



The dyeing of poly(lactic acid) fibres with disperse dyes using ultrasound: Part 1 – Initial studies

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ABSTRACT

Six disperse dyes were applied at 0.5%, 1% and 2% omf depths of shade to poly(lactic acid) fabric at 30–80 °C for 20, 50 and 90 min in both the presence and absence of ultrasound. Whilst ultrasound enhanced the colour strength obtained for three of the six disperse dyes used at temperatures up to 70 °C, ultrasound did not always result in enhanced colour strength being achieved in the case of the three other dyes. The observed intensification of colour strength was attributed to dye disaggregation. 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.

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1. Introduction

Textile fibres made from the aliphatic polyester, poly(lactic acid) (PLA), which is derived from an annually renewable resource such as corn, resemble those obtained from their more famous relative, poly(ethylene terephthalate) (PET), in that they are dyeable with disperse dyes. However, the high temperatures (125/130 °C) that are normally used for the aqueous phase dyeing of PET with disperse dyes cannot be used for PLA because of its marked hydrolytic sensitivity and lower T_m (~170 °C compared to ~250 °C for PET); hence, dyeing conditions of 110–115 °C for 15–30 min at pH 4.5–5 are recommended for poly(lactic acid) [1,2]. Disperse dyes generally behave differently on PLA fibre than on PET fibre [1,3–5], displaying lower exhaustion [1,3,6], with dyeings being brighter [1], of higher colour yield [1,3] and with λ_{max} occurring at a shorter wavelength than on PET [1]. The low sorption of disperse dyes on PLA compared to PET has been explored [7] using solubility parameter concept [8] as well as molecular modelling [9]; several researchers have sought to maximise dye uptake onto PLA through the synthesis of specific disperse dyes [6,10–12].

Ultrasound, with a frequency above the upper limit of human hearing (>20 kHz), has been widely explored as a means of

intensifying various wet textile processes, including dyeing, since ultrasound influences mass transfer processes within textile substrates by means of transient cavitation in the vicinity of the textile surface [13]. Several workers have reported that the use of ultrasound can have beneficial effects upon dyeing, resulting in lower dyeing temperatures, reduced dyeing times, increased colour yield and reduced consumption of dyeing auxiliaries. Whilst research interest has attended the dyeing of various substrates with ultrasound, including cotton [14–20], wool [21,22], silk [23,24], nylon [25–27], polyacrylonitrile [28], polyester [29–33] and leather [34–36], hitherto, the effects of ultrasound on the dyeing of poly(lactic acid) has not received attention. The purpose of this work was to determine whether ultrasound could be used to intensify the dyeability of PLA with disperse dyes and, at the same time, enable dyeings of adequate wet fastness to be obtained. This first part of the paper concerns the effects of ultrasound on the dyeing of PLA with disperse dyes; the second part of the paper will report the effects of ultrasound on the fastness of dyeings to both rubbing and repeated washing.

2. Experimental

2.1. Materials

Poly(lactic acid) knitted fabric (224.8 gm⁻²), obtained from NatureWorks LLC, was scoured using 2 gl⁻¹ Na₂CO₃ and 1 gl⁻¹

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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	

Sandozin NIN (non-ionic surfactant; Clariant) using a 20:1 liquor ratio at 60 °C for 15 min. The scoured sample was rinsed thoroughly in tap water and allowed to dry in the open air. 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 [37]. 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.

