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〈Research Paper〉

PET Dyeing in Black Shade with Disperse Dyes of Three Primary Color

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Abstract— PET fabric was dyed in black shade with disperse dyes of three primary color. The effect of combination ratio of dye concentration and energy type of disperse dye on dyeing and color property of black dyed PET fabric was investigated. Dyeing compatibility of disperse dyes of three primary color on PET was evaluated by comparison of dyeing rates of them. From the results, color property was dependent upon the combination ratio of dye concentration. In order to obtain low lightness (L*) and black color, blue dye should be added enough. S-type disperse dye and 130℃ dyeing was suitable rather than SE-type dyes or 120℃ dyeing. Disperse dyes of three primary color used showed good dyeing compatibility at high dye concentration. Washfastness was fair to moderate and lightfastness was moderate.

Keywords: *black dyeing, disperse dye, combination ratio of dye concentration, energy type, compatibility*

1. INTRODUCTION

In black dyeing of PET, it is an interesting and important issue how to obtain a deep black-colored fabric. Many efforts has been made and they can be categorized into three approaches. First one is to make surface of fiber to be rough, which reduce reflectance of incident light. For this, PET polymer is mixed with inorganic particles in the fiber spinning tank, and then spinned into fiber yarn. Once fabric is made from the yarn, the inorganic particles are removed by alkali treatment forming micro crater on the surface of fiber. Low temperature plasma treatment or sputter etching of fabric is also the methods for roughness of fiber[1-6]. Second approach is to coat fabric surface with low refractive compounds[7-8]. Third one involves selection of high performance dye or process optimization in dyeing so that more amount of dyes can be exhausted into the fiber.

Many studies reported are related to surface treatment which are first or second approaches. However, efforts to investigate dye or dyeing process for deep black color has not been studied much. In commercial black dyeing of PET fabric, high concentrations of disperse dyes of three primary color or black dye where the three dyes are already mixed are generally used. But this leads low exhaustion level of dyes and discharge of unfixed dye into dyehouse effluent. Therefore, it is needed to obtain black color of required quality using dyes as little as possible. Technically, disperse dye can be classified as high energy level range or S-type, medium energy level range or SE-type, and low energy level range or E type dyes. S-type disperse dyes have big size of dye molecule and are usually used for deep shade of dyeing while E-type dyes are small size and used for pale shade. SE-type dyes have properties between S and E type.

In this study, the effect of combination ratio of dye concentration and energy type of disperse dye on dyeing and color property of black dyed PET fabric was investigated. Dyeing compatibility of disperse dyes of three primary color on PET was evaluated by comparison of their dyeing rates. Wash fastness and light fastness were also investigated.

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2. EXPERIMENTAL

2.1 Materials

Scoured and bleached PET woven fabric (KS K 0905, plain, 107×97 tpi, weight 70±5 g/㎡) was used. Six disperse dyes were provided from Kyung-in Synthetic Co. Energy types and chemical structures of the dyes are shown in Table 1.

2.2 PET dyeing

PET fabric was dyed in an IR dyeing machine (DTC-6000, DaeLim Korea) at a liquor ratio of 10:1. Dye mixture was used with disperse dyes of three primary color (yellow, red, and blue). Total concentration of dye mixture was fixed to 10% owf while combination ratio of dye concentration was varied as shown in Table 2. Commercial dispersing agent (1 g/*l*) was used to maintain dispersion stability of disperse dye during dyeing.

Dyebath pH was buffered as pH 5 with sodium acetate (0.05M)/acetic acid.

Dyeing was commenced at 70℃. The dyebath temperature was raised at a rate of 1.5℃/min to 130℃, maintained at the temperature for 60 min and then rapidly cooled to room temperature. The dyed fabrics were rinsed and then reduction cleared in an aqueous solution of 1 g/*l* sodium hydroxide and 1 g/*l* sodium hydrosulfite at 80℃ for 10 min.

For measuring dyeing rate, PET fabrics were dyed with single disperse dye of each three primary color using the same dyeing condition. The dyed samples were extracted regularly during heating step from 70 to 130℃. Then each dyed sample was rinsed and reduction cleared.

The color parameters of the dyed fabrics were determined on a Macbeth coloreye 3100 spectrophotometer, under illuminant D65 using the 10° standard observer with specular component excluded and UV component included.

Table 1. Disperse dyes used in this study

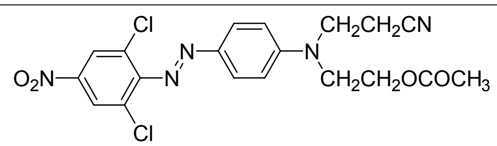
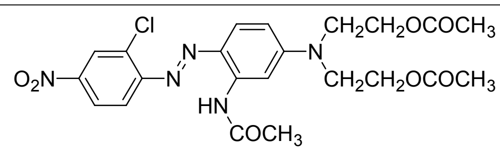
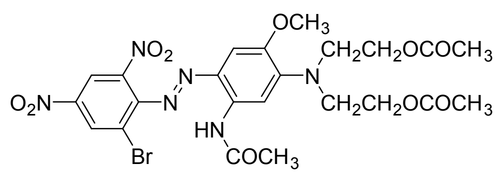
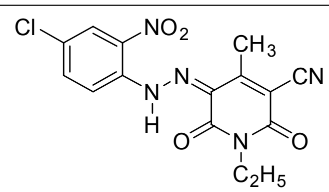
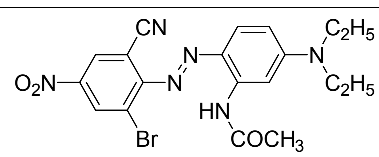
Energy Type	Commercial name	C.I. Generic name	Structure
S	Synolon Yellow Brown K-2RS	C.I. