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The dyeing of poly(lactic acid) fibres with disperse dyes using ultrasound: Part 2 – Fastness

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ABSTRACT

The use of ultrasound imparted greater colour strength to 1% omf dyeings of five of six disperse dyes studied, both before and after reduction clearing and had no effect on either the colour or λ_{max} of the dyeings. The lower fastness obtained to both wet and dry rubbing as well as to the first two/three of the five repeated wash cycles was attributed to the greater colour strength of the dyeings imparted by the use of ultrasound during dyeing.

1. Introduction

Although poly(lactic acid) (PLA) is dyeable with disperse dyes, the greater hydrolytic sensitivity and lower glass transition temperature (T_g) of the substrate dictates that lower temperatures are employed for its dyeing with disperse dyes than its more famous polyester counterpart, poly(ethylene terephthalate) (PET) [1,2]. Disperse dyes generally display lower exhaustion on PLA than PET [1,3,4], yield brighter dyeings [1] of greater colour strength [1,3] and with λ_{max} occurring at shorter wavelengths than on PET [1]. The generally low uptake of disperse dyes on PLA fibres compared to PET has been interpreted in terms of dye structure [5,6] and has prompted the synthesis of specific disperse dyes [4,7–9].

The first part of this paper [10] showed that ultrasound enhanced the colour strength obtained for three disperse dyes on PLA fabric at temperatures upto 70 °C, this being attributed to dye disaggregation. However, ultrasound did not always result in enhanced colour strength being achieved in the case of three other disperse dyes. Dyeing at 80 °C in the presence of ultrasound resulted in pale, dull dyeings of reduced colour strength, which was attributed to breakdown of the dye dispersions at this particular temperature. This part of the paper concerns the effect of ultrasound on the fastness of 1% omf dyeings obtained at 70 °C to both repeated washing and rubbing.

2. Experimental

2.1. Materials

The scoured, knitted PLA fabric (224.8 g m^{-2}) obtained from NatureWorks LLC described previously [10] was used. Commercial samples of the six disperse dyes shown in Table 1 were used without purification; the structures of only four of the dyes (C.I. Disperse Red 60 (**I**), C.I. Disperse Blue 56 (**II**), C.I. Disperse Yellow 42 (**III**) and Disperse C.I. Red 167:1(**IV**) (Fig. 1)) are disclosed in the Colour Index [11]. The dyes were selected for use on the basis that they provided two representatives of low, medium and high energy classes of disperse dye (Table 1). All other chemicals were of general laboratory grade supplied by Aldrich.

2.2. Dyeing

1% omf depths of shade were produced, using the equipment described earlier [10] following the method shown in Fig. 2; the pH was the pH was maintained at 4.5 using acetic acid/sodium acetate buffer. At the end of dyeing, the dyed samples were removed, rinsed in tap water and allowed to dry in the open air.

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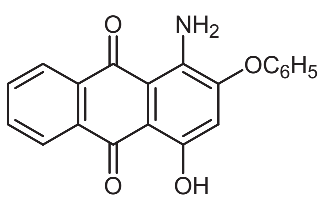
Table 1
Dyes used.

Commercial name	C.I. Disperse	Energy level	Supplier
Foron Brilliant Red E-2BL 200	Red 60	Low	Clariant
Foron Blue E-BL 200	Blue 56	Low	
Foron Yellow SE-FL	Yellow 42	Medium	
Foron Rubine S-GFL 150	Red 167:1	High	
Dianix Yellow Brown CC	None ascribed	Medium	DyStar
Dianix Crimson SF	None ascribed	High	

2.3. Reduction clearing

The dyeing was rinsed in warm water (50 °C) and treated in a solution comprising 2 gl^{-1} $Na_2S_2O_4$ and 1.5 gl^{-1} Na_2CO_3 at 60 °C employing a 50:1 liquor ratio. The reduction cleared sample was rinsed thoroughly in water and allowed to dry in the open air.