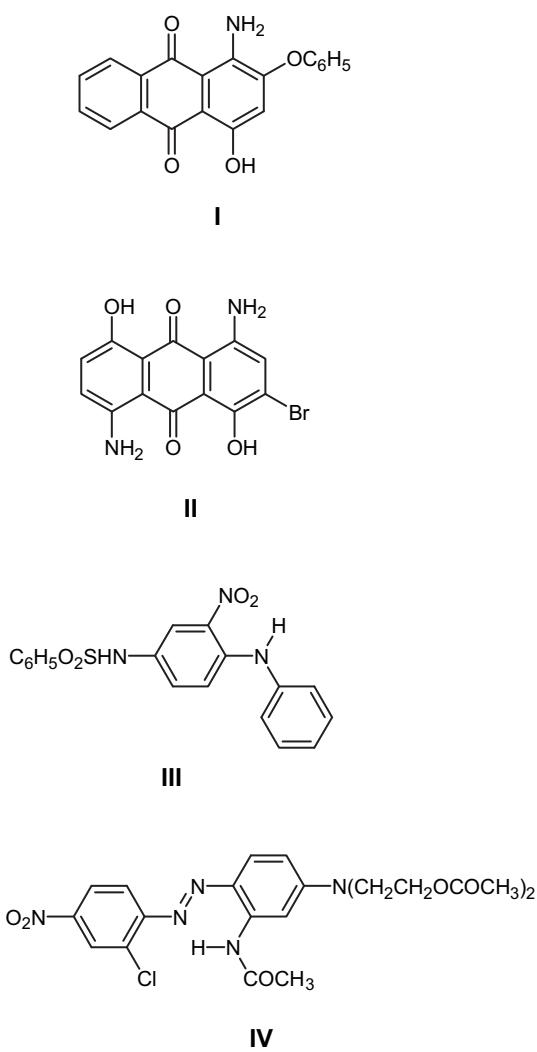


Fig. 1. Dye structures.

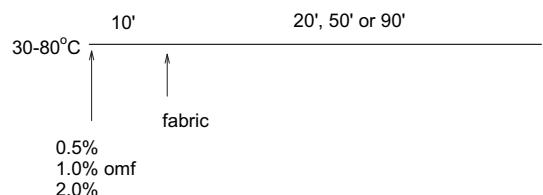


Fig. 2. Dyeing method.

2.2. Dyeing

PLA fabric was dyed at 0.5, 1.0 and 2.0% omf depths of shade, for 20, 50 and 90 min at 30, 40, 50, 60, 70 and 80 °C, in both the absence and presence of ultrasound, using a 100:1 liquor ratio in partially sealed glass dyepots of 250 cm³ capacity housed in a Grant OLS 200 laboratory scale shaker water bath using the method shown in Fig. 2; the pH was maintained at 4.5 using acetic acid/sodium acetate buffer. In the case of the ultrasound assisted dyeing, a Grant MXB 22 (32–38 kHz; 300 W) ultrasound bath was employed. At the end of dyeing, the dyed samples were removed, rinsed in tap water and allowed to dry in the open air.

2.3. Colour measurement

The CIE L^* a^* b^* C^* and h° co-ordinates and the corresponding f_K values were calculated from the reflectance values for each dyeing, obtained using a *Datacolor Spectroflash 600* spectrophotometer under illuminant D_{65} , employing a 10° standard observer with UV component included and specular component excluded. The samples were folded so as to realise four thicknesses.

3. Results and discussion

The colorimetric data for 1% omf dyeings obtained for each of the six dyes at 30–80 °C, in both the absence and absence of ultrasound, are shown in Tables 2–7; for comparison purposes, the colorimetric data for the scoured, undyed fabric is shown in Table 2. For ease of discussion, the results obtained for C.I. Disperse Blue 56 (displayed in Table 2) will be used as an exemplar. In terms of dyeing in the absence of ultrasound, Table 2 shows that, as might be expected, the depth of shade of the dyeings increased with increasing dyeing temperature, as evidenced by the decrease in lightness (L^* value) that accompanied an increase in temperature from 30 to 80 °C. This is also reflected in the corresponding colour strength (f_K values) of the dyeings (Fig. 3) from which it is evident that the increase in f_K that accompanied an increase in dyeing temperature was much

Table 2
Colorimetric data for C.I. Disperse Blue 56 (1% omf; 50 min).

Treatment	Temp./°C	L^*	a^*	b^*	C^*	h°	λ_{\max}/nm
Undyed fabric	—	95.3	-0.1	0.6	0.7	94.6	400
Absence of ultrasound	30	85.1	-1.6	-6.6	6.8	256.6	620
	40	83.1	-1.6	-8.6	8.8	259.4	620
	50	81.3	-2.1	-11.0	11.2	259.4	620
	60	76.9	-2.1	-16.4	16.5	262.6	620
	70	70.3	-1.7	-23.2	23.2	265.8	620
	80	59.8	-0.6	-32.9	32.9	271.1	620
Presence of ultrasound	30	83.4	-1.5	-7.8	7.9	259.2	620
	40	82.9	-2.0	-9.0	9.2	257.7	620
	50	80.1	-1.9	-11.9	12.1	261.2	620
	60	73.8	-1.1	-18.1	18.1	266.7	620
	70	68.9	-1.2	-24.1	24.2	267.3	620
	80	63.4	-0.7	-30.1	30.1	268.8	620

Table 3

Colorimetric data for C.I. Disperse Red 60 (1% omf; 50 min).

