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The clearing of poly(lactic acid) fibres dyed with disperse dyes using ultrasound. Part 1: Colorimetric analysis

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Abstract

Three types of clearing processes namely water, ECE detergent and reduction clearing were used to aftertreat the dyeings of six disperse dyes on PLA fibre. Reduction clearing imparted the greatest changes to the colour strength and colour of the dyeings while treatment with ECE detergent removed surplus dye and also improved the chroma of the dyeings; treatment with water had very little effect on the colour strength and colour of dyeings, even in the presence of ultrasound. Both depth of shade reduction and colour change were greater when aftertreatment was carried out at 60 °C rather than at 50 °C due to a corresponding increase in the amount of removed dye as a result of greater kinetic energy at the higher temperature. Ultrasound neither impaired nor overly enhanced the effectiveness of either the ECE detergent or the reduction clearing processes.

Keywords: Ultrasound; Disperse dyes; PLA fibre; Reduction clearing

1. Introduction

Although WH Carothers first made poly(lactic acid) (PLA) in the early 1930s through direct condensation polymerisation of lactic acid, development of the polymer as a textile fibre was, at the time, abandoned owing to its low melting point and research efforts were focussed elsewhere, resulting in the introduction of the first commercially available, wholly synthetic textile fibre, *nylon 6,6*, in 1939. Nowadays, the thermoplastic polyester, PLA, is derived from annually renewable sources, such as corn and, in recent years, has enjoyed increasing commercial interest as an apparel fibre; although the linear, aliphatic nature of the polymer imparts susceptibility to hydrolysis, this particular characteristic allows the polymer to be industrially composted.

The dyeability of PLA [1–9] is akin to that of polyesters, such as poly(ethylene terephthalate) (PET) insofar as it is dyeable with disperse dyes, although differences exist between the two fibre types in terms of dye behaviour [6]. The lower T_m of PLA fibres (around 170 °C compared to 250–260 °C for PET) coupled with the hydrolytic sensitivity of the polymer means that neither elevated temperatures (125/130 °C) nor thermofixation (commonly 210 °C), which are commonly used to apply disperse dyes to PET, can be employed for PLA. Dyeing conditions of 110–115 °C for 15–30 min at pH 4.5–5 have been recommended for PLA [6]; the use of higher temperatures, longer times of dyeing at 110–115 °C or higher pH can lead to fibre hydrolysis.

As with disperse dyed polyester, a *reduction clear* treatment is needed to remove surplus dye and auxiliaries from PLA which has been dyed with disperse dyes. In the case of disperse dyes on polyester, reduction clearing comprises, typically, submitting the rinsed, dyed material to treatment for 20–30 min at 60–80 °C in an aqueous bath containing 2–3 g l^{-1} NaOH, 2–3 g l^{-1} $Na_2S_2O_4$ and, so as to restrain the washed-off dyes and auxiliaries from re-depositing on the dyed substrate, a surfactant; the reduction cleared substrate

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is then rinsed and, if necessary, neutralised with aqueous acetic acid. Reduction clearing relies on the marked hydrophobicity of the fibre preventing aqueous agencies from penetrating the substrate and also on the fact that the process is carried out at temperatures below the T_g (typically 80–90 °C in the case of PET), which means that not only will water and dissolved chemicals (NaOH and $Na_2S_2O_4$) not easily penetrate the dyed fibre but also dye molecules will not tend to migrate from the interior of the fibre to the surface during the reduction clearing treatment. In comparison to the reduction clearing of PET, that of PLA which has been dyed with disperse dyes employs more weakly alkaline conditions (Na_2CO_3 rather than NaOH) [6,10] and for a shorter time (15 min) [6,10] because of the greater alkali sensitivity of PLA and, as the T_g of PLA is in the range 55–65 °C, reduction clearing is undertaken, preferably at 60 °C [6,10,11].

In the context of the susceptibility of PLA to hydrolysis, and bearing in mind that such hydrolysis increases with increasing time, temperature and pH, it was decided to establish whether or not a clearing treatment could be developed for dyed PLA which offered a low risk of hydrolytic damage and which constituted a more environmentally friendly approach through reduced chemical usage. To this end, the decision was made to utilise the well-known abilities of ultrasound, videlicet, process acceleration and the attainment of similar/improved results under less extreme conditions (e.g. lower temperature, reduced chemical usage). Ultrasound (sound with a frequency above the upper limit of human hearing; >20 kHz), enjoys manifold applications, in fields as diverse as medical imaging, non-destructive testing and cleaning. The intensification of various wet textile processes using ultrasound is well-known and many papers have concerned the application of the technique to dyeing, as described in two comprehensive reviews [12,13]. Essentially, in textile wet processes such as dyeing, ultrasound influences mass transfer within the inter-yarn and intra-yarn regions of the substrate and it is generally considered that transient cavitation in the vicinity of the textile surface, rather than the ultrasound waves themselves, is responsible for the intensification observed [12–14].

This paper concerns the effect of ultrasound on the clearing of disperse dyed PLA fibre and the development of a hydrosulfite- and alkali-free clearing treatment; this part of the paper focusses on the effects of clearing on the colour of disperse dyeings on PLA.

