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Coloration of Poly(lactic acid) with Disperse Dyes. 1. Comparison to Poly(ethylene terephthalate) of Dyeability, Shade and Fastness

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Abstract: The dyeability of poly(lactic acid) [PLA] with a range of commercial disperse dye was examined and compared to that of poly(ethylene terephthalate) [PET] in addition to the colour and fastness of the resultant dyeings. A screening exercise in which twenty dyes of differing energy types and chemical classes were applied to PLA revealed a substantial variation between the dyes in terms of dye uptake (12–88 % at 4 %o.w.f.). Nine dyes exhausted above 70 % and were selected for further study, which involved comparison of shade and fastness of PLA dyeings with those of the corresponding PET dyeings. Differences in shade depended on hue while wet fastness of each of the PLA dyeings was either the same or 0.5-1.0 point lower than its PET counterpart. In all but one case, dye photostability in PLA was found to be very similar to that in PET. Dye build-up profiles on PLA were also investigated and from these results mixtures of compatible dyes identified.

Keywords: PLA fiber, Disperse dyes, Dye uptake, Exhaustion yield, Fastness

Introduction

Polylactic acid (PLA) fibre is a biodegradable synthetic material manufactured from lactic acid that is in turn derived from fermentation of glucose contained in cornstarch. In common with other fibres derived from renewable resources, such as soya, bamboo and casein, PLA is seen as a way of producing more environmentally friendly goods [1-3]. Some general fibre properties have been compared in Table 1. The development of practical and economic coloration methods for PLA-based textiles is critical for successful commercialisation of the substrate. Despite study of the dyeing characteristics of the fibre becoming important, with papers recently appearing on the wet processing of PLA [4-7], relatively little has been published.

Since PLA is a relatively hydrophobic polymer, it is not unexpected that disperse dyes have been found to possess relatively high substantivity and affinity for the fibre as is the case with other hydrophobic polymers such as poly(ethylene terephthalate) (PET). However, whereas PET is conventionally dyed at around 130 °C (when carriers are not used) to enable disperse dyes to diffuse at an acceptable rate into the material, PLA must be dyed at lower temperatures over generally shorter times (typically 110 °C for 30 min). Use of excessive dyeing temperatures and/or extended dyeing times leads to fibre degradation, manifested as reductions in molecular weight, tensile strength and percentage elongation at break [2]. In addition, processing other than dyeing, such as heat setting, bleaching and scouring, can have an influence on the physical strength of PLA fibre [2,9,10].

Table 1. Comparison of fibre properties [8]

Property	PLA	PET	Nylon 6	Cotton
Specific gravity	1.25	1.39	1.14	1.52
T_g (°C)	55~60	125	90	-
T_m (°C)	130~175	255	215	None
Refractive index	1.35~1.45	1.54	1.52	1.53
Tenacity (g/d)	2.0~6.0	2.4~7.0	5.5	4.0
Moisture gain (%)	0.4~0.6	0.2~0.4	4.1	7.5

Comparisons of the dyeing properties of PLA and PET fibres have been reported in terms of dyebath exhaustion as well as the colorimetric and fastness properties of the resultant dyeings [5,11,12]. The lower reflectance of PLA compared to PET was cited as the reason for the greater colour yields (~30 %) on the former substrate compared to the latter when the same amounts of dye had been absorbed [12]. Additionally, it was concluded that PLA dyeings possessed lower wash and crock fastness than PET dyeings at a given dye concentration in the substrate, whereas the converse was true for light fastness. No correlation between dye uptake and chemical class or energy type was found.

An interesting paper investigating the disperse dyeability of PLA from the perspective of solubility parameters revealed that dyes with those of less than 25 $(\mathrm{J/cm^3})^{0.5}$ tended to perform best (exhibiting greater than 70 % sorption on PLA fibre at 2 %o.w.f. and dyeing temperature of 110 °C) [13]. This cut-off value is close to that of PLA, 20.2 $(\mathrm{J/cm^3})^{0.5}$. Dyes with a value greater than this cut-off generally showed poor exhaustion (11-58 % sorption). In contrast, there was little correlation between sorption of dye on PET at 130 °C and 2 %o.w.f., exhaustion being 95 % or greater for a range of dyes with parameters 24-39 $(\mathrm{J/cm^3})^{0.5}$.

