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Sulphur dyes on nylon 6,6. Part 3. Preliminary studies of the nature of dye—fibre interaction

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Abstract

Four different types of nylon 6,6 fabric (deep-dyeable, standard-dyeable, cationic-dyeable and acetylated variants) were dyed using solubilised sulphur dyes as well as sulphur dyes. The colour strength of the dyeings on the four different types of nylon 6,6 fabric was little influenced by either the AEG content of the fibre or the nature of the polymer from which the substrates had been made. The finding that the colour of dyeings on each of the four types of fibre was not dissimilar also implied that the interactions between both solubilised sulphur dyes as well as sulphur dyes and the different fabrics was little influenced by the nature of the polymer. It is postulated that the adsorption of both solubilised sulphur dyes and sulphur dyes onto nylon 6,6 is predominantly non-site-specific and, therefore, that dye—fibre substantivity occurs principally through H-bonding, dispersion forces and polar van der Waals' forces of interaction.

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1. Introduction

Despite the enormous body of literature that has been published on the use of many types of dye (e.g. acid, mordant, direct, reactive, disperse) on nylon 6,6 since the fibre's commercial introduction over 60 years ago, it is only in recent times that interest has attended the application of sulphur dyes to nylon 6,6 and, unsurprisingly, very few publications have appeared on the dyeing of nylon 6,6 with sulphur dyes.

It is well known that the presence of amino end groups (AEG) in nylon fibres imparts substantivity towards various classes of anionic dye and it is widely held that the substantivity of such anionic dyes, under

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acidic conditions, towards nylon 6,6 is based mainly on electrostatic forces of interaction operating between anionic (typically sulphonate) groups in the dye and the protonated, amino end groups in the fibre; the adsorption of anionic dyes on nylon is thus considered to be site-specific [1]. However, in terms of the dyeing of nylon with sulphur dyes, the nature of the interaction between dye and fibre has not been studied.

Very little is known about the chemical structure of sulphur dyes and the mechanism of their interaction with cellulosic fibres, despite their widespread use on these fibres over several decades; even less is known of the mechanism of dyeing nylon 6,6 with sulphur dyes. In terms of sulphur dye—cellulosic fibre substantivity, it can be proffered that H-bonding and dispersion forces are important contributors, as is assumed to be the case for the dyeing of cellulosic fibres with vat dyes. In essence, the cellulosic fibre provides a plethora of non-specific adsorption sites at which H-bonding

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can occur; by further analogy with the vat dyeing of cellulosic fibres, multi-layer adsorption/dye aggregation may also contribute towards sulphur dyecellulosic fibre substantivity. It is accepted that in the dyeing of cellulosic fibres with sulphur dyes, dyeing comprises two main stages, namely reduction and oxidation (Fig. 1). Reduction of the waterinsoluble, macromolecular sulphur dye under alkaline conditions affects the constituent di- and polysulphide bonds in the dye and results in cleavage of the large $M_{\rm r}$ dye, with the formation of the low $M_{\rm r}$, alkalisoluble, thiol form of the dye; it is this leuco variant of the dye which is adsorbed on to the cellulosic fibre during dyeing. At the end of dyeing, subsequent oxidation of the leuco form of the dye results in the oxidative condensation of the insoluble, macromolecular sulphur dye in situ within the fibre and the generation of the final colour (Fig. 1). From the foregoing, it is not surprising that the character (i.e. the colour and fastness properties) of the final dyeing is influenced by both the reductant and oxidant used. From a mechanistic point of view, this implies that the colour and fastness properties of sulphur dyeings on cellulosic fibres are determined by the nature of the reduced species present in the dyebath (i.e. the state of reduction of the dye) and their subsequent oxidative condensation (i.e. the nature of the oxidation process).

In the context of the sulphur dyeing of nylon fibres, if it is initially assumed that the overall reduction and oxidation mechanisms which are thought to apply to the dyeing of cellulosic fibres with sulphur dyes also apply to the dyeing of nylon 6,6 with sulphur dyes then the nature of the final colorant achieved at the end of sulphur dyeing will depend upon the nature of the reduced species present in the dyebath (i.e. the state of reduction of the dye) and their subsequent oxidative condensation (i.e. the nature of the oxidation process). Evidence in support of this accrues from the finding [2] that, for five commercial sulphur dyes, different reductants affected not only the colour strength of the dyeings but also the

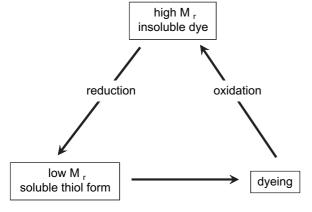


Fig. 1. Idealised dyeing mechanism.

final colour of the dyeings. In addition, it was found that whilst there was very little difference between three oxidising systems examined in terms of the colour strength of dyeings, the colour of the dyeings differed for the three oxidation systems studied [2]. Thus, initially at least, there appear to be some similarities between the dyeing of nylon 6,6 and cellulosic fibres with sulphur dyes.