Table 1
Dyes used

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

2. Experimental

2.1. Materials

Poly(lactic acid) knitted fabric (224.8 g m^{-2}), which was obtained from NatureWorks LLC, was scoured using 2 g l^{-1} Na_2CO_3 and 1 g l^{-1} *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 generously supplied by Dystar and Clariant; the dyes were used without purification. 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. ECE detergent was sourced from the SDC. All other chemicals were of general laboratory grade supplied by Aldrich.

2.2. Ultrasound treatment

There are two methods of applying ultrasound namely *direct* and *indirect*. In the direct method, the solution of interest (in this case a reduction clearing solution) is placed directly in the ultrasound bath and the substrate (in this case, dyed PLA fibre) is immersed in the clearing bath. The advantages of this direct method of treatment are its simplicity of operation and its effectiveness; however, the removed dye and auxiliaries remain in the bath and, therefore, the reduction clear solution can be used only once; in addition, the highly alkaline solution can erode the ultrasound bath surface. The indirect method involves placing the reduction clearing solution and the dyed fibre in a container that is then placed inside the ultrasound bath. This method provides more flexibility and control over the ultrasound treatment; the removed dye and auxiliaries remain in the container and one or more solutions can be used at the same time.

In this work, the indirect method of ultrasound application was used.

2.3. Dyeing

A 2% omf depth of shade was used as this provided typical medium depth dyeings. Dyeing was carried out using a 10:1

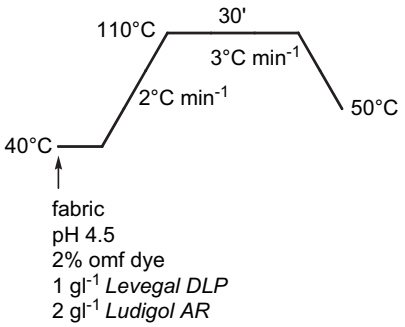

Fig. 1. Dyeing method.

Table 2
Clearing treatments used

Treatment		Temp./°C
None		
Absence of ultrasound	ECE detergent 'Standard' reduction clear	60
Presence of ultrasound	ECE detergent Modified reduction clear	60
	Water ECE detergent Modified reduction clear	50

liquor ratio, in the presence of the anionic levelling agent *Levegal DLP* (Dystar) and the anti-reducing agent *Ludigol AR* (BASF), in 300 cm^3 capacity sealed, stainless steel dyepots housed in a Roaches *Pyrotec S* dyeing machine using the method shown in Fig. 1; the pH was maintained at 4.5 using acetic acid/sodium acetate buffer.

2.4. Clearing treatments

Each of the 2% omf dyeings was subjected to the various treatments listed in Table 2.

2.4.1. In the absence of ultrasound

After dyeing, the fabric was rinsed in warm water (50 °C) and subjected to two different clearing treatments at 60 °C using a 50:1 liquor ratio (Fig. 2).

- *'Standard' reduction clear* using 2 g l^{-1} $Na_2S_2O_4$ and 1.5 g l^{-1} Na_2CO_3;
- 2 g l^{-1} ECE detergent.

In both cases, the treated dyeing was rinsed in warm water (50 °C), then in cold running water and finally allowed to dry at room temperature.

2.4.2. In the presence of ultrasound

The dyed substrate was rinsed in warm water (50 °C) and subjected to three different clearing treatments at 50 °C and 60 °C using a 50:1 liquor ratio (Fig. 3).

- *Modified reduction clearing* using 1 g l^{-1} $Na_2S_2O_4$ and 0.75 g l^{-1} Na_2CO_3, this being half the quantity of reductant and alkali employed in the above reduction clearing method;
- 2 g l^{-1} *ECE Detergent*;
- water only.

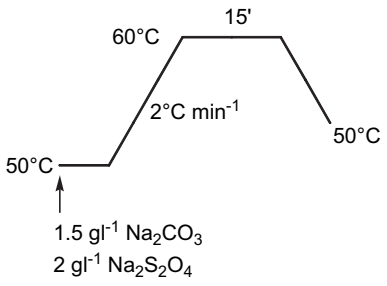

Fig. 2. Reduction clearing method.

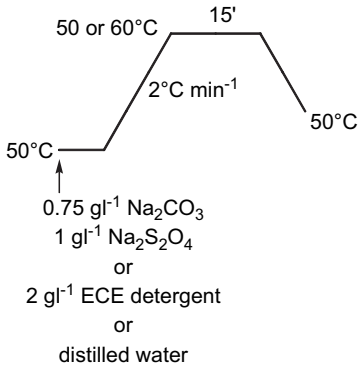

Fig. 3. Clearing methods.

In all cases, the treated dyeing was rinsed in warm water (50 °C), then in cold running water and finally allowed to dry at room temperature.