As part of a larger study into the coloration of PLA, this paper reports the results of a preliminary investigation of the

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dyeability of various types of commercial disperse dye on PLA based on recommended dyeing conditions (110 °C). Those dyes found to perform best in terms of exhaustion were examined in more detail. Build-up profiles obtained by means of a step dyeing technique were put into context by comparison with those obtained upon application to PET using conventional conditions (130 °C). The colour properties and fastness of the resultant dyeings were compared. Finally, from a palette of dyes showing the greatest uptake on PLA, dye compatibility was explored.

Experimental

Materials

100 % PET (75 denier/36 filament) fabric was provided by Korea Dyeing Technology Center and 100 % PLA (84 denier/36 filament) fabric was woven by Shinpung Textile Co. using PLA filament yarn (Terramac, Unitika). PLA fabric was scoured at 60 °C using with Na_2CO_3 (2 g/*l*) and Kieralon jet B con.* (BASF, non-ionic surfactant; 0.5 g/*l*) for 20 min. To compare the dyeability with PLA and PET fibres, 20 disperse dyes were used, as shown in Table 2.

Dyeing and Reduction Clearing Procedure

Dyeing operations were performed by means of a KS-W24 Inter Cooler IR dyeing machine (Korea Scientific Co.). Fabric (5.0 g) was dyed at a liquor-to-goods ratio of 10:1 in a dyebath containing disperse dye (4.0 % o.w.f.) and Sunsolt RM-340 (Nicca Korea, mixture of non-ionic and anionic surfactant; 1 g/*l*) as dispersing agent. The pH of dyebath was adjusted to 5.0 using AcOH/NaOAc and then dyeing commenced at a dyebath temperature of 50 °C, which was ramped up to the maximum dyeing temperature at a rate of 2 °C/min. These maximum temperatures were 130 °C and 110 °C for PET and PLA fibres, respectively. After holding at the maximum temperature for 40 min, the dyebath was cooled back to 70 °C at a gradient of 2.5 °C/min. Fabric was then removed from the dyebath, and followed by a reduction clearing. Dyed PLA fabric was immersed in a clearing bath at 60 °C containing NaOH (97 %, 0.5 g/*l*) and $Na_2S_2O_4$ (85 %, 0.5 g/*l*) for 20 min. For PET fabrics, the treatment involved immersion in a bath containing NaOH (97 %, 1 g/*l*) and $Na_2S_2O_4$ (85 %, 1 g/*l*) at 80 °C for 20 min. The rinsed fabrics were then heat set for 60s at 160 °C for PET fibre and 130 °C for PLA fibre.

For investigation of dyeing profiles, fabric (2.5 g) was dyed at a liquor-to-goods ratio of 10:1 in a dyebath containing disperse dye (2.0 % o.w.f.) and Sunsolt RM-340 (1 g/*l*). Dyebath pH was adjusted to 5.0 with AcOH/NaOAc, then dyeing commenced at 50 °C. In the case of PLA fibre, dyed fabrics were removed at 10 °C intervals until maximum dyeing temperature was attained, whence samples were removed after 10 min intervals (i.e. 60 °C, 70 °C, 80 °C, 90 °C, 100 °C, 110 °C, 110 °C after 10 min, 110 °C after 20 min, 110 °C after 30 min, 110 °C after 40 min). A similar procedure was employed for PET fibre except that the maximum dyeing temperature was 130 °C.

Table 2. Disperse dyes used in the investigation of dyeing PET and PLA fibres

Dye	Commercial name	Chemical class
C.I. Disperse Yellow 42		Nitrodiphenylamine
C.I. Disperse Yellow 86		Nitrodiphenylamine
C.I. Disperse Yellow 126		Hydrazone
C.I. Disperse Yellow 211	Lumacron Yellow SE-4G	Hydrazone
C.I. Disperse Orange 30	Lumacron Y/Brown SER 150	Monoazo
C.I. Disperse Orange 31	Lumacron Orange 2RF	Monoazo
C.I. Disperse Orange 44		Monoazo
C.I. Disperse Orange 80		Monoazo
C.I. Disperse Brown 1	Lumacron Brown LS	Monoazo
C.I. Disperse Red 5	Lumacel Rubine 3B	Monoazo
C.I. Disperse Red 50	Lumacron Scarlet 2GH	Monoazo
C.I. Disperse Red 54	Lumacron Scarlet 2R	Monoazo
C.I. Disperse Red 74		Monoazo
C.I. Disperse Red 177	Lumacron Red FRL 150	Monoazo
C.I. Disperse Violet 33	Lumacron Rubine CB	Monoazo
C.I. Disperse Blue 56	Lumacron Blue FBL-E	Anthraquinone
C.I. Disperse Blue 148	Lumacron Blue S-3RT	Monoazo
C.I. Disperse Blue 284	Lumacron Blue S-HW	Monoazo
C.I. Disperse Blue 319		Monoazo
C.I. Disperse Blue 374		Monoazo

Colorimetric Measurements

The colorimetric properties of the dyeings were determined by a Datacolor SF 600+ spectrophotometer (Datacolor International) using D_{65} illuminant and 10° observer, UV excluded and specular component included. The fabrics were measured at three locations and the results were averaged.

Determination of Dye Exhaustion Yields

Depth of shade was assessed in terms of the colour yield (K/S) values calculated according to the Kubelka-Munk equation. Dye uptake was determined spectrophotometrically, measuring the absorbance of diluted dyebath samples at the wavelength of maximum absorption (λ_{max}) of the dye. The percentage of dyebath exhaustion (%E) was calculated using the following formula:

$$\%E = [(A_0 - A_1)/A_0] \times 100\ \%$$

where A_0 and A_1 are the absorbance at λ_{max} of the dye in the dyebath before and after dyeing respectively.

Wash and Light Fastness Test

The wash fastness and light fastness of all the heat-set dyed fabrics were evaluated according to the Marks & Spencer C4A and ISO 105 B02 methods respectively [14].

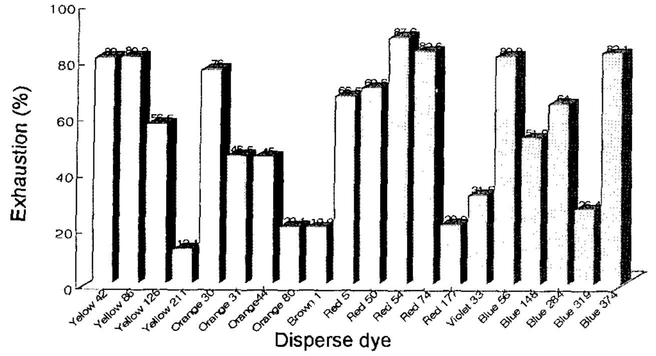

Figure 1. Percentage exhaustion of disperse dyes applied to PLA at 4.0 %o.w.f.

Results and Discussion

Dye Exhaustion in PLA Dyeing

Percentage dye exhaustion figures for each of the twenty dyes when applied to PLA are given in Figure 1. These values ranged widely, being as low as 12 % for C.I. Yellow 211 and as high as 88 % for C.I. Disperse Red 54. Just under half of the dyes had exhaustion values above or close to 70 % at the heavy depth applied (4 %o.w.f.): C.I. Disperse Yellows 42 and 86; Orange 30; Disperse Reds 50, 54 and 74; Disperse Blues 56, 284 and 374. Exhaustion of less than 30 % was observed for a quarter of the dyes, including C.I. Disperse Orange 80, Disperse Brown 1, Disperse Red 177 and Disperse Blue 319.

The exhaustion figures correlate well in some instances with previously reported data: for example, C.I. Disperse Yellows 42 and 86 were found to have values of 80 % as against literature figures of 85 % (2 %o.w.f., 50:1 LR at 100 °C) [11] and 89 % (2 %o.w.f., 15:1 LR at 110 °C) [7] respectively. The poor exhaustion of C.I. Disperse Yellow 211 on PLA has been observed previously [7]. There are however also some discrepancies, for example, C.I. Disperse Violet 33 being reported to exhaust to a level of 98 % on PLA [5] as against a figure of just 32 % in this study: the difference can be explained in that only a relatively low amount of dye was applied (0.72 %o.w.f. at 110 °C), thereby favouring high exhaustion relative to the conditions employed in this investigation. Differences for C.I. Disperse Brown 1 and Blue 56 are harder to rationalize: while lower exhaustion (20 %) than that reported previously on PLA (52 % for 2 %o.w.f. with 50:1 LR at 100 °C) [11] for the brown dye may be related to differences in the amount of dye applied (as was the case with Violet 33 as mentioned above), the exhaustion of Blue 56 was much higher (81 %) than that reported in previous work (42 %; 2 %o.w.f. at 15:1 LR and 110 °C) [7].

