Abstract

A series of yellow to greenish-blue aziridinyl azo dyes and their azo precursors containing a thienyl coupling moiety has been applied to conventional polyester fibre as well as microdenier polyester by high temperature exhaust dyeing. Heat transferability of these dyes onto polyester fibre has also been examined, using conventional heat-transfer printing techniques. The relevant dyeing characteristics, heat transferability, build-up, dyeability on microfibre polyester, washfastness and lightfastness are given. These aziridinyl dyes are reactive to polyester fibres under HT dyeing conditions. Fabrics dyed with aziridinyl dyes are more resistant to solvent extraction than those dyed with conventional dyes. Residual liquors showed only a pale colour when fabric dyed with aziridinyl dyes was dissolved and then precipitated, whereas a coloured polyester precipitate was obtained.

Keywords: Aziridinyl azo dyes; Heat transfer printing; HT exhaust dyeing; Microfibre polyester; Reactive dyes

1. Introduction

In the last three decades, innovations in azo dye chemistry based on heterocyclic systems have been made as a result of intensive studies stimulated by the mounting need for bright blue dyes. Generally speaking, many of the heterocyclic azo dyes show dramatic bathochromic shifts combined with brilliance of shade and high tinctorial strength compared with conventional anthraquinone dyes and aminobenzene azo dyes [1–3]. Of most importance are azo dyes derived from 2-aminothiophenes as diazo components resulting in the extension of hues to a greenish-blue [4–12]. In contrast to the success of heterocyclic diazo components, relatively few heterocyclic compounds have been used as coupling components [13–15].

Pyrazolone derivatives and pyridone compounds have been used to provide yellow to orange azo dyes, while some red azo dyes have been prepared from aminopyrimidines and cyanopyridines. There have been very few blue disperse dye derived from heterocyclic coupling components except for certain examples obtained from aminothiazoles, 4-phenylthiophenes and a tetrahydroquinoline coupling component [13].

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This paper reports the dyeing characteristics, heat transferability and fastness properties of the resultant dyeings using some azo precursor dyes **2** and aziridinyl dyes **1** derived from 2-aminothiophene coupling components (Fig. 1).

2. Results and discussion

2.1. *Dyeing characteristics on polyester fibres*

2.1.1. *Heat transfer printing*

Heat transfer printing is a technically-simple to use and highly economic process for polyester coloration. The dye is first placed on an intermediate substrate, usually paper, followed by subsequent heat treatment, the temperature of which is approximately 210°C. This process enables the dye to be transferred directly from the intermediate paper onto polyester fabric. The most important parameter for heat transfer printing is the sublimation ability of disperse dyes, which is generally associated with the relative molecular mass of the dyes; the smaller the dye molecules, the better the transferability onto polyester.

In this study, direct dependency of the transferability of dye into fabric is related to the M_r of each dye for both aziridinyl dyes **1** and their precursors **2** (see Tables 1 and 2). Thus, those dyes **1c**–**1f**, **1n** having M_r below 450 and other dyes **2a**–**2f** with M_r below 540 show good suitability for heat transfer printing. Not surprisingly, relatively larger dye molecules, such as **2l**, **2m** and **1m**, produce only pale hues on polyester. Emphasis must be placed on the exceptionally good performance in heat transfer printing observed for 2-CF_3-4-NO_2 substituted dyes **2j**, **1j** and the isomeric 4-CF_3-2-NO_2 substituted dyes **2h**, **1h** despite their high relative molecular mass. In line with unusually poor solubility in organic solvents, dyes **2o**–**2r** and **1o**–**1r** provide very limited applicability for heat transfer printing on polyester fabrics.

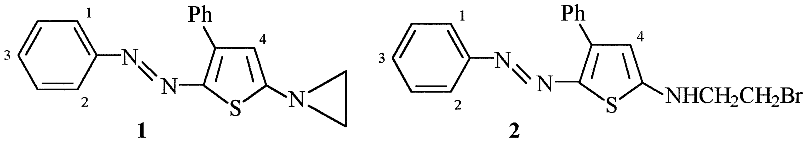
Tables 1 and 2 summarise the results of polyester coloration by heat transfer printing.