2.5. Colour measurement

The CIE L^*, a^*, b^*,C^* and h° co-ordinates were measured and the $f(k)$ values calculated from the reflectance values at the appropriate λ_{max} for each dyeing, using a *Datacolor Spectroflash 600* spectrophotometer under illuminant D_{65}, using 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 obtained for each of the 2% omf dyeings obtained using the six dyes under consideration, before and after the seven clearing treatments (Table 2) are shown in Tables 3–8. For ease of discussion, the results obtained

Table 3
Colorimetric data for C.I. Disperse Red 60

Clearing treatment		Temp./°C	L^*	a^*	b^*	C^*	h°	$f(k)$
None		–	55.4	59.1	12.3	60.4	11.8	34.1
Absence of ultrasound	ECE detergent	60	56.1	59.4	12.2	60.6	11.7	32.6
	Reduction clear		55.6	58.4	11.8	59.6	11.5	32.6
Presence of ultrasound	ECE detergent	60	55.4	59.6	12.5	60.9	11.9	33.7
	Modified reduction clear		55.8	58.9	12.1	60.1	11.6	33.5
	Water	50	55.3	59.1	12.3	60.3	11.8	34.3
	ECE detergent		55.4	59.3	12.4	60.6	11.8	33.5
	Modified reduction clear		55.7	58.6	11.9	59.8	11.5	33.2

Table 4
Colorimetric data for C.I. Disperse Blue 56

Clearing treatment		Temp./°C	L^*	a^*	b^*	C^*	h°	$f(k)$
None		–	39.7	6.9	−39.4	40.0	280.0	50.1
Absence of ultrasound	ECE detergent	60	39.9	8.0	−40.2	40.9	280.8	49.1
	Reduction clear		40.5	7.0	−39.6	40.2	280.0	46.9
Presence of ultrasound	ECE detergent	60	40.5	7.5	−40.4	41.1	280.5	47.2
	Modified reduction clear		40.7	7.4	−40.3	41.0	280.3	46.7
	water	50	39.7	6.9	−39.2	39.8	280.0	49.7
	ECE detergent		40.7	7.4	−40.4	41.1	280.4	46.7
	Modified reduction clear		40.3	7.5	−40.3	41.0	280.7	47.7

Table 6
Colorimetric data for *Dianix Yellow Brown CC*

Clearing treatment		Temp./°C	L^*	a^*	b^*	C^*	h°	$f(k)$
None		–	59.8	40.9	70.9	81.8	60.0	108.0
Absence of ultrasound	ECE detergent	60	60.4	41.6	71.9	83.0	59.9	107.7
	Reduction clear		60.3	41.3	71.6	82.6	60.0	107.1
Presence of ultrasound	ECE detergent	60	60.4	41.6	71.9	83.1	59.9	107.6
	Modified reduction clear		60.4	40.9	71.5	82.4	60.2	105.0
	Water	50	59.9	41.1	71.5	82.5	60.1	110.5
	ECE detergent		60.4	41.5	72.1	83.2	60.1	108.1
	Modified reduction clear		60.2	41.2	71.9	82.8	60.2	109.5

for C.I. Disperse Red 60 (displayed in Table 3) will be used as an exemplar.

The 'standard' reduction clearing process, which comprised treatment with 2 g l^{-1} $Na_2S_2O_4$ and 1.5 g l^{-1} Na_2CO_3 at 60 °C in the absence of ultrasound, lowered the depth of shade of the dyeing, as shown by the lower $f(k)$ values of the cleared sample; this can be attributed to the reduction clearing process having removed surface dye from the dyeings. It is apparent that the colour (hue and chroma) of the dyeings was also changed by reduction clearing at 60 °C as evidenced by a comparison of the L^*, a^*, b^*, C^* and h^o values of the dyeings (Table 3) before and after reduction clearing. The colour strength and colorimetric changes imparted to the dyeing by reduction clearing are clearly evident in the a^* vs b^* and C^* vs $f(k)$ plots shown in Fig. 4(a) and (b); the reduction in the chroma of the dyeing, imparted by reduction clearing, is especially apparent. The corresponding reflectance plots (Fig. 4(c)) for the dyeings show that the λ_{max} of the dyeing was not changed by reduction clearing.

As a previous study [15] of the reduction clearing of disperse dyed PET showed that a traditional hydrosulfite based reduction clearing treatment could be replaced by a non-hydrosulfite based wash-off at 98 °C that utilised ECE detergent, it was decided to employ this particular surfactant, which is used in standard wash testing [16], as an alternative to the standard reduction clear treatment at 60 °C in the absence of ultrasound (Table 2). Although Table 3 shows that treatment of the dyeings with 2 g l^{-1} ECE detergent also changed the colour of the dyeings and reduced colour strength, the magnitude of these changes were lower than obtained for the standard reduction clear process. A comparison of the results obtained for the standard reduction clearing and the detergent treatment emphasises the quite marked reduction in chroma imparted by reduction clearing and the slight increase in chroma that resulted from treatment with the detergent. The differences between the two clearing treatments, in terms of changes in chromaticeness and colour strength of the C.I. Disperse Red 60 dyeings, are further represented by the plots shown in Fig. 4(a) and (b); in particular, the increase in chroma brought about by treatment with the detergent is apparent, despite identical colour strength having been achieved for the two clearing treatments. The corresponding reflectance plots (Fig. 4(c)) for the dyeings again show that neither of the aftertreatments changed, markedly, the λ_{max} of the dyeings.