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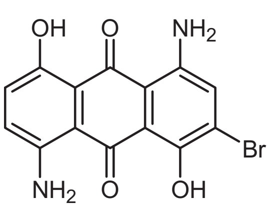

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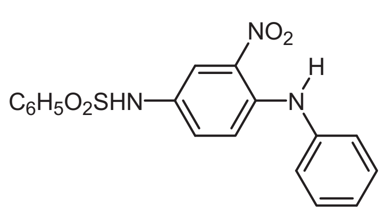

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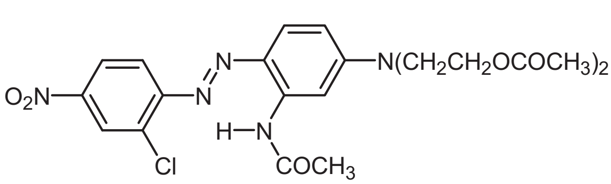

IV

Fig. 1. Dye structures.

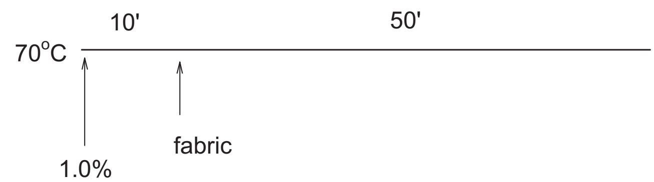

Fig. 2. Dyeing method.

2.4. Wash-fastness

The ISO CO6/B2S (50 °C) test method [12] was used but was modified in that dyeings were subjected to five, consecutive wash tests and, at the end of each wash test, the washed sample was rinsed thoroughly in tap water (but was not dried) and a fresh sample of SDC multifibre strip was used to assess the extent of staining for each of the five wash tests.

2.5. Rub fastness

The ISO 105:X12 test method [12] was employed to determine both the with wet and dry rub fastness of the dyeing.

3. Results and discussion

The six dyes were selected for use on the basis that they provided two representatives of low, medium and high energy classes of disperse dye (Table 1). A 1% omf depth of shade was used as this provided a typical pale depth dyeing; a repeated wash fastness protocol was employed, rather than a single wash test, as it was considered to more accurately reflect the progressive nature of the removal of dye and the redeposition of vagrant dye that occurs during domestic washing.

3.1. Fastness

The fastness to the ISO CO6/B2S (50 °C) test method [12] of the 1% omf dyeings, carried out in both the presence and absence of ultrasound, for each of the six dyes under consideration, are shown in Tables 2–7. For ease of discussion, the results obtained for C.I. Disperse Blue 56 (displayed in Table 2) will be used as an exemplar.

It is evident that the dyeing obtained in the absence of ultrasound displayed much lower fastness to repeated washing at 50 °C prior to reduction clearing than the corresponding dyeings which had received a reduction clear treatment, as measured in terms of both the change in shade that occurred as a result of repeated washing and the levels of staining of the adjacent multifibre strip imparted by vagrant dye which had been removed from the dyeing during washing. A reduction clear treatment is routinely given to disperse dyeings on hydrophobic fibres such as PET and PLA to remove surplus dye and auxiliaries so as to ensure that optimum fastness is achieved. As reduction clearing is designed to remove surface dye from dyeings, it was expected that the fastness of the reduction cleared dyeings would be higher than that of their non-reduction cleared counterparts. From Table 2, it is also apparent in the case of the dyeings obtained before reduction clearing in the absence of ultrasound, that the extent of staining achieved at the end of the first wash test was much greater than that obtained at the end of the second wash test and that the level of staining levelled thereafter. These findings are due to dye having been removed from the dyeings during washing. In this context, the greatest extent of staining was obtained for the adjacent nylon 6,6 multifibre strip material, which was expected bearing in mind the very well-known proclivity for this type of polymer to absorb vagrant

Table 2
Fastness of C.I. Disperse Blue 56 dyeings.