2.1.2. *High-temperature exhaust dyeing on conventional polyester fibre*

The hue of dyes **1** and **2** varied from yellow to greenish-blue depending on the substituents, as shown in Fig. 1. For dyes with a 3-NO_2 group, red-shifts were found compared with those of the dyes which had other 3-substituents. Dyes **2a**–**2e**, **2g**, **2h** and **1c**–**1e**, **1g**, **1h** showed yellow–pink shades, whereas dyes **2f**, **2i**–**2n** and **1i**–**1n** had red–violet colours.

More significant effects on the hue were found in the unsubstituted and 3-CO_2Et substituted coupling components. The inductive effect and the steric hindrance associated with a 3-substituent in relation to the lone pair electrons of the terminal nitrogen atom contribute to the bathochromic shifts exerted by azo dyes without a 3-substituent (see Table 3). All the dyes **2o**–**2r** and **1o**–**1r** appeared to be blue to greenish-blue shades on polyester fibre.

Table 3 illustrates the relative colour strength (% K/S) of dyed polyester fabrics depending on the amounts of dye applied (% o.m.f.), which is directly proportional to the build-up properties of dyes **1** and **2**.



1 **2**

1 = H, Cl, NO_2, CF_3, CN

2 = H, Br

3 = H, Br, Ac, CF_3, NO_2, CH_3SO_2, CN

4 = H, CO_2Et

Fig. 1. Structure of the precursor and aziridinyl azo dyes.

In connection with the colorimetric data, Fig. 2 and Fig. 3 show build-up curves of some dyes obtained by plotting the relative colour strengths on polyester fabrics of different % dye o.m.f., 1, 2, 3 and 4%. These diagrams show the amounts of dye in the dye bath for which the optical yield increases. At higher concentrations, build-up ceases to be linear, so that the amount needed to match a given colour shade has to be markedly increased.

Some of the precursor dyes, such as **2c**, **2f**, **2h**, **2k**, **2m**, **2q** and **2r**, built up well to give high colour

Table 1
Results of heat transfer printing onto polyester for precursors **2**

Dye	Substituents				Colour shade on polyester	M_r	Transferability
	1	2	3	4			
2a	H	H	H	CO_2Et	Yellowish orange	458	Good
2b	H	H	Br	CO_2Et	Yellowish orange	537	Good
2c	H	H	CF_3	CO_2Et	Orange	526	Good
2d	H	H	Ac	CO_2Et	Orange–scarlet	500	Good
2e	H	H	CN	CO_2Et	Orange	483	Good
2f	H	H	NO_2	CO_2Et	Red	503	Good
2g	Cl	H	CH_3SO_2	CO_2Et	Pink	571	Poor
2h	NO_2	H	CF_3	CO_2Et	Scarlet	571	Good
2i	Cl	H	NO_2	CO_2Et	Scarlet–red	539	Moderate
2j	CF_3	H	NO_2	CO_2Et	Bordeaux	571	Good
2k	NO_2	H	NO_2	CO_2Et	Brown	548	Poor
2l	CN	Br	NO_2	CO_2Et	Violet	607	Poor
2m	NO_2	Br	NO_2	CO_2Et	Violet	627	Poor
2n	CN	H	NO_2	CO_2Et	Violet	528	Moderate–good
2o	CF_3	H	NO_2	H	Greenish blue	499	Poor–moderate
2p	CN	Br	NO_2	H	–	535	Poor
2q	NO_2	Br	NO_2	H	–	555	Poor
2r	CN	H	NO_2	H	Greenish blue	456	Poor–moderate

Table 2
Results of heat transfer printing onto polyester for aziridinyl dyes **1**

Dye	Substituents				Colour shade on polyester	M_r	Transferability
	1	2	3	4			
1c	H	H	CF_3	CO_2Et	Orange	445	Good
1d	H	H	Ac	CO_2Et	Orange	420	Good
1e	H	H	CN	CO_2Et	Orange–scarlet	402	Good
1f	H	H	NO_2	CO_2Et	Red	422	Good
1g	Cl	H	CH_3SO_2	CO_2Et	Pink	490	Poor
1h	NO_2	H	CF_3	CO_2Et	Scarlet	490	Moderate–good
1i	Cl	H	NO_2	CO_2Et	Bordeaux	458	Poor–moderate
1j	CF_3	H	NO_2	CO_2Et	Bordeaux	490	Good
1k	NO_2	H	NO_2	CO_2Et	–	467	Poor
1l	CN	Br	NO_2	CO_2Et	Violet	526	Moderate
1m	NO_2	Br	NO_2	CO_2Et	Violet	546	Poor
1n	CN	H	NO_2	CO_2Et	Violet	447	Good
1o	CF_3	H	NO_2	H	–	418	Poor
1p	CN	Br	NO_2	H	Greenish blue	454	Moderate
1q	NO_2	Br	NO_2	H	–	474	Poor
1r	CN	H	NO_2	H	–	375	Poor