As mentioned, the aim of this work was to determine whether or not ultrasound could be used to intensify the reduction clearing process and enable similar/improved results to be attained under less extreme conditions. In this context, it was decided to carry out clearing in the presence of ultrasound, at 60 °C, but using half the standard reduction clearing concentrations (i.e. using 1 g l^{-1} $Na_2S_2O_4$ and 0.75 g l^{-1} Na_2CO_3 rather than 2 g l^{-1} $Na_2S_2O_4$ and 1.5 g l^{-1} Na_2CO_3; Table 2). In addition, treatment with 2 g l^{-1} ECE detergent was also undertaken at 60 °C in the presence of ultrasound (Table 2). The results in Table 3 show that in the presence of ultrasound at 60 °C, both the ECE detergent treatment and the modified

Table 5
Colorimetric data for C.I. Disperse Yellow 42

Clearing treatment		Temp./°C	L^*	a^*	b^*	C^*	h°	$f(k)$
None		–	66.6	31.3	55.1	63.4	60.4	34.3
Absence of ultrasound	ECE detergent	60	67.8	31.5	56.1	64.3	60.6	32.7
	Reduction clear		67.8	31.5	55.6	63.9	60.5	32.0
Presence of ultrasound	ECE detergent	60	67.7	31.9	56.1	64.6	60.4	33.0
	Modified reduction clear		67.5	31.3	55.9	64.1	60.6	33.1
	Water	50	66.6	31.3	55.1	63.4	60.4	34.2
	ECE detergent		67.4	31.7	56.1	64.5	60.5	33.5
	Modified reduction clear		67.5	31.2	5.8	63.9	60.4	32.8

Table 7
Colorimetric data for Disperse Red 167:1

Clearing treatment		Temp./°C	L^*	a^*	b^*	C^*	h°	$f(k)$
None		–	40.6	53.9	2.0	59.8	25.8	125.5
Absence of ultrasound	ECE detergent	60	41.2	55.0	26.9	61.3	26.1	124.8
	Reduction clear		41.5	54.5	26.3	60.5	25.8	118.3
Presence of ultrasound	ECE detergent	60	42.9	55.3	26.2	61.2	25.3	105.9
	Modified reduction clear		42.7	54.8	25.9	60.6	25.2	105.0
	Water	50	40.8	54.2	26.5	60.4	26.1	126.2
	ECE detergent		41.1	54.7	26.8	60.9	26.1	125.1
	Modified reduction clear		42.6	54.5	25.6	60.2	25.2	106.1

Table 8
Colorimetric data for *Dianix Crimson SF*

Clearing treatment		Temp./°C	*L**	*a**	*b**	*C**	*h*°	*f*(*k*)
None		–	67.8	48.6	31.2	57.8	32.7	17.7
Absence of ultrasound	ECE detergent	60	68.8	49.9	3.1	59.3	32.7	17.2
	Reduction clear		68.6	49.6	31.8	58.9	32.6	17.2
Presence of ultrasound	ECE detergent	60	68.8	49.7	32.1	59.1	32.9	17.2
	Modified reduction clear		68.3	49.2	31.7	58.6	32.5	17.2
	Water	50	68.5	48.8	31.4	58.0	32.7	17.0
	ECE detergent		68.8	49.8	31.8	59.1	32.5	17.0
	Modified reduction clear		68.7	48.8	31.7	58.2	33.6	16.9

reduction clearing treatment lowered the depth of shade of the C.I. Disperse Red 60 dyeings, as shown by the lower *f*(*k*) values of the cleared samples; however, there was little difference between the two treatments in terms of the extent of this reduction in colour strength. Also, while both treatments changed the colour of the dyeings, it is apparent that reduction clearing lowered chroma whilst treatment with the ECE detergent improved chroma, slightly. The differences in the effects of the two clearing treatments on both the colour and colour strength of the dyeings are clearly evident in the corresponding plots shown in Fig. 4(a)–(c); the latter plot shows that neither aftertreatment changed, markedly, the λ_{max} of the dyeings. A comparison of the results obtained for the treatment of the dyeings at 60 °C in the absence and presence of ultrasound (Table 3 and Fig. 4(a)–(c)) shows that using half the standard quantity of $Na_2S_2O_4$ and Na_2CO_3 in the modified reduction clearing process, resulted in a smaller reduction in *f*(*k*) values than that achieved using the standard concentrations. This finding, which is clearly demonstrated by the results displayed in Fig. 4(b), was not surprising and can be attributed to the lower concentrations of alkali and reductant having removed less dye from the fibre surface. It is also clear from Fig. 4(a) and Table 3 that the lower alkali and reductant concentrations imparted less change to both the chroma and hue of the dyeings, which can be attributed to the hydrosulfite having had a less detrimental effect on the colour of the dye at the lower concentration used.

When the results obtained at 60 °C in both the absence and presence of ultrasound are compared, it appears that ultrasound neither impaired nor overly enhanced the effectiveness of the ECE detergent treatment. It is also apparent that there was little difference between the results obtained using the standard and half-standard concentrations of alkali and hydrosulfite.

In the context of the possible enhancement of the clearing process which ultrasound might affect, it was decided to use the lower temperature of 50 °C for treatment of the dyeings. For this set of experiments, the ECE detergent clearing process and the modified reduction clearing treatment (the latter employing half the standard reduction clearing concentrations) were used. The corresponding colorimetric data (Table 3) reveal that both the detergent and the modified reduction clear