Dyeing	Treatment	No. of washes	Change in shade	Diacetate	Cotton	Nylon	Polyester	Acrylic	Wool
Absence of ultrasound	Before reduction clear	1	3	2–3	4–5	1–2	3–4	5	4–5
		2	3	4–5	5	3–4	5	5	5
		3	2–3	5	5	4	5	5	5
		4	2–3	5	5	4–5	5	5	5
		5	2–3	5	5	4–5	5	5	5
	After reduction clear	1	4	4–5	5	4	4–5	5	5
		2	4	5	5	4–5	5	5	5
		3	3–4	5	5	4–5	5	5	5
		4	3–4	5	5	4–5	5	5	5
		5	3–4	5	5	4–5	5	5	5
Presence of ultrasound	Before reduction clear	1	3	2	4	1	3	5	4–5
		2	3	4–5	5	3–4	4–5	5	5
		3	2–3	5	5	4	5	5	5
		4	2–3	5	5	4–5	5	5	5
		5	2–3	5	5	4–5	5	5	5
	After reduction clear	1	4	4	5	3–4	4–5	5	5
		2	4	4–5	5	4–5	5	5	5
		3	3–4	5	5	4–5	5	5	5
		4	3–4	5	5	4–5	5	5	5
		5	3–4	5	5	4–5	5	5	5

Table 3
Fastness of C.I. Disperse Red 60 dyeings.

Dyeing	Treatment	No. of washes	Change in shade	Diacetate	Cotton	Nylon	Polyester	Acrylic	Wool
Absence of ultrasound	Before reduction clear	1	4	3–4	5	2	4	5	4–5
		2	3–4	4–5	5	4–5	5	5	5
		3	3–4	5	5	4–5	5	5	5
		4	3–4	5	5	5	5	5	5
		5	3–4	5	5	5	5	5	5
	After reduction clear	1	4	5	5	4–5	5	5	5
		2	4	5	5	5	5	5	5
		3	4	5	5	5	5	5	5
		4	4	5	5	5	5	5	5
		5	4	5	5	5	5	5	5
Presence of ultrasound	Before reduction clear	1	4	2	5	1	3–4	5	4–5
		2	4	4–5	5	4	5	5	5
		3	4	5	5	4–5	5	5	5
		4	4	5	5	5	5	5	5
		5	4	5	5	5	5	5	5
	After reduction clear	1	4–5	4–5	5	4	5	5	5
		2	4–5	5	5	4–5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5

Table 4
Fastness of C.I. Disperse Yellow 42 dyeings.

Dyeing	Treatment	No. of washes	Change in shade	Diacetate	Cotton	Nylon	Polyester	Acrylic	Wool
Absence of ultrasound	Before reduction clear	1	3	3–4	4	1–2	4	5	5
		2	2–3	4–5	5	4–5	5	5	5
		3	2	5	5	5	5	5	5
		4	2	5	5	5	5	5	5
		5	2	5	5	5	5	5	5
	After reduction clear	1	4	5	5	4–5	5	5	5
		2	3–4	5	5	5	5	5	5
		3	3–4	5	5	5	5	5	5
		4	3–4	5	5	5	5	5	5
		5	3–4	5	5	5	5	5	5
Presence of ultrasound	Before reduction clear	1	3–4	3–4	3–4	1	4	5	5
		2	3	4–5	5	4–5	5	5	5
		3	3	5	5	5	5	5	5
		4	3	5	5	5	5	5	5
		5	3	5	5	5	5	5	5
	After reduction clear	1	4–5	5	5	3–4	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5

Table 5
Fastness of Dianix Yellow Brown CC dyeings.

Dyeing	Treatment	No. of washes	Change in shade	Diacetate	Cotton	Nylon	Polyester	Acrylic	Wool
Absence of ultrasound	Before reduction clear	1	3–4	2–3	5	3	4–5	5	5
		2	3	4–5	5	5	5	5	5
		3	3	5	5	5	5	5	5
		4	3	5	5	5	5	5	5
		5	3	5	5	5	5	5	5
	After reduction clear	1	4–5	5	5	5	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5
Presence of ultrasound	Before reduction clear	1	3–4	2	5	2–3	4	5	5
		2	3	4–5	5	5	5	5	5
		3	3	5	5	5	5	5	5
		4	3	5	5	5	5	5	5
		5	3	5	5	5	5	5	5
	After reduction clear	1	4–5	4–5	5	4-5	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5

Table 6
Fastness of C.I. Disperse Red 167:1 dyeings.