Table 3
Relative colour strength of dyed polyester by H.T. exhaust dyeing

Dye	Colour shade on polyester	Colour strength (% K/S)[a]		
		% Dye o.m.f.		
		2	3	4
1. Precursor dyes **2**				
2a	Yellow	183	241	287
2b	Yellow	187	228	247
2c	Yellowish orange	199	255	322
2d	Orange–scarlet	127	143	152
2e	Orange–scarlet	140	176	205
2f	Red	183	267	315
2g	Scarlet	176	217	262
2h	Orange–scarlet	197	285	366
2i	Bordeaux	165	227	254
2j	Bordeaux	178	251	294
2k	Brown	186	262	358
2l	Blueish violet	144	179	198
2m	Reddish violet	201	298	366
2n	Reddish violet	147	191	240
2o	Blue	177	258	310
2p	Greenish blue	175	271	302
2q	Greenish blue	161	245	327
2r	Greenish blue	171	264	336
2. Aziridinyl dyes **1**				
1c	Yellowish orange	151	210	251
1d	Orange–scarlet	155	215	255
1e	Orange–scarlet	180	206	229
1f	Red	190	204	222
1g	Scarlet	161	206	240
1h	Scarlet	153	214	260
1i	Bordeaux	144	195	215
1j	Bordeaux	192	273	323
1k	Brown	189	265	320
1l	Violet	131	177	202
1m	Violet	142	222	281
1n	Violet	135	187	236
1o	–	156	209	257
1p	Greenish blue	177	259	297
1q	Greenish blue	149	189	234
1r	Greenish blue	164	215	265

[a] Relative to K/S values of 1% o.m.f. dyeing.

depth; from these the deepest dyeing was found with dye **2m** containing 2,4-di-NO_2-6-Br substituents. For dyes **2o**–**2r**, with a 3-H atom, relatively higher build-up properties were seen compared with dyes substituted by a 3-CO_2Et group together with similar extent of dye uptake. In Table 3, it is evident that each aziridinyl dye **1c**–**1n** built-up very similarly in contrast with the corresponding precursor dyes **2c**–**2n**, which is attributable to the more compact size of the aziridine ring.

2.1.3. High-temperature exhaust dyeing on microfibre polyester

Because of the greater surface area of microfibre polyester, which may contain many times the number of filaments as normal fibres, colour depth on microfibres looks far lighter owing to the increased reflection of light at the fibre surface. Therefore, larger amounts of dye are needed to obtain an equal depth of colour on microfibres compared with normal polyester [16–18].

Table 4 exemplifies the amounts of dye required for the same colour depth on textured polyester of different counts [16].

The amount of dyestuff needed can be calculated according to the Fothergill method as shown in the following equation [17,19]:

$$C_1 = \sqrt{\frac{\mathrm{dtex}_c}{\mathrm{dtex}_m}} \times C_2$$

where, C_1 = % dye on microfibre, C_2 = % dye on conventional fibre, dtex_m = d.p.f. of microfibre, dtex_c = d.p.f. of conventional fibre.

Therefore, simple calculations for both microfibre and regular polyester used in this study suggest that 1.87% of dye will be needed in microfibre dyeing to obtain the same shade as a 1.0% dyeing on regular fibre from a theoretical point of view, as shown in the following equation.

$$C_1 = \sqrt{\frac{5.22}{1.49}} \times 1 = 1.87(\%)$$

Thus, the microfibres used for this research are dyed to approximately 53% of the colour depth associated with regular polyester (see Table 5).

Table 6 summarises relative colour depths on microfibre polyester applied with aziridinyl dyes **1**, their precursors **2** and some other dyes compared with the corresponding colour depths on regular polyester. These measurements reflect the dyeability of dyes on microfibre.