Treatment	Temp./°C	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	λ_{\max}/nm
Absence of ultrasound	30	91.7	5.5	0.5	5.6	5.3	520
	40	90.3	8.4	0.3	8.4	2.3	520
	50	88.2	12.7	0.0	12.7	359.8	520
	60	83.4	22.0	-0.1	22.0	359.7	520
	70	77.5	33.9	1.5	33.9	2.5	520
	80	69.2	48.7	5.9	49.0	6.6	520
Presence of ultrasound	30	86.2	10.6	-1.3	10.7	353.2	520
	40	87.0	10.6	-0.9	10.6	355.2	520
	50	86.7	14.4	-0.4	14.4	358.6	520
	60	83.4	20.8	-0.4	20.8	359.0	520
	70	77.1	34.0	1.2	34.0	2.0	520
	80	70.7	46.2	4.4	46.4	5.4	520

greater above 50 °C. This latter finding was not surprising in view of the fact, as previously mentioned, the T_g of the PLA polymer is in the region 55–65 °C [13] and it is well known that T_g is of great significance in the context of the dyeing of synthetic fibres such as PLA insofar as dyeing must be carried out at temperatures $>T_g$, since, owing to the virtual absence of segmental mobility of the component macromolecular chains at temperatures $<T_g$, little, if any, dye diffusion occurs within the substrate below this temperature. Table 2 and Fig. 3(a) show that at temperatures between 30 and 70 °C, the dyeings carried out in the presence of ultrasound were of greater depth of shade (as evidenced by lower *L** values and higher f_k values), bluer (as indicated by the higher *b** values but similar *a** values) and brighter (as shown by the higher *C** values) than those which had been undertaken in the absence of ultrasound. However, Table 2 also reveals that the dyeing obtained at 80 °C in the presence of ultrasound was paler (as shown by higher *L** values as well as lower f_k (Fig. 3)), less blue (as evidenced by the lower *b** values and almost same *a** and *h*° values), and duller (of lower chroma) than the corresponding dyeing carried out in the absence of ultrasound. Interestingly, Table 2 shows that the λ_{\max} of the dyeings was not changed by the use ultrasound at each of the six temperatures used.

In the case of the other dyes used in this work, Tables 3–7 and Fig. 3(b)–(f) show that similar findings were obtained insofar as, at temperatures between 30 and 70 °C, the dyeings carried out in the presence of ultrasound were of greater depth of shade (as evidenced by lower *L** values and higher f_k values), higher chroma and redder (as indicated by higher *h*° values) than those which had been undertaken in the absence of ultrasound. For each of the five dyes used, the λ_{\max} of the dyeings was not changed by the use ultrasound for each of the six temperatures used (Tables 3–7). Furthermore, with the exception of Dianix Crimson SF (Fig. 3(f)) and Table 7, all other dyeings obtained at 80 °C in the presence of

Table 5

Colorimetric data for Dianix Yellow Brown CC (1% omf; 50 min).

Treatment	Temp./°C	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	λ_{\max}/nm
Absence of ultrasound	30	91.3	2.8	11.4	11.7	76.5	440
	40	90.3	4.2	15.6	16.1	75.1	440
	50	88.0	6.3	22.0	22.9	73.9	440
	60	85.8	9.0	29.2	30.6	72.8	440
	70	81.1	15.8	42.1	45.0	69.4	440
	80	73.8	26.1	58.8	64.3	66.1	440
Presence of ultrasound	30	89.6	4.8	14.3	15.1	71.2	440
	40	88.7	5.7	17.0	17.9	71.4	440
	50	87.4	7.1	23.0	24.1	72.8	440
	60	85.7	9.3	29.0	30.4	72.3	440
	70	80.9	16.0	42.0	45.0	69.1	440
	80	74.9	25.0	57.6	62.8	66.5	440