As the two types of fibre differ, both chemically and physically, this preliminary study of the nature of the interaction between sulphur dyes and nylon 6,6 focussed on the role of the terminal amino end groups in dye adsorption and, therefore, whether or not the interaction between sulphur dyes and nylon 6,6 was sitespecific.

2. Experimental

2.1. Fabrics

Scoured, woven fluorescent brightener-free cotton fabric (130 gm⁻²), obtained from Whaleys, Bradford and four types of scoured, knitted nylon 6,6 fabrics, generously supplied by DuPont (UK) (Table 1) were used. Cationic-dyeable nylon 6,6 fabric was acetylated [3] by treating fabric (5 g) with 30 g acetic anhydride in xylene (90 cm³) for 10 h. At the end of the process the fabric was rinsed with acetone/water (20:80) solution and then left to dry in the open air. The AEG content of the acetylated nylon 6,6 was found to be 2.3 meq kg⁻¹ [4].

2.2. Dyes and auxiliaries

A total of thirteen, arbitrarily chosen, acid, reactive, solubilised sulphur and sulphur dyes (Table 2) were used; the commercial grade dyes used were kindly supplied by the respective maker; *Formosul Gr*, (reductant) and *Diresul BRI* (oxidant) were kindly supplied by Clariant UK. All other chemicals used were laboratory grade reagents obtained from Aldrich.

2.3. Dyeing

All dyeings were carried out in 300 cm³ capacity sealed stainless steel dyepots, housed in a *Roaches Pyrotec S* dyeing machine.

Table 1 Nylon 6,6 fabrics used

Designation		AEG/meq kg ⁻¹
Deep dyeable	78f68, 1.15 dtex per filament	70
Standard dyeable	78f68, 1.15 dtex per filament	45
Cationic dyeable	78f68, 1.15 dtex per filament	15
Acetylated	78f68, 1.15 dtex per filament	2.3

Table 2 Commercial dyes used

Commercial name	C.I. Generic name	Manufacturer
Commercial name	C.I. Generic manie	Manufacturer
Nylosan Yellow FL Nylosan Black FWLN Nylosan Red FGS	Acid Yellow 236 Acid Black 194 Non-ascribed	Clariant
Lanasol Blue 3R Lanasol Red 2G	Reactive Blue 50 Reactive Red 116	Ciba-Geigy
Stanalan Black MFFN	Non-ascribed	Dystar
Sulphosol Yellow SR	Solubilised Sulphur Yellow 23	J Robinson
Sulphosol Bordeaux SB	Solubilised Sulphur Red 6	
Sulphosol Brown SGR	Solubilised Sulphur Brown 10	
Diresul Black liq. RL 200% Diresul Green liq. RDT N Diresul Navy 2R Diresul Bordeaux 6R	Sulphur Black 1 Sulphur Green 2 Sulphur Navy 11 Sulphur Red 10	Clariant

2.3.1. Acid dyes

Dyeings of 2% omf were obtained using the method shown in Fig. 2; the pH was adjusted to 5.0 using McIlvaine buffer [5]. At the end of dyeing, the dyed sample was removed, rinsed thoroughly in tap water and allowed to dry in the open air.

2.3.2. Reactive dyes

Dyeings of 3% omf were produced using the method shown in Fig. 3, the pH being adjusted to 4.5, using McIlvaine buffer. The dyeings were removed, rinsed thoroughly in tap water and allowed to dry in the open air.

2.3.3. Solubilised sulphur dyes

Dyeings of 10% omf were obtained using the method shown in Fig. 4 employing *Formosul GR* as reducing agent. At the end of dyeing, samples were rinsed for 5 min in cold water and then 5 min in hot (60 °C) water and were then oxidised by treatment in 300 cm³ capacity, sealed, stainless steel dyepots housed in a *Roaches Pyrotec S* infrared dyeing machine, using

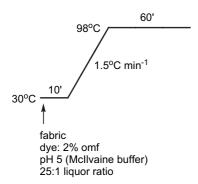


Fig. 2. Dyeing method for acid dyes.