Whilst most of the aziridinyl dyes **1** responded to the theoretical expectation of 53% of colour

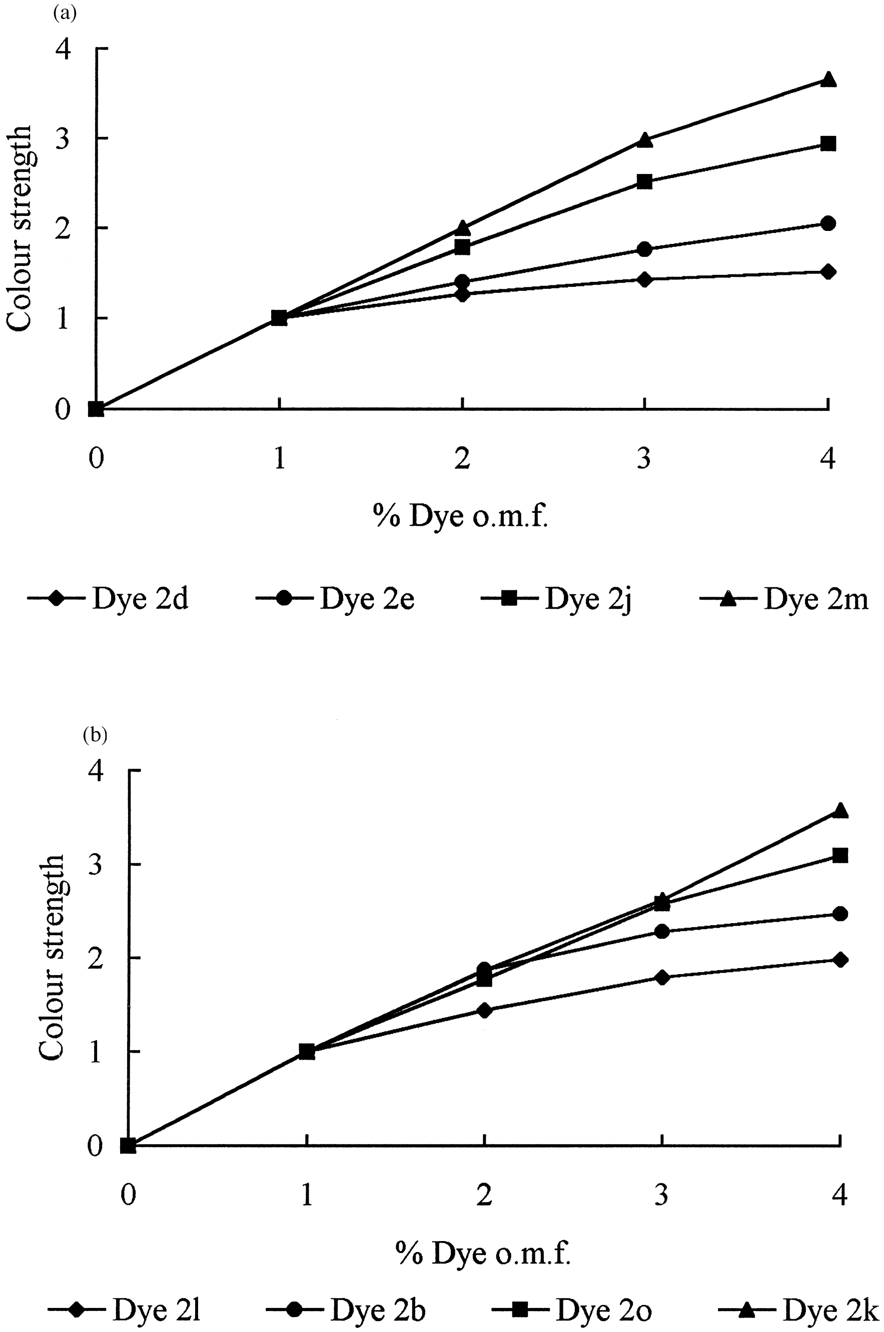


Fig. 2. Build-up curves of some precursors **2**.