Dyeing	Treatment	No. of washes	Change in shade	Diacetate	Cotton	Nylon	Polyester	Acrylic	Wool
Absence of ultrasound	Before reduction clear	1	3	4	5	2–3	4	5	5
		2	2–3	5	5	5	5	5	5
		3	2–3	5	5	5	5	5	5
		4	2–3	5	5	5	5	5	5
		5	2–3	5	5	5	5	5	5
	After reduction clear	1	4	5	5	5	5	5	5
		2	4	5	5	5	5	5	5
		3	4	5	5	5	5	5	5
		4	4	5	5	5	5	5	5
		5	4	5	5	5	5	5	5
Presence of ultrasound	Before reduction clear	1	3–4	3–4	5	2	3–4	5	5
		2	4	5	5	5	5	5	5
		3	4	5	5	5	5	5	5
		4	4	5	5	5	5	5	5
		5	4	5	5	5	5	5	5
	After reduction clear	1	4	5	5	4–5	5	5	5
		2	4	5	5	5	5	5	5
		3	4	5	5	5	5	5	5
		4	4	5	5	5	5	5	5
		5	4	5	5	5	5	5	5

Table 7
Fastness of Dianix Crimson SF dyeings.

Dyeing	Treatment	No. of washes	Change in shade	Diacetate	Cotton	Nylon	Polyester	Acrylic	Wool
Absence of ultrasound	Before reduction clear	1	4–5	5	5	5	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5
	After reduction clear	1	4–5	5	5	5	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5
Presence of ultrasound	Before reduction clear	1	4–5	4–5	5	4–5	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5
	After reduction clear	1	4–5	5	5	5	5	5	5
		2	4–5	5	5	5	5	5	5
		3	4–5	5	5	5	5	5	5
		4	4–5	5	5	5	5	5	5
		5	4–5	5	5	5	5	5	5

Table 8
Rub fastness of 1% omf dyeings.

Dyeing	Treatment	Dry		Wet		Dry		Wet		Dry		Wet	
		Staining	Shade change	Staining	Shade change	Staining	Shade change	Staining	Shade change	Staining	Shade change	Staining	Shade change
		C.I. Disperse Blue 56				C.I. Disperse Red 60				C.I. Disperse Yellow 42			
Absence of ultrasound	Before reduction clear	4	4	5	4–5	4–5	4	5	4–5	4–5	4	5	4–5
	After reduction clear	4–5	4–5	5	4–5	5	4–5	5	4–5	5	4–5	5	4–5
Presence of ultrasound	Before reduction clear	3–4	4	5	4–5	4	4	5	4–5	4	4	5	4–5
	After reduction clear	4–5	4–5	5	4–5	4–5	4–5	5	4–5	4–5	4	5	4–5
		Dianix Yellow Brown CC				C.I. Disperse Red 167:1				Dianix Crimson SF			
Absence of ultrasound	Before reduction clear	4	4	5	4–5	4–5	4	5	4–5	5	4–5	5	4–5
	After reduction clear	4–5	4	5	4–5	5	4–5	5	4–5	5	4–5	5	4–5
Presence of ultrasound	Before reduction clear	3–4	3–4	5	4–5	4	4	5	4–5	5	4–5	5	4–5
	After reduction clear	4	4	5	4–5	4–5	4	5	4–5	5	4–5	5	4–5

dye during laundering [13]. The moderate level of staining observed for diacetate and polyester strip materials can be attributed to the low substantivity of the vagrant disperse dye towards these two types of fibre at the relatively low washing temperature involved (50 °C). The small extent of staining of both the wool and cotton multifibre strip materials can be ascribed to their polar, absorbent nature whilst the lack of staining of the acrylic strip material is attributable to its lack of substantivity for disperse dyes at temperatures below its T_g. Clearly, reduction clearing markedly improved the fastness of the 1% omf dyeings of C.I. Disperse Blue 56 (Table 2) in terms of both shade change and staining of adjacent materials; this can be attributed to reduction clearing having removed surplus dye from the dyeings.

Similar trends in fastness were obtained for the dyeings which had been carried out in the presence of ultrasound as those which had been undertaken in the absence of ultrasound, namely, the fastness of the dyeings before reduction clearing was lower than that of their reduction cleared counterparts and the extent of staining achieved at the end of the first wash test was much greater than that obtained at the end of the second wash test; in addition, the greatest extent of staining was observed for the nylon adjacent material. The dyeings after reduction clearing displayed much better wash fastness in terms of both shade change and staining of

Table 9
Colourimetric data for C.I. Disperse Blue 56 dyeings.