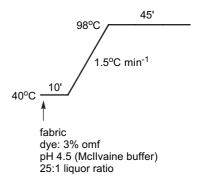


Fig. 3. Dyeing method for reactive dyes.

a liquor ratio of 50:1, at 60 °C in a bath containing $2 \, \mathrm{g} \, \mathrm{l}^{-1} \, 35\% \, \mathrm{w/w} \, \mathrm{H}_2\mathrm{O}_2$ at pH 4.5 (McIlvaine buffer) for 15 min. At the end of oxidation, samples were rinsed in running cold water for 5 min and allowed to dry in the open air.

2.3.4. Sulphur dyes

Dyeings of 10% omf were carried out using the method shown in Fig. 5 at pH 7.0 (McIlvaine buffer). Dyeings were oxidised using 5 g l⁻¹ *Diresul BRI* at pH 4.5 (McIlvaine buffer) using a liquor ratio of 50:1 at 60 °C for 15 min in 300 cm³ capacity, sealed, stainless steel dyepots housed in a *Roaches Pyrotec S* infrared dyeing machine. At the end of the oxidation the samples were rinsed in running cold water for 5 min and allowed to dry in the open air.

2.4. Colour measurement

All measurements were carried out using the equipment and procedure described earlier [2,6].

2.5. Wash fastness

The dyed fabrics were subjected to five consecutive ISO CO6/C2 wash tests as described earlier [2,6].

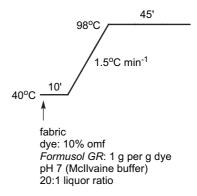


Fig. 4. Dyeing method for solubilised sulphur dyes.

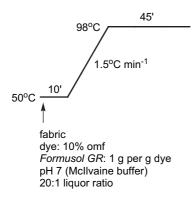


Fig. 5. Dyeing method for sulphur dyes.

3. Results and discussion

The substantivity of anionic dyes towards nylon fibres can be modified by altering the concentration of amino end groups in the substrate. By increasing the AEG content, dye-fibre substantivity is enhanced; such anionic-dyeable variants are commonly referred to as low, standard, deep or ultra-deep, depending on AEG content [1]. In contrast, the introduction of anionic groups, such as carboxylic or sulphonic acid groups, into the substrate lowers the substantivity of nylon fibres towards anionic dyes; the resulting cationic-dyeable fibres also display substantivity towards cationic dyes [1]. For this work, it was also decided to further reduce the AEG content of the cationic-dyeable variant and thus secure a modified fibre which contained a very low AEG through acetylation. Thus, in this work, four nylon 6,6 variants were selected:

standard dyeing: AEG 45 meq kg⁻¹;
deep dyeing: AEG 70 meq kg⁻¹;
cationic-dyeable: AEG 15 meq kg⁻¹;
acetylated: AEG 2.3 meq kg⁻¹.

The polymer used for the cationic-dyeable variant (and, thus, that of the acetylated fabric) differed to that employed for the standard and deep dyeing types of fibre [7].

Fig. 6 shows the colour strength (f(k)) secured for the three acid dyes on each of the four nylon 6,6 variants. In the case of C.I. Acid Yellow 236, colour strength decreased in the order:

deep dyeable>standard dyeable≫cationic dyeable > acetylated.

Similar results were observed when the four types of nylon 6,6 fabric had been dyed using C.I. Acid Black 194 and *Nylosan Red FGS* (Fig. 6). This finding, that colour strength decreased with decreasing AEG content of the four types of nylon 6,6 fabric, supports the widely accepted mechanism of dyeing nylon 6,6 with anionic

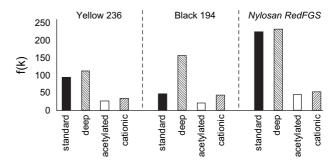


Fig. 6. Colour strength obtained for acid dyes.