Colour Properties of the PET and PLA Dyeings

Eight of the disperse dyes that exhausted well on PLA (uptake above 70 %) were applied to PET in order to assess the effect of substrate on shade. For each dye, the application level was adjusted so as to give the similar depth of shade on PET as that obtained with PLA. Colorimetric data using the CIELAB system are given in Table 3.

Table 3. Colorimetric properties of the PET and PLA dyeings

Dye	Fiber	L^*	a^*	b^*	C^*	H^*
Yellow 42	PET	81.9	6.5	80.2	80.5	85.3
	PLA	85.8	-5.1	82.8	83.0	93.5
Yellow 86	PET	74.6	23.8	71.0	74.9	71.4
	PLA	79.4	13.6	87.6	88.6	81.2
Orange 30	PET	48.4	42.6	47.2	63.6	48.0
	PLA	53.8	49.1	62.4	79.4	51.8
Red 50	PET	39.1	51.1	30.8	59.7	31.1
	PLA	45.1	57.9	48.0	75.2	39.7
Red 54	PET	46.3	53.6	43.8	69.2	39.3
	PLA	48.5	58.7	54.6	80.2	43.0
Blue 56	PET	32.4	6.2	-40.9	41.4	278.6
	PLA	32.5	19.0	-50.1	53.6	290.8
Blue 284	PET	21.6	11.1	-29.4	31.4	290.7
	PLA	23.8	17.5	-40.0	43.6	293.6
Blue 374	PET	28.0	-6.3	-21.9	22.8	254.0
	PLA	21.3	2.5	-21.2	21.4	276.7

Generally, the PLA dyeings were lighter and brighter than the corresponding PET dyeings as indicated by the greater L^* (lightness) and C^* (chroma) values of the former, except in the case of C.I. Disperse Blue 374.

The difference in hue of the dyes on PLA compared to PET varied with hue. In the case of the yellow and blue dyes (with the exception of Blue 374), shades were greener on PLA compared to PET, whereas the orange and red dyes were yellower on PLA than PET.

Identification of Dye Compatibility Using Step Dyeing Method

In commercial dyeing operations, mixtures of dyes are usually required in order to obtain the desired hue and depth of shade as well as fastness. It is important that the dyes that constitute the mixture have similar rates of dyeing, otherwise faults associated with differential build-up such as dyeing spots are easily caused. Use of dyes that are closely matched in terms of dyeing rate has several benefits including improved reproducibility. Such dyes are said to be compatible: a combination of three compatible dyes of different hues, a trichromat, is particularly useful for achieving a gamut of shades in a reliable manner. The compatibility of the nine dyes discovered to build-up well on PLA (exhaustion 70+%) was examined by comparing their dyeing rates through analysis of dyebath samples at various stages in the procedure.

K/S values of the dyed PLA and PET samples are shown

Table 4. K/S values of PET and PLA dyed by step dyeing procedure for selected yellow and orange disperse dyes

	Yellow 42		Yellow 86		Orange 30	
	PET	PLA	PET	PLA	PET	PLA
60 °C	0.29	0.15	0.27	0.15	0.45	0.57
70 °C	0.28	0.18	0.27	0.17	0.47	0.68
80 °C	0.29	0.42	0.29	0.35	0.52	1.40
90 °C	0.33	1.79	0.31	1.66	0.68	4.00
100 °C	0.47	6.15	0.53	7.37	1.73	15.2
110 °C	1.85	7.81	1.91	10.0	4.43	19.4
110 °C × 10'		11.4		7.22		20.1
110 °C × 20'		10.9		9.43		19.7
110 °C × 30'		11.1		9.04		19.7
110 °C × 40'		10.8		9.48		19.8
120 °C	4.05		3.15		7.69	
130 °C	4.79		3.89		11.5	
130 °C × 10'	5.32		4.06		13.2	
130 °C × 20'	5.30		4.12		12.6	
130 °C × 30'	5.37		4.08		12.6	
130 °C × 40'	5.40		4.33		12.7	

Table 5. K/S values of PET and PLA dyed by step dyeing procedure using selected red disperse dyes