Dyeing	Treatment	No. of washes	L^*	a^*	b^*	C^*	$h°$	f_k
Absence of ultrasound	Before reduction	0	71.9	−2.3	−21.3	21.4	263.9	4.5
	clear	5	76.0	−1.9	−22.8	22.9	265.3	3.0
	After reduction	0	75.2	−2.9	−20.3	20.5	261.8	3.3
	clear	5	76.2	−1.9	−22.7	22.8	265.1	2.9
Presence of ultrasound	Before reduction	0	70.8	−2.3	−21.4	21.6	263.9	4.9
	clear	5	75.2	−1.8	−23.4	23.5	265.6	3.2
	After reduction	0	78.8	32.2	1.3	32.2	2.3	2.9
	clear	5	80.0	32.0	0.7	32.0	1.3	2.5

Table 10
Colorimetric data for C.I. Disperse Red 60 dyeings.

Dyeing	Treatment	No. of washes	L^*	a^*	b^*	C^*	$h°$	f_k
Absence of ultrasound	Before reduction	0	78.2	32.1	0.9	32.1	1.5	3.0
	clear	5	80.4	31.3	0.6	31.3	1.1	2.4
	After reduction	0	80.4	31.2	0.9	30.2	1.7	2.4
	clear	5	81.0	30.1	0.4	30.1	0.8	2.2
Presence of ultrasound	Before reduction	0	77.9	32.7	1.0	32.7	1.8	3.1
	clear	5	79.5	32.3	0.8	32.3	1.4	2.7
	After reduction	0	79.3	31.7	1.1	31.7	1.9	2.7
	clear	5	80.0	32.0	0.7	32.0	1.3	2.5

Table 11
Colourimetric data for C.I. Disperse Yellow 42 dyeings.

Dyeing	Treatment	No. of washes	L^*	a^*	b^*	C^*	$h°$	f_k
Absence of ultrasound	Before reduction	0	84.5	10.9	28.2	30.2	68.9	3.5
	clear	5	88.2	7.0	25.4	26.3	74.6	2.2
	After reduction	0	87.6	7.3	25.5	26.6	74.1	2.3
	clear	5	88.5	6.8	25.2	26.1	74.9	2.1
Presence of ultrasound	Before reduction	0	84.6	10.5	28.0	29.9	69.5	3.4
	clear	5	87.8	7.9	27.2	28.3	73.9	2.4
	After reduction	0	87.2	7.5	26.3	27.4	74.1	2.5
	clear	5	87.7	7.6	26.8	27.9	74.2	2.4

Table 12
Colourimetric data for Dianix Yellow Brown CC dyeings.

Dyeing	Treatment	No. of washes	L^*	a^*	b^*	C^*	$h°$	f_k
Absence of ultrasound	Before reduction	0	81.2	15.1	40.8	43.5	69.7	6.8
	clear	5	83.8	13.5	40.2	42.4	71.5	5.4
	After reduction	0	84.0	12.0	39.2	41.0	73.0	5.2
	clear	5	84.7	12.2	38.8	40.7	72.6	4.8
Presence of ultrasound	Before reduction	0	79.9	17.0	43.0	46.3	68.4	8.1
	clear	5	82.8	14.7	42.4	44.9	70.9	6.4
	After reduction	0	82.8	13.8	40.2	42.3	71.0	5.9
	clear	5	83.8	13.5	40.5	42.7	71.6	5.5

Table 13
Colourimetric data for C.I. Disperse Red 167:1 dyeings.

Dyeing	Treatment	No. of washes	L^*	a^*	b^*	C^*	$h°$	f_k
Absence of ultrasound	Before reduction	0	75.1	28.4	6.9	29.2	13.6	4.3
	clear	5	79.7	26.2	5.1	26.7	11.0	2.7
	After reduction	0	79.6	24.9	5.7	25.6	12.8	2.7
	clear	5	80.3	25.7	5.2	26.2	11.3	2.5
Presence of ultrasound	Before reduction	0	73.2	30.1	7.2	30.9	13.4	5.2
	clear	5	77.6	28.8	6.3	29.5	12.3	3.4
	After reduction	0	77.5	27.2	6.6	27.9	13.6	3.4
	clear	5	78.5	28.0	5.9	28.6	11.8	3.1

Table 14
Colourimetric data for Dianix Crimson SF dyeings.