dyes recounted above, namely that dye-fibre substantivity occurs predominantly via ion—ion forces operating between anionic groups in the dye and the protonated terminal amino groups in the fibre. However, although Fig. 6 shows that, for each of the three dyes used, the highest colour strength was obtained on the nylon 6,6 substrate which had the highest AEG content (i.e. deep dyeable), it is evident that with the exception of C.I. Acid Black 194, there was little difference in colour strength between deep-dyeable and standard-dyeable fibre. Although the AEG contents of deep-dyeable and standard-dyeable fabrics were 70 meq kg⁻¹ and 45 meq kg⁻¹, respectively, this being a difference of some 55%, the observed difference in colour strength achieved for C.I. Acid Yellow 236 and Nylosan Red FGS on the two types of nylon 6,6 fabrics was, clearly, lower than 55%. The similarity in colour strength observed for the deep- and standard-dyeable variants may be attributed to the overdyeing [1] of the standard-dyeable fibre as a result of the adsorption of dye via, for example, H-bonding, dispersion forces and polar van der Waals' forces. Table 3 shows the colorimetric data obtained for the three acid dyes on the nylon 6.6 variants from which it is evident that the standard-dyeable and deep-dyeable fabrics were similar in colour for each of the dyes used; this finding was not surprising as the polymers used in the two fibre types were themselves similar [3]. It is also clear from Table 3 that the colours of the standard and deep-dyeable fabrics were different to those of the dyed cationicdyeable and acetylated fabrics. This observation was also not surprising as the polymer used in the cationicdyeable fibre (and therefore, in the acetylated fabric, which was a chemical derivative of the cationic-dyeable variant) was different to that employed in the standarddyeable and deep-dyeable fibres [3].

Thus, the results displayed in Fig. 6 and Table 3 demonstrate the site-specific nature of the adsorption of acid dyes, under acidic conditions, on nylon 6,6 and therefore support the accepted mechanism of dyeing nylon 6,6 with anionic dyes, namely that dye—fibre substantivity occurs predominantly via ion—ion forces operating between anionic groups in the dye and the protonated terminal amino groups in the fibre.

Table 3
Colorimetric parameters obtained for acid dyes

Dye	Type of	L^*	a^*	b^*	C^*	h°	λ_{max}
	nylon						
C.I. Acid Yellow 236	standard	75.5	23.1	86.0	89.0	75.0	400
	deep	75.5	25.1	87.6	91.1	74.0	
	cationic	78.3	16.5	69.5	71.4	76.6	
	acetylated	77.4	11.5	61.3	62.4	79.4	
C.I. Acid black 194	standard	43.5	-1.7	-3.4	3.8	243.4	580
	deep	26.7	-3.1	-4.9	5.8	237.7	
	cationic	44.7	-2.7	-5.4	6.0	243.4	
	acetylated	57.0	-1.4	-0.9	1.7	212.7	
Nylosan Red FGS	standard	41.1	62.7	34.6	71.6	28.9	540
	deep	41.5	63.1	33.7	71.5	28.1	
	cationic	49.9	50.9	12.2	52.3	13.5	
	acetylated	53.0	53.9	15.8	56.2	16.3	

The results in Fig. 7 show that for each of the three reactive dyes, colour strength decreased in the order:

deep dyeable > standard dyeable > cationic dyeable > acetylated

which supports the mechanism of dyeing nylon 6,6 with anionic reactive dyes, namely that when applied under acidic conditions, dye-fibre substantivity occurs predominantly through electrostatic forces operating between anionic groups in the dye and the protonated, terminal amino groups in the substrate [1,8]. Fig. 7 also shows that, for each of the three dyes used, the highest colour strength was obtained on the nylon 6,6 substrate which had the highest AEG content (i.e. deep dyeable). The corresponding colorimetric data obtained for the three reactive dyes (Table 4) reveal that the standarddyeable and deep-dyeable fabrics were of similar colour for each of the dyes used but that these were different to those of the dyed cationic-dyeable and acetylated fabrics. As discussed earlier, these findings can be attributed to the different polymers that were used in the cationic-dyeable fibre and in the deep-dyeable and standard-dyeable fibres. Hence, the results in Fig. 7 and Table 4 clearly demonstrate the site-specific nature of the adsorption of anionic reactive dyes insofar as under

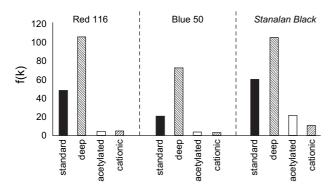


Fig. 7. Colour strength obtained for reactive dyes.