	Red 50		Red 54		Red 74	
	PET	PLA	PET	PLA	PET	PLA
60 °C	0.33	0.50	0.31	0.48		0.50
70 °C	0.33	0.58	0.36	0.53		0.68
80 °C	0.36	0.79	0.41	0.70		1.24
90 °C	0.60	3.97	0.76	3.38		6.18
100 °C	2.10	16.8	1.97	16.2		16.7
110 °C	5.90	22.5	6.45	22.0		19.2
110 °C × 10'		23.0		21.0		19.1
110 °C × 20'		23.1		20.6		18.4
110 °C × 30'		22.7		20.8		18.9
110 °C × 40'		22.8		21.0		18.4
120 °C	14.0		9.74			
130 °C	14.6		10.7			
130 °C × 10'	15.8		12.0			
130 °C × 20'	15.1		12.5			
130 °C × 30'	15.7		12.0			
130 °C × 40'	16.4		12.3			

Table 6. K/S values of PET and PLA dyed by step dyeing procedure using selected blue disperse dyes

	Blue 56		Blue 284		Blue 374	
	PET	PLA	PET	PLA	PET	PLA
60 °C	0.35	0.48	0.34	0.49	0.35	0.51
70 °C	0.36	0.52	0.39	0.55	0.35	0.51
80 °C	0.57	0.60	0.37	0.65	0.39	0.59
90 °C	1.24	2.30	0.53	2.05	0.51	2.35
100 °C	3.28	7.43	1.23	12.1	1.07	14.2
110 °C	6.79	8.71	3.62	21.4	2.82	21.7
110 °C × 10'		10.9		22.6		22.3
110 °C × 20'		11.0		22.8		20.5
110 °C × 30'		10.7		23.3		21.1
110 °C × 40'		10.8		22.2		21.4
120 °C	8.63		10.4		6.53	
130 °C	10.0		18.0		9.98	
130 °C × 10'	9.57		19.3		13.2	
130 °C × 20'	9.22		19.5		15.3	
130 °C × 30'	9.83		18.8		13.3	
130 °C × 40'	9.65		20.7		14.4	

in Tables 4-6. There was almost no dye sorption below 80 °C in the case of both substrates. A sharp increase in the uptake of dye occurred with PLA at around 90-100 °C, whereas marked sorption did not take place with PET until temperatures of 110-120 °C were reached, which is consistent with the higher glass transition temperature of PET compared to that of PLA.

Although equal amounts of dye were applied to PLA and PET, colour yields were greater on the former substrate, i.e. K/S values were higher, than the latter material at the end of the dyeing procedures. Since the refractive index of PLA is lower than that of PET, a smaller proportion of light is reflected from the surface of PLA compared to that of PET giving the PLA dyeings the appearance of a deeper shade [12,15].

The energy type of a disperse dye is a classification of how readily the dye build up onto polyester: dyes tend to be most compatible when they are of the same energy type. While the limited set of data, there are instances of dyes of the similar energy type being compatible: for example, dyeings of C.I. Disperse Yellow 42 and C.I. Disperse Blue 56 showed very similar increases in K/S during the dyeing procedure as shown in Figure 2. This figure also indicates that a combination of C.I. Disperse Orange 30, C.I. Disperse Red 50 and C.I. Disperse Blue 374 could form a useful trichromat as their build-up profiles are similar.

Fastness

The wash and light fastness ratings of PLA and PET dyeings of similar depth are shown in Table 7. With the exception of Yellow 42, Blue 284 and Blue 374, wash fastness of the PLA dyeings was 0.5 to 1 points lower than the ratings for the corresponding PET dyeings. Since colour yields for PLA are greater than PET for a given concentration of dye in the fibre, it is likely that for dyed PLA and PET fabrics containing the same concentration of dye, the former dyeings would

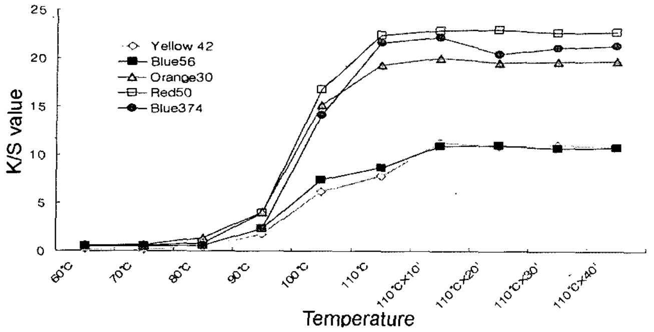

Figure 2. Dyeing profiles for selected disperse dyes on PLA.