Dyeing	Treatment	No. of washes	L*	a*	b*	C*	h°	f_k
Absence of ultrasound	Before reduction clear	0	85.0	22.5	11.9	25.4	27.9	1.9
		5	86.3	22.2	11.7	25.2	27.8	1.6
	After reduction clear	0	85.9	21.8	11.7	24.7	28.2	1.7
		5	86.7	21.5	11.1	24.2	27.4	1.5
Presence of ultrasound	Before reduction clear	0	85.4	21.6	11.3	24.4	27.5	1.7
		5	86.5	21.5	10.8	24.1	26.6	1.5
	After reduction clear	0	85.9	22.1	11.7	25.0	27.8	1.7
		5	86.8	21.6	11.1	24.3	27.1	1.5

adjacent materials (Table 2). However, a comparison of the staining results presented in Table 2 for the absence and presence of ultrasound clearly show that the dyeings which had been carried out in the presence of ultrasound were 0.5–1 units lower than the corresponding dyeings which had been carried out in the absence of ultrasound, in the cases of the first two or three wash tests.

Similar findings, in terms of trends in fastness for dyeings which had been carried out before and after reduction clearing, in both the presence and presence of ultrasound were obtained for each of the other five dyes used in this work (Tables 3–7). In addition, for all dyes except Dianix Crimson SF, the staining results obtained