Table 4
Colorimetric parameters obtained for reactive dyes

Dye	Type of nylon	L^*	a*	<i>b</i> *	C*	h°	λ_{max}
C.I. Reactive Red 116	standard	51.3	58.4	2.3	58.4	2.3	520
	deep	46.1	65.5	8.3	66.0	7.2	
	cationic	76.2	32.2	-5.1	32.6	351.0	
	acetylated	74.0	33.9	-5.2	34.3	351.3	
C.I. Reactive Blue 50	standard	55.0	0.9	-25.2	25.2	272.0	620
	deep	49.3	3.4	-37.1	37.3	275.2	
	cationic	77.9	-3.5	-16.5	16.9	258.0	
	acetylated	78.7	-3.6	-13.9	14.4	255.5	
Stanalan Black MFFN	standard	32.4	-1.8	-3.0	3.5	239.0	680
	deep	32.0	-1.0	-3.0	3.2	251.6	
	cationic	55.8	-2.7	-2.7	3.8	225.0	
	acetylated	66.7	-4.9	1.0	5.0	168.5	

acidic conditions, dye—fibre substantivity occurs predominantly via ion—ion forces operating between anionic groups in the dye and the protonated terminal amino groups in the fibre.

Fig. 8 shows the colour strength obtained for the three solubilised sulphur dyes on each of the four nylon 6,6 variants from which it is evident that for each dye the colour strength decreased in the order:

deep dyeable > standard dyeable > acetylated > cationic dyeable.

It is also apparent that although highest colour strength was obtained on deep-dyeable nylon 6,6, there was comparatively little difference in colour strength between the four variants; this was especially apparent in the case of C.I. Solubilised Sulphur Brown 10. These findings can be explained in terms of the nature of the fibre and the dyes under the particular dyeing conditions used. The three dyes were applied at a pH of 7 because it had been earlier found [6] that, for five commercial sulphur dyes, maximum colour strength on nylon 6,6 was achieved at this particular pH. In the context of nylon 6,6, as protonation of the terminal amino groups is an equilibrium situation (1) in which the position of the equilibrium is pH dependent, it follows that the equilibrium depicted in Eq. (1) will move to the left with increasing pH.

$$-NH_2 + H^+ \rightleftharpoons -^+ NH_3 \tag{1}$$

As it is considered [9] that when dyeing at 95 °C, the pK_a of the amine conjugate acid $(-CH_2-)_6-^+NH_3$ in nylon 6,6 will be about 8, it follows that under the particular conditions that had been used to apply the solubilised sulphur dyes in this work (pH 7 at 98 °C), the equilibrium shown above (1) would be situated well to the left and there would be few protonated amino end groups present in the substrate. As the three dyes would be in their reduced, *anionic* thiol form during dyeing, the

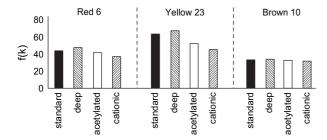


Fig. 8. Colour strength obtained for solubilised sulphur dyes.

finding (Fig. 8) that highest colour strength was obtained on deep-dyeable nylon 6,6 may be attributed to electrostatic forces operating between the anionic thiol groups in the dye and the, relatively few, protonated terminal amino groups in the substrate. However, the fact (Fig. 8) that there was little difference in colour strength between the deep-dyeable and other three dyed fibre variants implies that if such ion—ion forces of interaction were involved, their contribution to dye-fibre substantivity was not marked. Indeed, the observation that whilst highest colour strength was obtained on deep-dyeable nylon 6,6, there was little difference in colour strength observed between the four fibre variants, can be attributed to forces of interaction, such as H-bonding, dispersion forces and polar van der Waals, contributing to dye-fibre substantivity. The corresponding colorimetric data (Table 5) obtained for the three solubilised sulphur dyes show that the colour of the dyeings on the four different types of nylon 6,6 fabrics was not dissimilar. This result contrasts with that obtained for both the acid dyes and reactive dyes (Tables 3 and 4), for which a marked difference in colour was recorded between the dyed standard and deepdyeable variants and the cationic-dyeable and acetylated fibre types, which was attributed to the different polymers that were used in the latter two fibre variants. Clearly, the fact that the colour of the dyeings on each of the four types of fibre was similar (Fig. 8) indicating that the nature