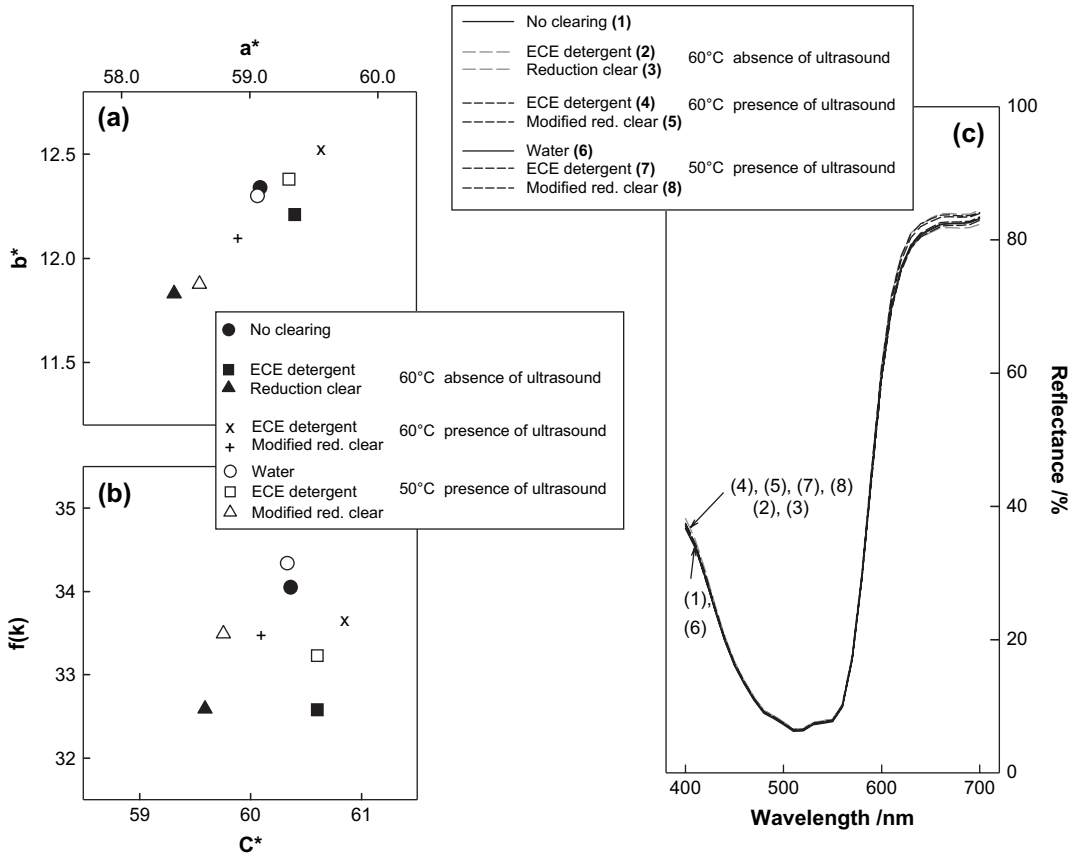

Fig. 4. Colorimetric and spectral data for C.I. Disperse Red 60.

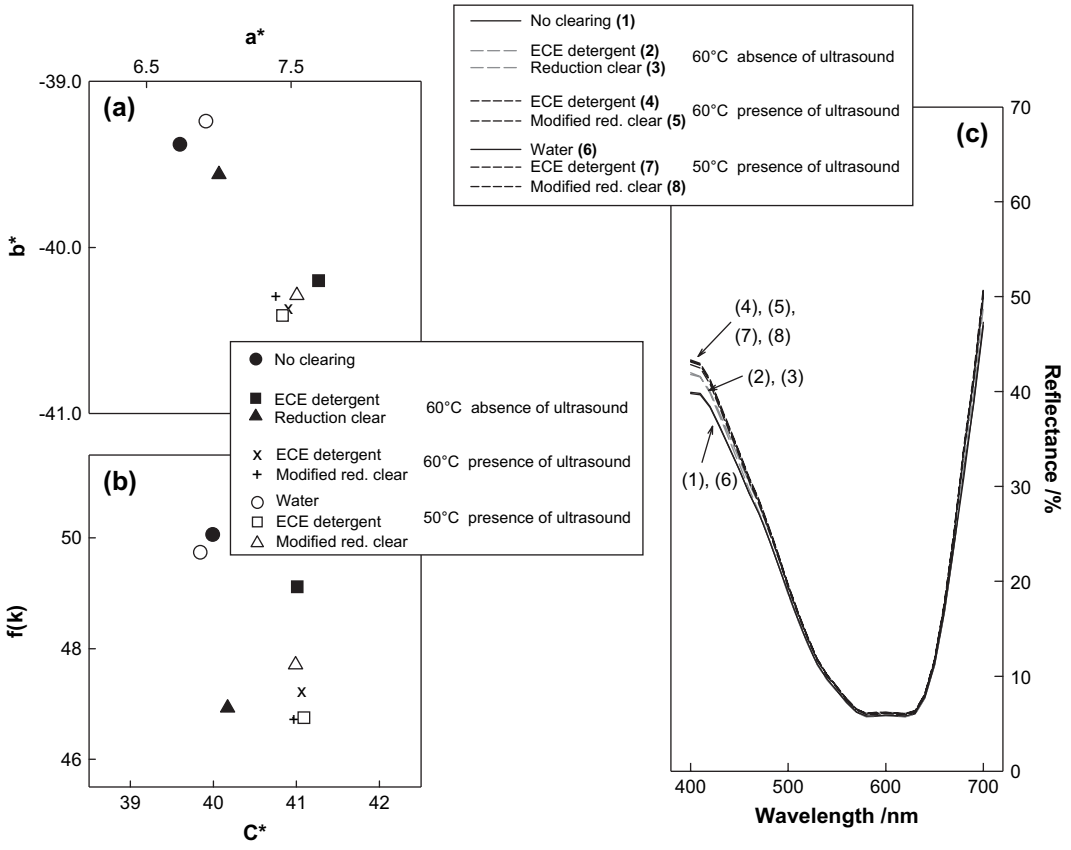

Fig. 5. Colorimetric and spectral data for C.I. Disperse Blue 56.