Table 7. Wash and light fastness of dyed PET and PLA fabrics

Dye	Wash fastness (PET/PLA)		Light fastness (PET/PLA)
	Stain on acetate	Stain on nylon	
Disperse Yellow 42	4-5/4-5	4-5/4-5	4-5/4-5
Disperse Yellow 86	4/3-4	4/3	4-5/4-5
Disperse Orange 30	4-5/3-4	4-5/3-4	4-5/4-5
Disperse Red 50	4/3-4	3-4/3	4-5/4-5
Disperse Red 54	4/3	4/3	4-5/4-5
Disperse Blue 56	4/3-4	3-4/3	4-5/4-5
Disperse Blue 284	4-5/4-5	4-5/4-5	4-5/2-3
Disperse Blue 374	4-5/4-5	4-5/4-5	2/2-3

Table 8. Changes in colour, lightness and chroma of PET and PLA dyeings upon light fastness testing

Disperse dye	ΔE^* (PET/PLA)	ΔL^* (PET/PLA)	ΔC^* (PET/PLA)
Yellow 42	2.72/3.92	-1.11/-1.99	-0.50/-2.17
Yellow 86	1.49/0.37	0.22/-0.23	-1.48/-0.19
Orange 30	0.44/1.35	-0.24/-0.08	-0.07/-1.34
Red 50	1.80/2.42	-0.38/0.73	-1.28/-2.12
Red 54	1.55/2.10	-0.13/-0.31	-1.46/-2.07
Blue 56	1.84/4.31	0.42/-0.29	-1.66/-4.25
Blue 284	2.16/2.91	-0.74/2.36	-1.91/-0.55
Blue 374	2.94/5.24	2.33/3.81	0.78/3.16

exhibit lower wet fastness.

It has previously been reported that light fastness of PLA dyeings was much lower than that of corresponding PET dyeings [16]. However, the data reported in Table 8 are more in agreement with another study in which dyeings of each substrate possessed similar fastness [12]: the tabulated data reveal no difference in photostability for six of the eight dyes. The only dye for which substrate appears to be an influence is C.I. Disperse Blue 284 since light fastness is two points lower on PLA than PET. The poor light fastness of C.I. Disperse Blue 374 on both PLA and PET seems to be a consequence of intrinsically low photostability of the dye.

Differences in colour (ΔE^*), lightness (ΔL^*) and chroma (ΔC^*) brought about by exposure during testing were examined. As shown in Table 8, colour differences were larger for the PLA dyeings (aside from that of C.I. Disperse Yellow 86) although the small size of the difference reflects the similar photostabilities of the dyes on PLA and PET. However, while C.I. Disperse Blue 284 showed significantly better light fastness on PET than PLA, ΔE^* values for both dyeings were similar.

Conclusions

In this study, the dyeability of PLA and PET fibres with a selection of commercial disperse dyes was investigated in addition to the colour and fastness properties of the resultant dyeings. A wide range of dyebath exhaustion percentages was observed (as has been noted in other studies) with only nine of the dyes exhibiting relatively high exhaustion when applied to PLA (above 70 % at 4.0 %o.w.f.). These levels are still significantly lower than would be expected on PET which may in part be a consequence of PLA being dyed at a lower temperature to prevent unacceptable fibre damage.

The best-performing dyes were utilized for further investigation of PLA dyeing. Of these, the yellow and blue dyes were greener in hue on PLA than on PET, while orange/red dyes were yellower. The PLA dyeings exhibited greater L^* and C^* values. Step dyeing experiments indicated that there were instances of dyes of the same energy type potentially being compatible on PLA. Wash fastness of the PLA dyeings was generally similar or slightly worse than the corresponding PET dyeings; however, it is likely that for dyed PLA and PET fabrics containing the same concentration of dye, the former dyeings would exhibit lower wet fastness. Photostabilities of nitrodiphenylamine, monoazo and anthraquinone dyes on PLA were very similar to those observed on PET aside from the case of one blue monoazo dye.

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