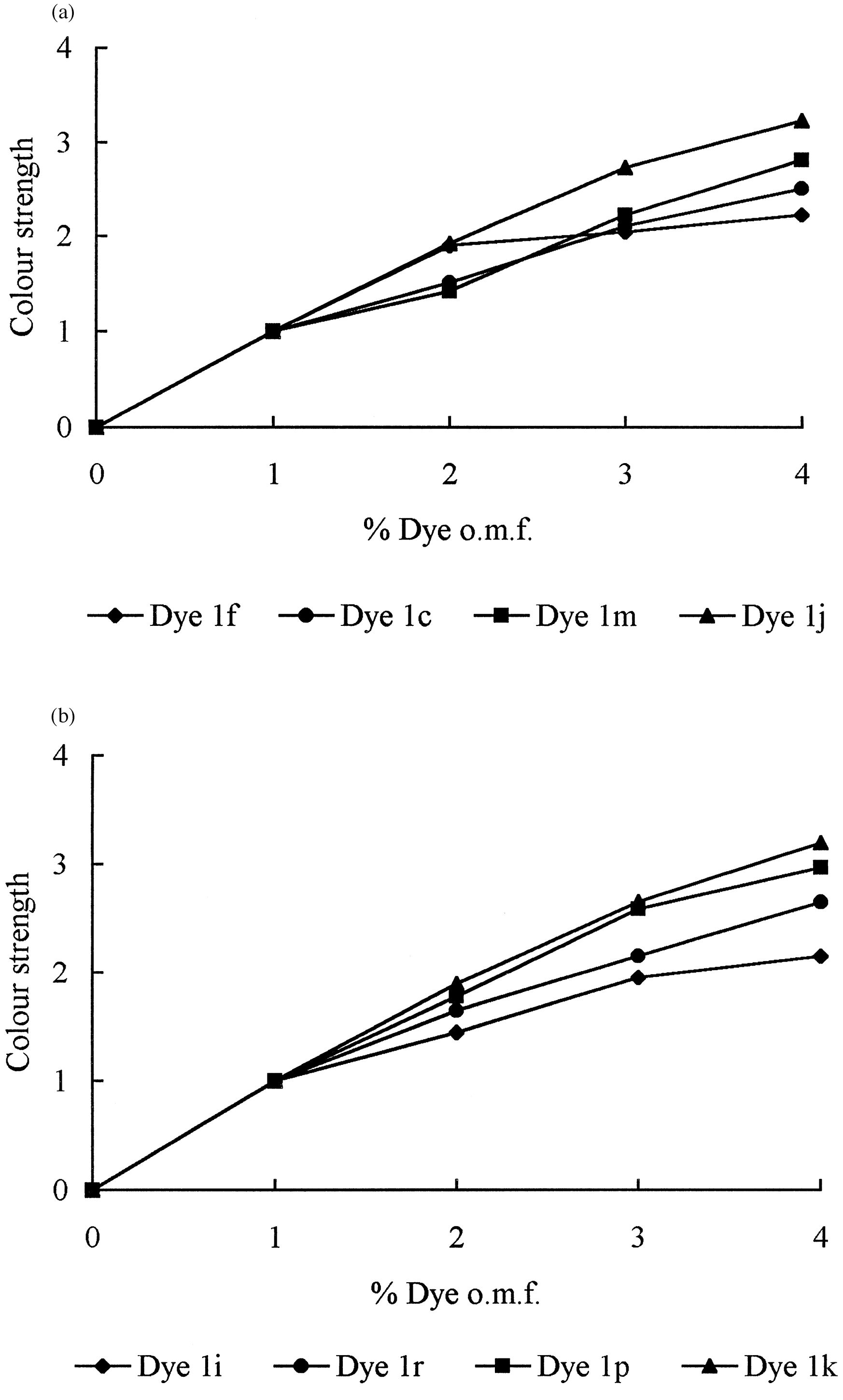


Fig. 3. Build-up curves of some aziridinyl dyes **1**.