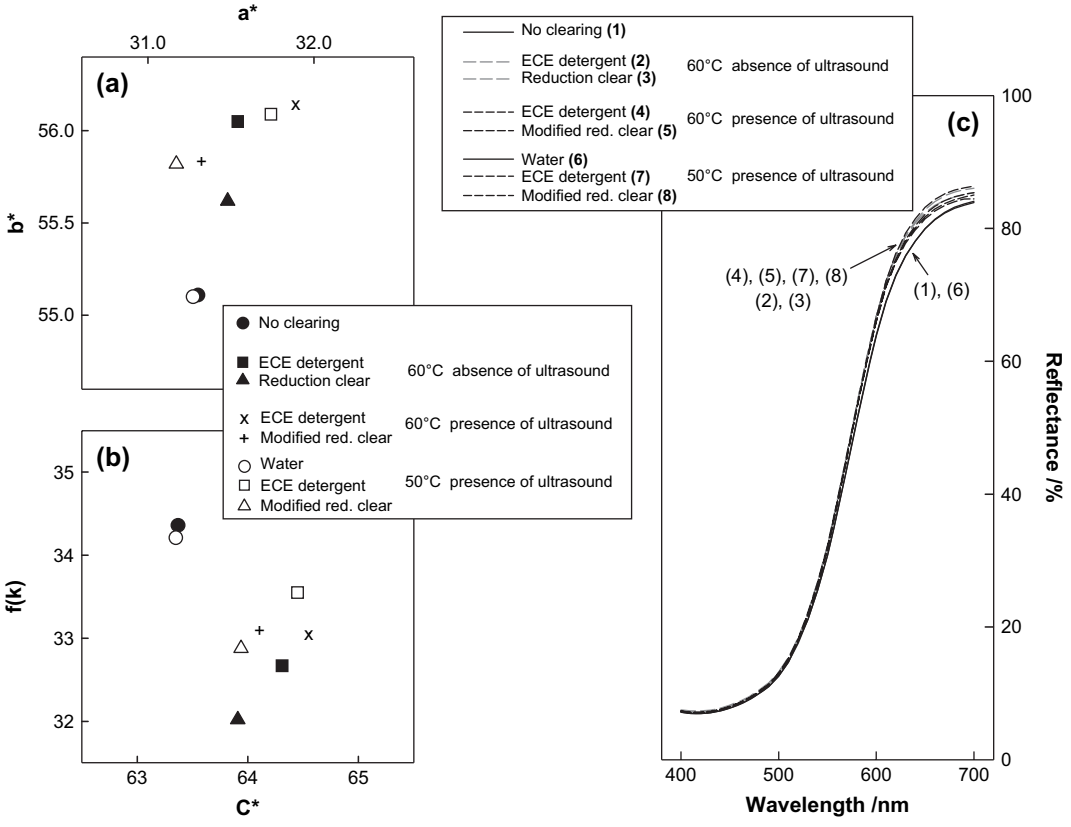

Fig. 6. Colorimetric and spectral data for C.I. Disperse Yellow 42.