Table 5
Colorimetric parameters obtained for solubilised sulphur dyes

Dye	Type of nylon	L^*	a*	b*	C*	h°	λ_{\max}
C.I. Sol. Sulphur	standard	45.5	17.0	6.1	19.6	20.6	460
Red 6	deep	44.9	17.7	6.9	19.0	21.3	
	cationic	48.5	17.2	5.5	18.1	17.7	
	acetylated	47.0	17.2	7.0	18.6	22.1	
C.I. Sol. Sulphur	standard	68.9	8.1	61.6	62.1	82.5	400
Yellow 23	deep	69.0	9.0	62.9	63.5	81.9	
	cationic	65.3	6.2	49.3	49.7	82.8	
	acetylated	69.6	8.5	59.1	59.7	81.8	
C.I. Sol. Sulphur	standard	65.0	12.1	43.4	45.1	74.4	400
Brown 10	deep	64.3	10.5	43.0	44.3	76.3	
	cationic	66.0	13.4	4.4	46.4	73.2	
	acetylated	64.9	13.3	2.7	44.7	72.7	

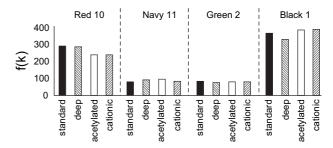


Fig. 9. Colour strength obtained for sulphur dyes.

of the interactions between the solubilised sulphur dyes and the different fabrics was little influenced by the nature of the polymer. Hence, the results in Fig. 8 and Table 5 imply that the adsorption of the solubilised sulphur dyes was predominantly non-site-specific and, therefore, that dye—fibre substantivity occurs principally through H-bonding, dispersion forces and polar van der Waals' forces of interaction.

The results in Fig. 9 show that for each of four sulphur dyes, there was little difference in colour strength achieved between the four types of fibre and, also, that highest colour strength was not obtained on deep-dyeable nylon 6,6. Although these results contrast markedly with those secured for the acid and reactive dyes (Figs. 6 and 7), they are in accord with those obtained for the three solubilised sulphur dyes (Fig. 8) and, therefore, it can be proposed that non-specific forces of interaction, such as H-bonding, dispersion forces and polar van der Waals, contributed mainly to dye-fibre substantivity. The colorimetric data displayed in Table 6 clearly show that dyeings of similar colour were achieved on the four nylon 6,6 fabric variants. This finding agrees with that secured for the three solubilised sulphur dyes (Table 5) and, as proposed for this latter

Table 6
Colorimetric parameters obtained for sulphur dyes

Dye	Type of nylon	L^*	a^*	b^*	C^*	h°	λ_{max}
C.I. Sulphur	standard	20.6	20.7	3.8	21.0	10.4	520
Red 10	deep	20.7	20.0	3.4	20.3	9.6	
	cationic	22.6	19.5	3.6	19.8	10.5	
	acetylated	22.6	20.4	3.0	20.6	9.4	
C.I. Sulphur	standard	34.9	-1.2	-14.6	14.6	265.3	600
Navy 11	deep	33.4	-0.9	-14.5	14.5	266.4	
	cationic	32.1	-0.4	-14.0	14.0	268.4	
	acetylated	34.1	-0.6	-14.0	14.0	267.5	
C.I. Sulphur	standard	35.1	-18.1	-8.7	20.1	205.7	640
Green 2	deep	39.5	-17.0	-9.8	19.6	210.0	
	cationic	37.3	-16.2	-8.4	18.2	207.4	
	acetylated	39.2	-16.0	-9.2	18.5	209.9	
C.I. Sulphur	standard	15.6	-1.3	-0.9	1.6	214.7	620
Black 1	deep	16.7	-1.1	-1.4	1.8	213.8	
	cationic	16.2	-1.2	-1.0	1.6	219.8	
	acetylated	15.8	-1.5	-1.3	2.0	220.9	

dye type, implies that the nature of the interactions between the four sulphur dyes and the different fabrics was little influenced by the nature of the polymer. Thus, the results in Fig. 9 and Table 6 indicate that the adsorption of the sulphur dyes occurred predominantly via non-site-specific forces such as H-bonding, dispersion forces and polar van der Waals' forces of interaction.

4. Conclusions

The findings of this preliminary study suggest that the extent of adsorption of both solubilised sulphur and sulphur dyes onto the four different types of nylon 6,6 fabric was little influenced by either the AEG content of the fibre or the nature of the polymer from which the fabric had been made. In addition, the findings that the colour of dyeings on each of the four types of fibre was not dissimilar also indicates that the interactions between both solubilised sulphur dyes as well as sulphur dyes and the different fabrics was little influenced by the nature of the polymer. Thus, it appears that the

adsorption of both solubilised sulphur dyes and sulphur dyes onto nylon 6,6 is predominantly non-site-specific and, therefore, that dye—fibre substantivity occurs principally through H-bonding, dispersion forces and polar van der Waals' forces of interaction.

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