ultrasound (Fig. 3(b)–(e) and Tables 3–6) were paler (as shown by higher *L** values as well as lower f_k (Fig. 3)), duller (of lower chroma) and yellower (as evidenced by lower *b** values and higher *a** values) than the corresponding dyeings undertaken in the absence of ultrasound. In the case of Dianix Crimson SF, Table 7 and Fig. 3(f) clearly show that ultrasound enhanced colour strength at 80 °C and also markedly improved the both brightness and hue angle of the dyeing. However, the findings for Dianix Crimson SF, which differed markedly to those obtained for the other five dyes used in the work, may be a corollary of the very low K/S values obtained for the 1% omf dyeings (Fig. 3(f)), as discussed below.

The potential beneficial effects which ultrasound can have upon dyeing are thought to involve various mechanical aspects, such as removal of air from fibre capillaries, agitation of dye liquor, accelerated dye diffusion within the fibre and dye disaggregation [38]. In the context of dyeing with disperse dyes, several workers reported that the use of ultrasound resulted in enhanced colour strength in the cases of nylon 6 [26], polybutylene terephthalate (PBT) [29], poly(trimethylene terephthalate) (PTT) [32] as well as PET [29,31,33]. Lee et al. concluded that the enhancement of disperse dye uptake within PET imparted by the use of ultrasound was attributable to dye disaggregation [31,33], a view recently adopted by Wang et al. in a study of the low temperature dyeing of poly(trimethylene terephthalate) (PTT) with disperse dyes [32]. Hence, the findings (Fig. 3(a)–(f) and Tables 2–7) that ultrasound enhanced the colour strength of the 1% omf dyeings on PLA fibre when dyeing had been carried out at 30–70 °C concur with those obtained for disperse dyes on nylon 6 [26], PBT [29], (PTT) [32] as well as PET [29,31,33]. The proposal that such an intensification of colour strength in the case of PET [31,33] and PTT [32] was achieved by disperse dye disaggregation may also apply to the results obtained herein, based on the observation that ultrasound often enhanced the chroma (brightness) of the dyeings which had been carried out at 30–70 °C (Table 2).

Table 4

Colorimetric data for C.I. Disperse Yellow 42 (1% omf; 50 min).

Treatment	Temp./°C	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	λ_{\max}/nm
Absence of ultrasound	30	90.7	3.7	9.6	10.2	69.0	400
	40	89.9	4.6	12.5	13.3	69.7	400
	50	88.5	5.9	16.3	17.3	70.0	400
	60	86.6	8.3	21.7	23.3	69.1	400
	70	84.4	11.1	29.7	31.7	70.0	400
	80	80.5	15.8	38.9	42.0	67.8	400
Presence of ultrasound	30	88.4	5.9	12.9	14.2	65.4	400
	40	89.2	5.5	12.9	14.0	67.0	400
	50	87.5	7.0	16.3	17.8	66.7	400
	60	86.6	8.3	20.5	22.1	68.0	400
	70	84.8	10.5	28.6	30.5	69.9	400
	80	81.7	14.5	35.9	38.7	68.0	400

Table 6

Colorimetric data for Disperse Red 167:1 (1% omf; 50 min).