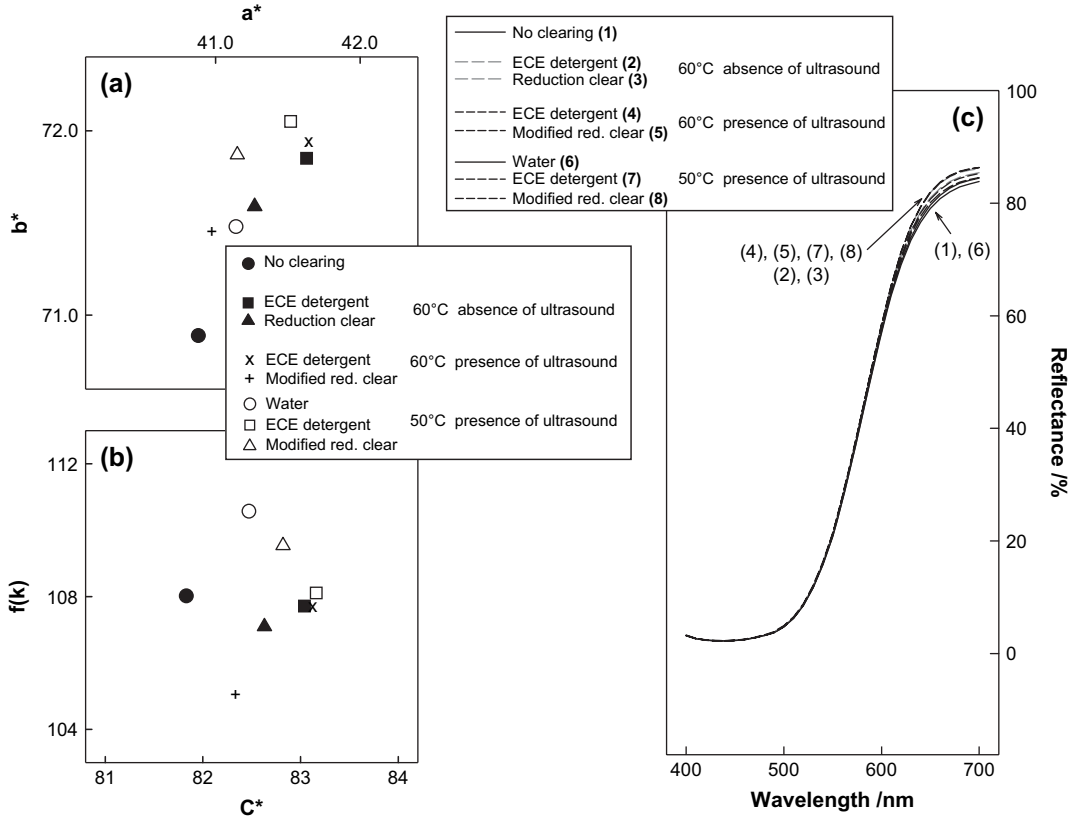

Fig. 7. Colorimetric and spectral data for *Dianix Yellow Brown CC*.

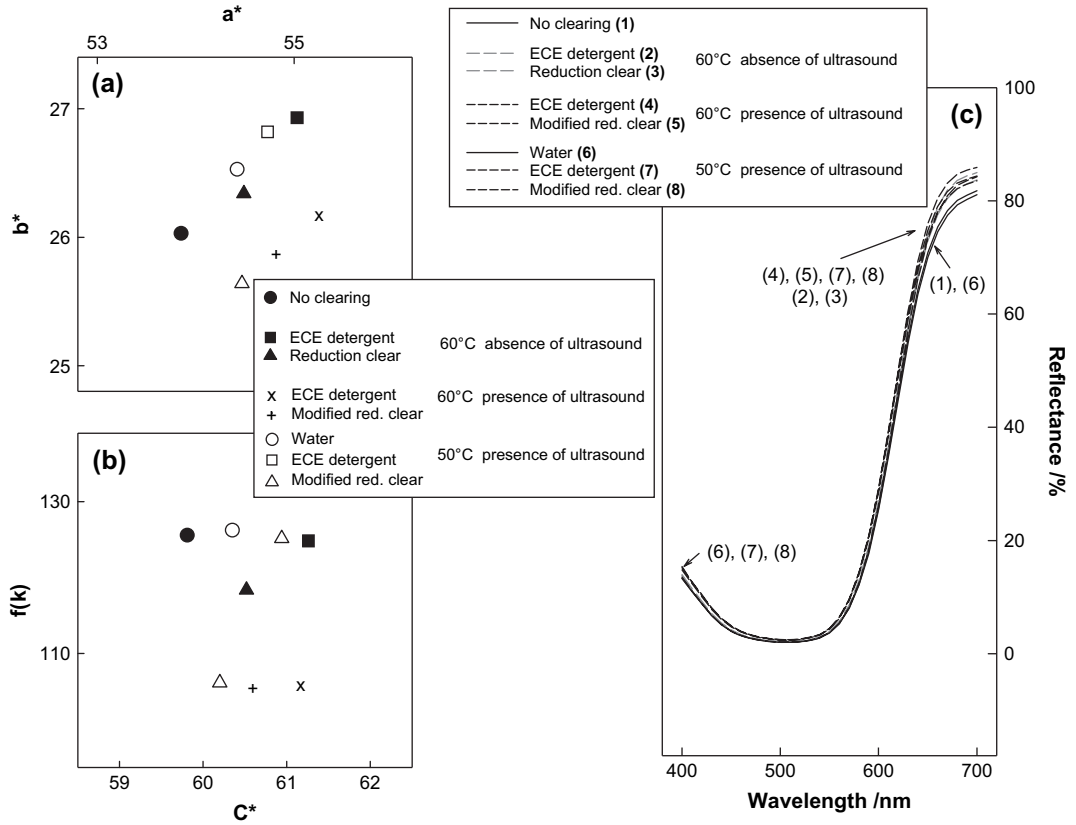

Fig. 8. Colorimetric and spectral data for C.I. Disperse Red 167:1.

treatments at 50 °C in the presence of ultrasound reduced the colour strength and changed the colour of the C.I. Disperse Red 60 dyeings. As had been observed in the case of treatment at 60 °C, the modified reduction clearing process lowered the chroma of the dyeings whilst treatment with the ECE detergent improved chroma, slightly (Fig. 4(a) and (b) and Table 3) while neither clearing treatment changed, markedly, the λ_{max} of the dyeings (Fig. 4(c)). When the effects of the detergent and the modified reduction clearing treatments at 50 °C in the presence of ultrasound are compared to those obtained at 60 °C in the presence of ultrasound (Table 3 and Fig. 4(a) to (c)), it is evident that although treatment at the lower temperature resulted in smaller changes in chromaticeness and $f(k)$, presumably, due to the lower kinetic energy involved at the lower treatment temperature, it is also clear that there were only very small difference between the effects of the treatments at 50 °C and 60 °C.

It was decided to treat the dyed PLA fibre with water at 50 °C in the presence of ultrasound in order to determine if ultrasound would enable surplus dye to be removed from the dyeing using a 'zero' chemical clearing treatment. The finding (Table 3 and Fig. 4(a)–(c)) that distilled water alone at 50 °C in the presence of ultrasound had very little effect on the C.I. Disperse Red 60 dyeing can be attributed to the marked hydrophobicity of the PLA fibre and the sparing water-solubility of disperse dyes.