Table 4
Amounts of dye required for identical shade depths on polyester of different counts

	C. I. Disperse			
dtex/f	Red 86	Red 302	Red 82	Blue 60
5.60	100	100	100	100
1.40	184	189	206	194
1.06	210	189	240	193
0.60	221	242	283	296
0.47	310	266	348	287

Table 5
Specifications of microfibres and regular polyester fibres used for dyeing

	Microfibre PES	Regular PES
Type of yarn	Draw textured	Draw textured
dtex of warp	2.08 (75/36)	5.22 (167/32)
dtex of weft	0.52 (150/288)	5.22 (167/32)
Density	137×84	–
Average dtex	1.49	5.22

Table 6
Relative colour strengths on microfibre polyester (4% o.m.f. dyeing)

Dye	% K/S[a]	Dye	% K/S[a]
2a	51	–	–
2b	56	–	–
2c	59	**1c**	52
2d	65	**1d**	47
2e	61	**1e**	51
2f	56	**1f**	56
2g	52	**1g**	52
2h	57	**1h**	35
2i	42	**1i**	55
2j	76	**1j**	60
2k	59	**1k**	44
2l	50	**1l**	49
2m	57	**1m**	43
2n	85	**1n**	49
2o	43	**1o**	55
2p	43	**1p**	61
2q	56	**1q**	52
2r	52	**1r**	49

[a] Relative to K/S values on conventional polyester.

depth previously calculated, their precursor dyes **2** exhausted stronger on microfibre polyester with a few exceptions, such as dyes **2i**, **2o** and **2p**. It seems likely that some aziridinyl dye decomposes due to sensitivity to aqueous acid during the dyeing stage. Consequently, relatively lower colour strength values (% K/S) were obtained with the aziridinyl dyes **1**. For dyes **2j** and **2n**, dyeability on microfibre polyester appeared to be promising in comparison with conventional azo dyes.

2.1.4. Fastness properties of the dyeings

2.1.4.1. Wash fastness. Using a standard multifibre fabric as the adjacent fabric, the staining of the multifibre fabric and the change of shade were assessed.

Judging from the fastness data, as shown in Table 7, appreciable improvements in the staining of adjacent fabrics for the fibre samples dyed with aziridinyl dyes **1** were achieved in comparison with the corresponding precursors **2**. The biggest advance appeared on the staining of the nylon 6.6 fabric among six different materials together with a slight increase in the ratings for the staining of the adjacent secondary cellulose acetate fabric.

Better washfastness is directly associated with the unique characteristic of aziridinyl dyes **1** of exceptional resistance to extraction from polyester fibres by chlorobenzene (see section 2.2. Examination of dyeing behaviour of aziridinyl dyes).

Although washfastness can vary independently with substituents in the dyes, ratings tend to increase somewhat with the presence of stronger electron-attracting groups in the acceptor ring of dyes **1** and **2**; the 4-nitro group seems to be particularly effective. Washfastness data are tabulated in Table 7.

2.1.4.2. Lightfastness. The lightfastness properties of substituted aziridinyl dyes **1** varied on polyester fibres with slight improvements compared with the corresponding precursors. The best examples were observed with dyes **2k** and **1k** containing 2,4-dinitro groups in the acceptor ring, whereas all dyes having a 4-substituent faded readily. The observed low lightfastness of precursors **2** may be attributable to the presence of the secondary amine group and/or the terminal bromine atom in the dye molecules.

As seen in Table 8, oxidative degradation is likely to dominate the reductive mechanism on

Table 7
Washfastness data (ISO 105-C 06, 60°C)

		Staining					
Dye	Change of shade	CA	CO	PA 6.6	PES	PAC	WO
1. Precursor dyes **2** (2% o.m.f. dyeing)							
2a	5	3–4	4–5	3	4–5	4–5	3–4
2b	5	3–4	4–5	2–3	4–5	4–5	4
2c	5	3–4	5	2–3	4–5	4–5	3–4
2d	5	4	5	3–4	4–5	4–5	4
2e	5	3–4	5	2–3	4–5	4–5	4
2f	5	4	5	3	5	5	4
2g	5	4	5	3	5	4–5	4–5
2h	5	4–5	5	3–4	5	4–5	4–5
2i	5	4	5	3–4	4–5	5	4
2j	5	4	5	3	5	5	4–5
2k	5	4–5	5	4	5	5	4
2l	5	4	5	3–4	5	5	5
2m	5	4–5	5	3–4	5	5	5
2n	5	3	5	2	4–5	5	4
2o	4	4	5	3	4–5	5	4–5
2p	5	4–5	5	3–4	5	5	4
2q	4	4–5	5	3–4	4–5	5	4–5
2r	4	4	5	2–3	5	5	4
2. Aziridinyl dyes 1 (3 % o.m.f. dyeing)							
1c	5	4	5	4	5	4–5	4
1d	5	4–5	5	3–5	4–5	4	
1e	5	4–5	5	3–4	5	4–5	4–5
1f	5	4–5	5	4	5	5	4
1g	5	4–5	5	4	5	4–5	4
1h	5	4–5	5	4	5	5	4–5
1i	5	4–5	5	4	5	5	4
1j	5	4–5	5	3–4	5	5	4–5
1k	5	4–5	5	4–5	5	5	4–5
1l	5	4–5	5	4	5	5	4–5
1m	5	4–5	5	4	5	5	5
1n	5	4	5	3–4	4–5	5	4–5
1o	5	5	5	4	5	5	4–5
1p	5	4–5	5	4	5	5	5
1q	5	5	5	3–4	5	5	5
1r	5	5	5	4	5	5	4–5