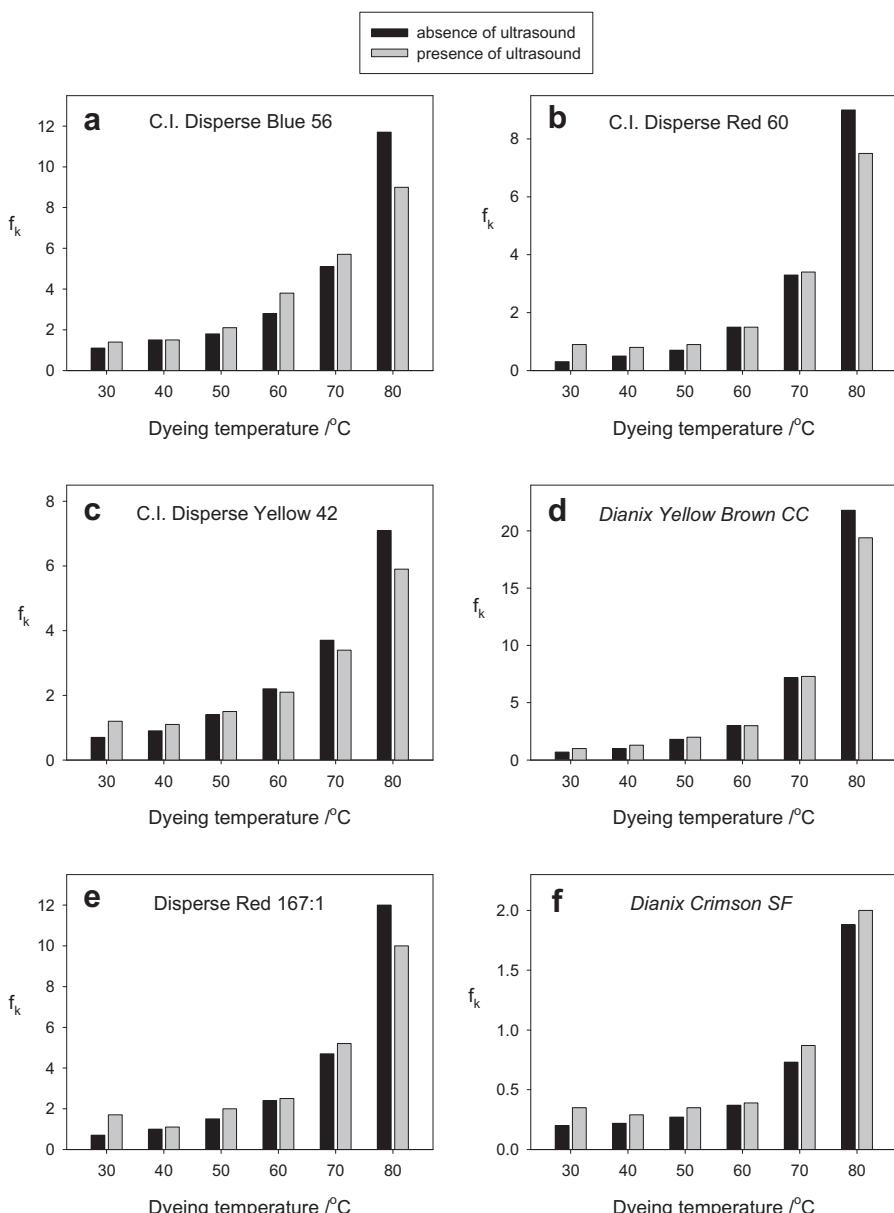
Treatment	Temp./°C	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	λ_{\max}/nm
Absence of ultrasound	30	88.6	8.0	4.1	8.9	27.0	500
	40	86.6	11.4	4.2	12.1	20.1	500
	50	83.7	15.5	4.2	16.1	15.1	500
	60	80.2	21.0	5.1	21.6	13.7	500
	70	74.4	29.7	7.2	30.6	13.6	500
	80	65.1	40.9	12.6	42.8	17.2	500
Presence of ultrasound	30	82.8	16.6	4.5	17.2	15.1	500
	40	85.9	12.3	4.1	13.0	18.2	500
	50	81.7	17.8	4.6	18.6	14.4	500
	60	79.7	21.4	4.9	22.0	13.0	500
	70	72.2	31.3	8.1	32.3	14.5	500
	80	67.0	38.7	11.3	40.3	16.2	500

Table 7Colorimetric data for *Dianix Crimson SF* (1% omf; 50 min).