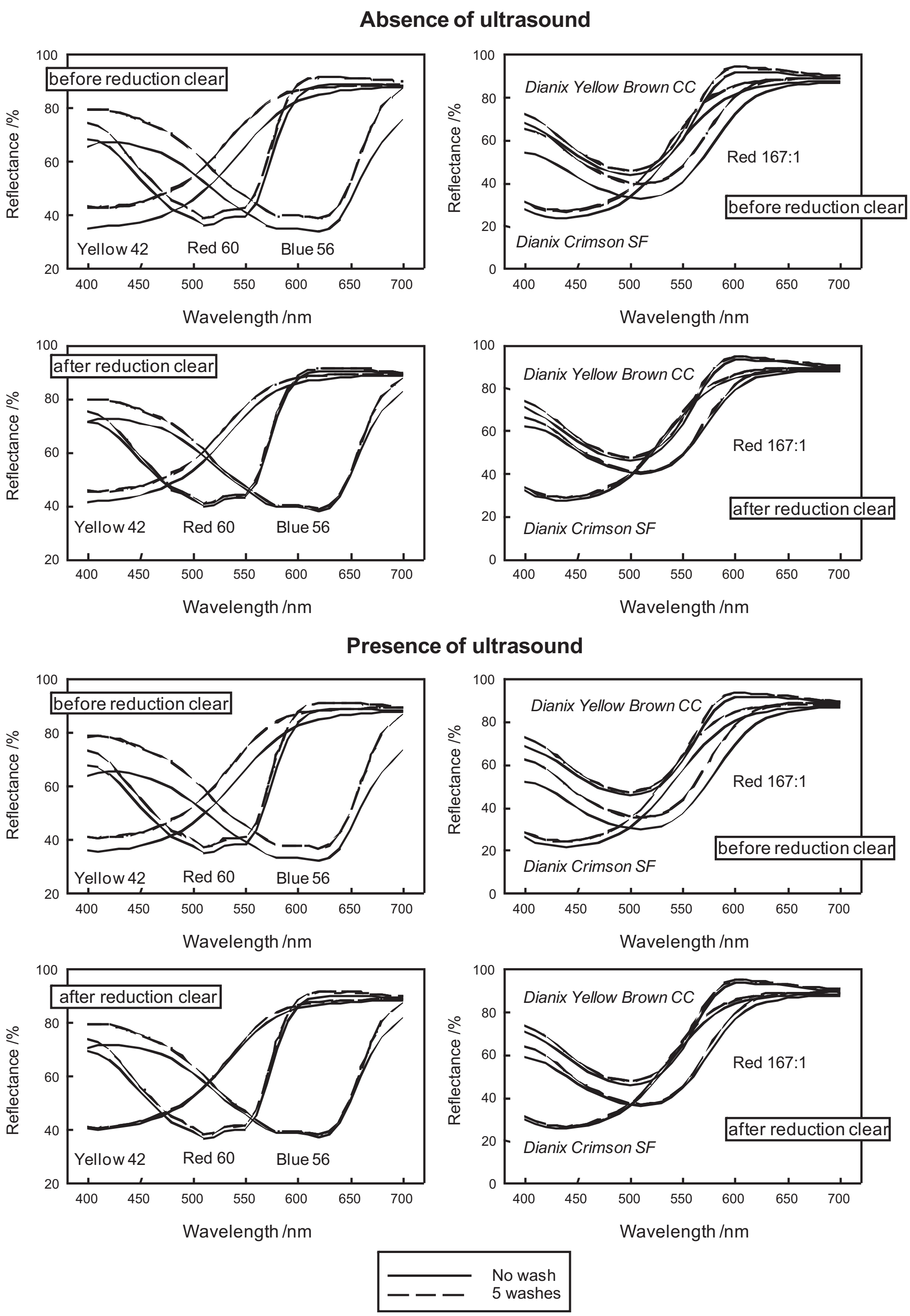

Fig. 3. Spectrophotometric plots of the dyeings.

(Tables 3–6) in the absence and presence of ultrasound revealed that the dyeings which had been carried out in the presence of ultrasound were generally 0.5–1 units lower than the corresponding dyeings which had been carried out in the absence of ultrasound, in the cases of the first two or three wash tests. In the case of Dianix Crimson SF, the finding that there was no difference between the fastness ratings secured for the dyeings which had been carried out in the absence and presence of ultrasound was attributed to the low colour strength of the dyeings (Table 14). Table 8 shows the wet and dry rub fastness results obtained for the 1% omf dyeings from which it is evident that the reduction cleared dyeings were, as expected, of higher fastness, when dyeing had been carried out in both the presence and absence of ultrasound, since surplus dye had been removed from the dyeing surface by treatment with the aq alkaline dithionite solution. Again, the fastness of the dyeings which had been undertaken in the presence of ultrasound were generally 0.5 units lower in fastness than those which had been carried out in the absence of ultrasound, with the exception of Dianix Crimson SF, the latter exception being attributable to the low colour strength of the dyeings. The possible causes for the observed lower fastness of the ultrasound dyeings were sought.

3.2. Colourimetric and spectrophotometric data

Tables 9–14 show the colourimetric data obtained for the 1% omf dyeings of the six dyes used, which had been carried out in the absence and presence and ultrasound, both before and after reduction clearing. Firstly, the marked effect which repeated washing had upon the colour of the dyeings is clearly evident; the higher L^* values and lower f_k values reflect dye which had been removed from the dyeings during wash testing. The use of ultrasound generally resulted in slightly higher colour strength, both before and after reduction clearing, with the exception of Dianix Crimson SF which appears to explain why the dyeings, using the other five dyes, obtained in the presence of ultrasound, displayed lower fastness to repeated wash testing (Tables 3–6) and rubbing (Table 8). Ultrasound had no effect on either the colour (Tables 3–7) or λ_{max} of the dyeings (Fig. 3) with the exception of C.I. Disperse Blue 56, insofar as, the reduction cleared dyeings were of much different hue to the dyeings which had been carried out in the absence of ultrasound, even though the λ_{max} of the dyeings was unchanged (Fig. 3). No reason can be given for this anomalous finding in the case of C.I. Disperse Blue 56.

4. Conclusions

The use of ultrasound imparted greater colour strength to the 1% omf dyeings both before and after reduction clearing in the case of five of the six dyes used, the exception being Dianix Crimson SX which produced dyeings of only very low f_k values. Ultrasound had no effect on neither the colour nor the λ_{max} of the dyeings, with the exception of C.I. Disperse Blue 56, insofar as, the reduction cleared dyeings were of much different hue to the dyeings which had been carried out in the absence of ultrasound, even though the λ_{max} of the dyeings obtained using C.I. Disperse Blue 56 was unchanged; no explanation for this anomalous finding could be proposed. The observed lower fastness obtained for five of the six dyes used (with the exception of Dianix Crimson SX) to both wet and dry rubbing as well as to the first two/three of the five repeated wash cycles can be attributed to the greater colour strength of the dyeings imparted by the use of ultrasound during dyeing.

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