polyester, since generally better ratings were obtained for 2,4-disubstituted and 2,4,6-trisubstituted dyes than for 4-monosubstituted dyes.

Nevertheless, these results imply that aziridinyl dyes **1** do not offer notable advantages over conventional disperse azo dyes in respect to lightfastness on polyester fibres. Table 8 gives detailed results of lightfastness measurements.

Table 8
Lightfastness data (ISO B02, Xenon, 2% o.m.f. dyeing)

Dye	Rating	Dye	Rating
2a	2	–	–
2b	2	–	–
2c	2–3	**1c**	2–3
2d	2	**1d**	2
2e	2	**1e**	2–3
2f	2–3	**1f**	2–3
2g	2–3	**1g**	3
2h	5	**1h**	3–4
2i	2–3	**1i**	3
2j	4	**1j**	4–5
2k	5	**1k**	5
2l	3–4	**1l**	3–4
2m	3	**1m**	3–4
2n	3–4	**1n**	4
2o	2	**1o**	2–3
2p	2–3	**1p**	3
2q	2–3	**1q**	3–4
2r	2	**1r**	3–4

2.2. Examination of dyeing behaviour of aziridinyl dyes

2.2.1. Extraction of dyes from dyed fabrics

Notable differences between aziridinyl dyes **1** and the corresponding precursors **2** were observed by extracting each dyed fabric with chlorobenzene, and assessing changes of colour depth. As Table 9 reveals, for the aziridinyl dyes comparatively smaller amounts of dye were removed from polyester fibre compared with the corresponding precursors.

Considering that chlorobenzene is the most popular solvent for the extraction of normal disperse dyes from polyester, it can be suggested that the aziridinyl dyes may react with ambient polyester molecules to form strong bonds. Thus, fabrics applied with aziridinyl dyes are more resistant to removal of dye by solvent extraction than those dyed with conventional disperse dyes.

2.2.2. Dissolution of polyester fabrics

Polyester fibres can be dissolved within 5 min by immersion in a 1:1 mixture of trichloroacetic acid and chloroform at 20–25°C [20]. Thereafter, precipitated polyester can be collected by pouring the resulting solution into acetone.

Table 9
Results of chlorobenzene extraction from dyed fabrics

	% K/S ÷ K/S[b]	
No. of dye	Precursor dye	Aziridinyl dye
c	22	64
d	17	53
e	42	74
f	14	46
g	11	45
h	74	89
i	33	58
j	21	51
k	51	76
o	2	45
p	26	57
q	9	48
r	17	42

[a] K/S value before extraction.
[b] K/S value after extraction.

This investigation also revealed different dyeing behaviour between the aziridinyl dyes **1** and their precursors **2**. Fabric dyed with aziridinyl dyes gave a coloured polyester precipitate and a residual liquor having only a pale colour which was not identical to that of the aziridinyl dye. In contrast, the corresponding fabric dyed with precursor dyes gave deeply coloured liquors containing the same dye molecule which was applied to the polyester (as shown by TLC).

Consequently, it is clear that aziridinyl dyes are reactive to polyester fibres under HT dyeing conditions.

3. Experimental

Details for the synthesis and purification of dyes **1** and **2** and their spectral properties have been reported previously [21].

3.1. Heat transfer printing

A strip of transfer printing paper (Sumitomo Chem., 2.5×2.5 cm size) was immersed in a dye solution (1–2% depending on the solubility of dyes) in acetone (2–3 ml). The strip was dried carefully in a dryer, placed on a piece of polyester fabric (5×5 cm size) and enclosed in aluminium foil. The sample was placed between two plates of a Badische hot press at 210°C for 30 s.