Treatment	Temp./°C	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> °	λ_{\max}/nm
Absence of ultrasound	30	94.3	1.1	1.5	1.9	55.0	500
	40	93.9	2.0	2.0	2.9	44.8	500
	50	93.2	4.0	2.6	4.8	33.3	500
	60	92.2	6.4	3.8	7.5	30.4	500
	70	89.6	12.9	6.6	14.5	27.1	500
	80	85.1	22.7	12.0	25.7	28.0	500
Presence of ultrasound	30	92.4	2.7	3.2	4.2	49.3	500
	40	93.0	3.3	3.1	4.5	43.8	500
	50	92.4	5.2	3.5	6.3	33.4	500
	60	92.0	6.6	3.9	7.6	30.6	500
	70	88.8	13.8	7.2	15.6	27.7	500
	80	84.7	23.1	12.5	26.2	28.5	500

However, as Wang et al. [32] also showed that ultrasound not only removed isomers from the surface of PTT fibre but also reduced the crystallinity of PTT fibre during dyeing with C.I. Disperse Red 60, it may also be possible that the intensification in colour strength observed during dyeing at 30–70 °C (Tables 2–7; Fig. 3) might accrue from an increase in the amorphous region of the fibre imparted by ultrasound.

The findings (Tables 2–6; Fig. 3(a)–(e)) that dyeings obtained at 80 °C in the presence of ultrasound were paler, duller, yellower and of lower f_k than corresponding dyeings undertaken in the absence of ultrasound may be due to breakdown of the dye dispersions at this particular temperature. As previously mentioned, other workers have attributed the enhancement in disperse dye uptake onto PET [33] and PTT [32] imparted by the use of ultrasound to disaggregation of the sparingly soluble dyes. However, each of these studies determined the effects of exposing dispersions of C.I. Disperse Red 60 to ultrasound for up to 60 min at 50 °C [33] and