In the cases of the five other dyes used in this work (Tables 4–8 and Figs. 5–9), similar findings were obtained in terms of the effects of the various clearing treatments and two different temperatures insofar as reduction clearing brought about the greatest changes in colour strength and colour of the dyeings, while treatment with ECE detergent not only removed surplus dye but also improved the chroma of the dyeings whilst treatment with water had very little effect on the colour strength and colour of dyeings. In the context of reduction clearing, there was little difference observed between the results obtained using the standard and half-standard concentrations of alkali and hydrosulfite. The reduction in depth of shade and the extent of the colour change of the dyeings imparted by reduction clearing and ECE detergent were both greater when aftertreatment had been carried out at 60 °C than at 50 °C, this being attributable to a corresponding increase in the amount of dye removed from the dyeings as a result of the greater kinetic energy of the clearing treatments at the higher temperature.

4. Conclusions

Reduction clearing imparted the largest change to the colour strength and colour of the dyeings. While treatment with ECE detergent removed surplus dye, it also improved the chroma of the dyeings. The observations that both the reduction in depth of shade and the extent of the colour change of

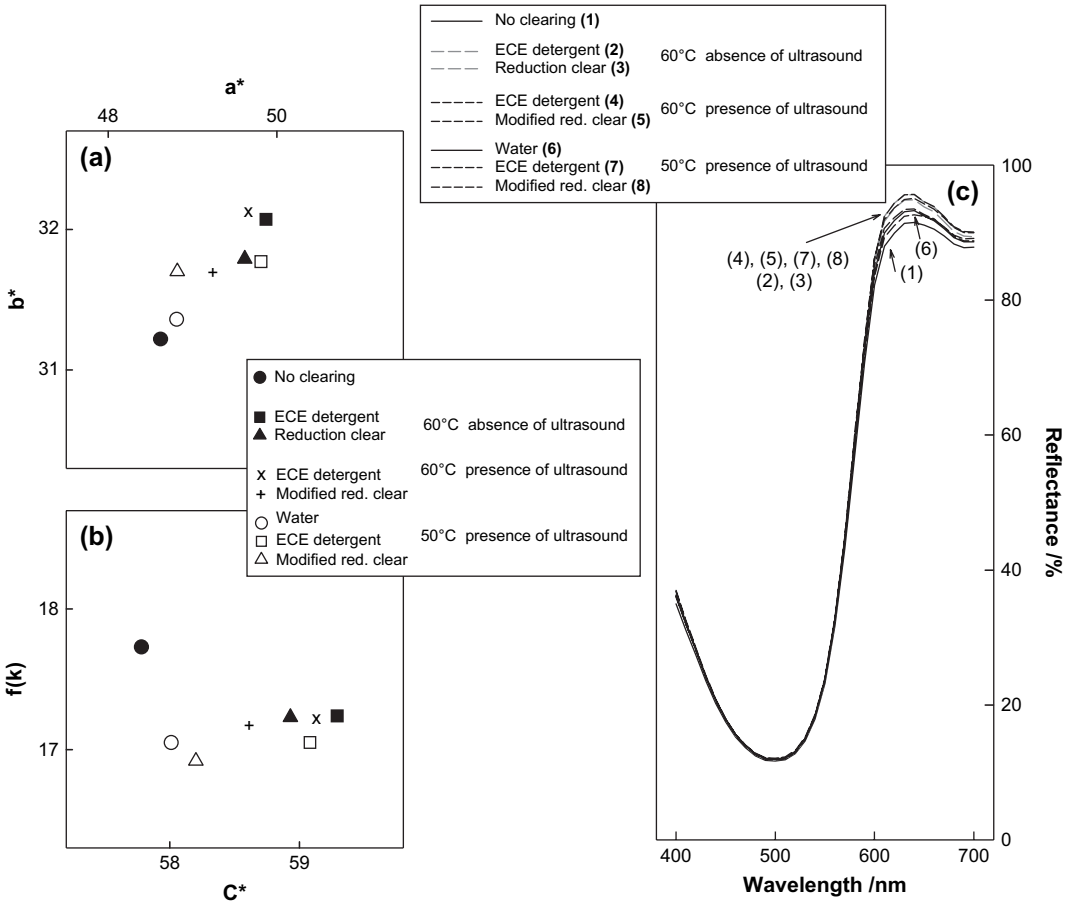

Fig. 9. Colorimetric and spectral data for *Dianix Crimson Brown SF*.

the dyeings were greater when aftertreatment had been carried out at 60 °C rather than at 50 °C can be attributed to a corresponding increase in the amount of dye removed from the dyeings owing to the greater kinetic energy of the aftertreatments at the higher temperature. From the results obtained at 60 °C in both the absence and presence of ultrasound, it appears that ultrasound neither impaired nor overly enhanced the effectiveness of either the ECE detergent treatment or the modified reduction clearing process.

The finding that treatment with water in the presence of ultrasound had very little, if any, effect on the colour strength and colour of dyeings shows that despite the well-known ability of ultrasound to intensify various wet textile processes, the use of the technology, in the manner adopted herein, did not permit a 'zero' chemical clearing process to be achieved.

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