3.2. Preparation of dye samples for HT dyeing

A stock solution of each dye sample was prepared by milling the mixture of dye (100 mg) and dispersing agent Ufoxane 2 (200 mg, Borregard Industries) in distilled water (10 g) with milling beads overnight. For those aziridinyl dyes highly unstable in water, the appropriate amount of dye was dissolved in acetone, then added to the dyebath without milling.

3.3. HT dyeing on polyester fabrics

Dyeing was carried out in sealed, stainless steel dye pots of 100 ml capacity in a Mathis LABOMAT. An appropriate volume of the stock solution was taken according to o.m.f. 1, 2, 3, 4%, and placed in each pot with 40 ml of distilled water for normal polyester or 70 ml of distilled water for microfibre polyester. Additional dispersing agent (Ufoxane 2), an equivalent amount to the dye, was also added, and the pH of the dyebath was adjusted to 5–5.5 with an aqueous acetic acid except for the dyebath of the aziridinyl dyes which were applied at neutral pH.

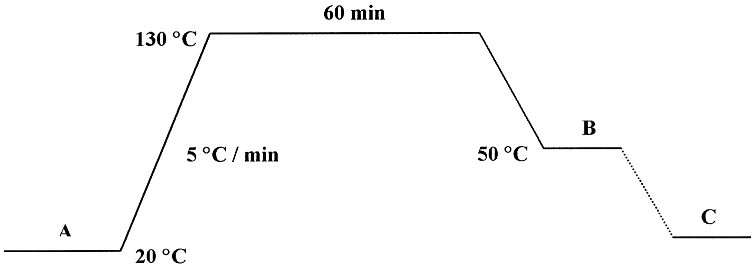
The dyeing process used is shown in Fig. 4.

3.4. Reduction clear

Dyed fabric was reduction cleared by stirring in an aqueous solution containing 2g/litre of sodium dithionite, 2g/litre of sodium hydroxide and 1g/litre of Sandozin NIE (Sandoz) for 15 min at 75–80°C. The treated polyester was then rinsed hot and cold, and acidified in an 1 ml/litre acetic acid solution, and then rinsed with water.

3.5. Washfastness

The reduction cleared, dyed fabrics were tested according to the ISO C06/C2 wash test using an ECE detergent. The change in shade and staining of adjacent multifibre fabrics (S.D.C. multifibre test fabric) were assessed using the S.D.C. grey scales.



A = polyester fabrics and dye solutions added

B = washed with hot water and cold water

C = reduction clear

Fig. 4. Diagram of the HT dyeing process used in this study.

Table 10
Lightfastness test conditions

Black panel temperature	45–50°C
Relative humidity	60%
Exposure time	24 h

3.6. *Lightfastness*

A lightfastness test was carried out in accordance with the BS B02 lightfastness in a Xenotest 150S (Heraeus). The conditions of the test are given in Table 10.

3.7. *Build-up properties*

Relative K/S values of each dyed fabrics to o.m.f. 1% dyeing were measured in a Colorgen Spectrophotometer (D65 1964 standard observer).

4. Conclusions

Dyeing properties and some fastness properties of aziridinyl azo dyes and their precursors containing a thienyl coupling moiety have been examined. A gamut of colour shades ranging from yellow to greenish-blue was obtained by applying the variously substituted dyes. Bathochromic colours, as expected, were observed as stronger electron-withdrawing substituent were introduced. All the dyes having small M_r values were applicable to heat transfer printing to produce strong colours on polyester fabrics via intermediate paper. In terms of build-up properties, it was found that a bulky substituent, such as 2-NO_2 and 2-CF_3, enhanced the extent of dyebath exhaustion according to the dye concentrations used. Most dyes built up well on microfibre polyester, somewhat better than the theoretical values calculated using the Fothergill method.

Better washfastness ratings were generally observed with the aziridinyl dyes compared with the corresponding precursors, in line with their exceptional resistance to extraction by solvent. Although the dyes were susceptible to photo-fading, oxidative degradation was likely to dominate the reductive mechanism on polyester.

The aziridinyl dyes are reactive to polyester fibres under HT dyeing conditions.

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