**Fig. 3.** Colour strength obtained for dyeings (1% omf; 50 min).

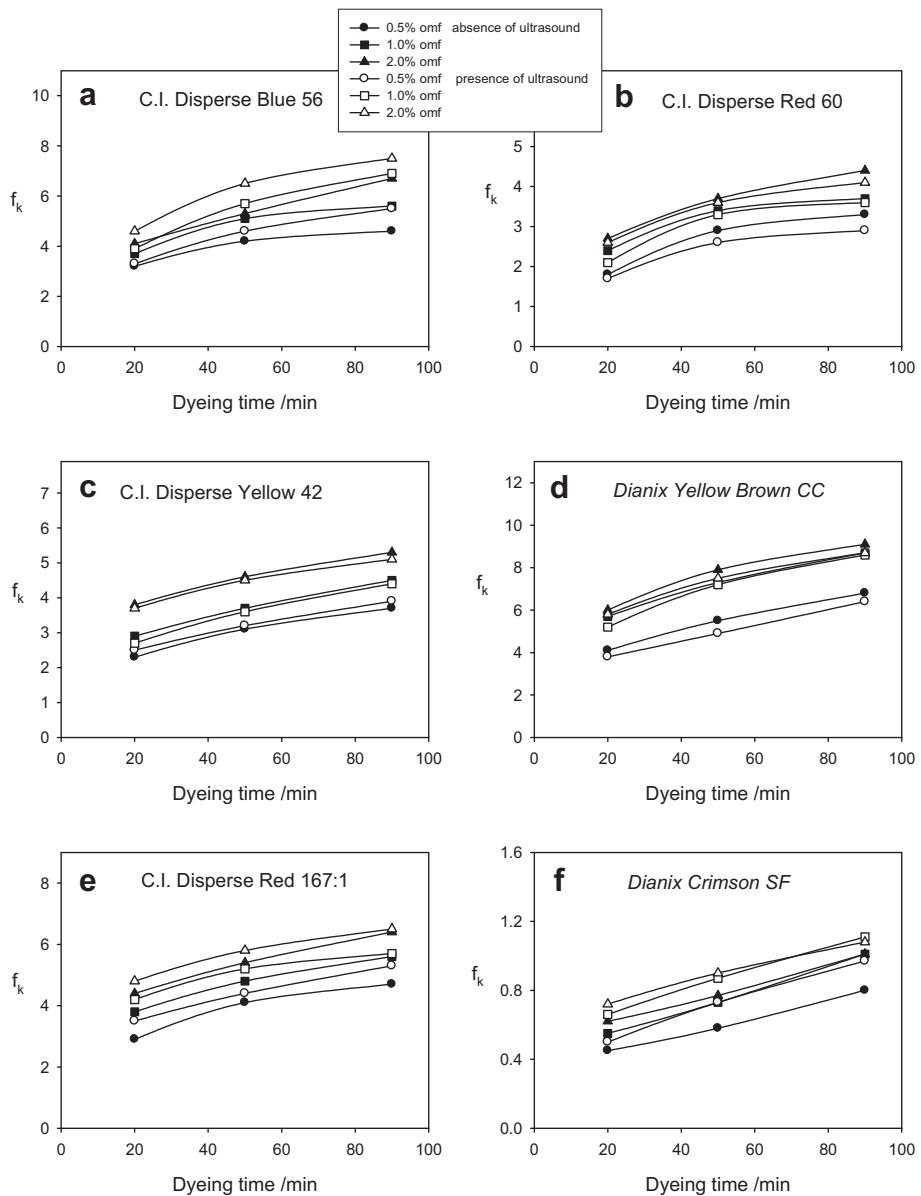


Fig. 4. Colour strength of dyeings (0.5; 1; 2% omf; 20; 50; 90 min) obtained at 70 °C.

65 °C [32] whereas, in this work, dyeing was undertaken for 50 min at 80 °C. Hence, at this higher temperature, the dye dispersions may have become overly disaggregated or even broken down, resulting in the observed reduction in dye uptake. However, this explanation does not explain the finding (Table 7 and Fig. 3(f)) that in the case of *Dianix Crimson SF*, ultrasound enhanced colour strength at 80 °C and also improved both the brightness and hue angle of the dyeing; clearly, for this particular dye, treatment with ultrasound at 80 °C appears not to have impaired the quality of the dye dispersion. Indeed, as mentioned earlier, the particular result obtained for *Dianix Crimson SF* at 80 °C in the presence of ultrasound may be a corollary of the very low *K/S* values obtained for the 1% omf dyeings (Fig. 3(f)), insofar as the disaggregating effect of ultrasound upon the dye dispersion improved dye uptake.

In view of the deleterious effect on colour strength and chroma imparted by the use of ultrasound at 80 °C on five of the dyes used herein, further work focussed on a maximum temperature of 70 °C. Fig. 4 shows the effect of different dyeing times (20, 50 and 90 min)

on the colour strength of 0.5, 1.0 and 2.0% omf dyeings obtained at 70 °C in both the presence and absence of ultrasound. As was expected, the f_k values increased in the order 0.5% < 1% < 2% omf dye applied and, in the order 20 min < 50 min < 90 min, for both the absence and presence of ultrasound. Overall, the shapes of the curves were quite similar for each of the six dyes used, which seems reasonable as the same fibre was employed throughout. However, it is also clear that whilst ultrasound enhanced the colour strength achieved for three of the six dyes used (C.I. Disperse Blue 56, C.I. Disperse Red 167:1 and *Dianix Crimson SF*), for each time of dyeing and concentration of dye used, it did not always result in enhanced colour strength for the remaining three dyes for each time of dyeing and concentration of dye used. Thus, only half of the dyes responded positively to the use of ultrasound over the range of dye concentrations and dyeing times employed, which was not entirely surprising, as the disperse dyes used had been developed for application not only in the absence of ultrasound but, perhaps more significantly, to PET rather than PLA fibre.

4. Conclusions

Ultrasound enhanced the colour strength of 0.5%, 1% and 2% omf dyeings in the cases of three of the six disperse dyes used (C.I Disperse Blue 56, C.I. Disperse Red 167:1 and Dianix Crimson SF) at each of the three dyeing times used (20, 50 and 90 min). In the case of the three other dyes used (C.I. Disperse Red 60, C.I. Disperse Yellow 42 and Dianix Yellow Brown CC), ultrasound did not always result in enhanced colour strength being achieved for each time of dyeing and concentration of dye used. The observed intensification of colour strength was attributed to dye disaggregation, based on the finding that ultrasound often enhanced the chroma (brightness) of the dyeings as well as the reports of previous workers on the effect of ultrasound on the dyeing of PET [31,33] and PTT [32] with disperse dyes. In addition, the intensification in colour strength observed may have accrued from a decrease in the crystallinity of the fibre imparted by ultrasound, as previously observed for the dyeing of PTT [32]. However, dyeing at 80 °C in the presence of ultrasound resulted in pale, dull dyeings of reduced colour strength for five of the dyes used, which was attributed to breakdown of the dye dispersions at this particular temperature.

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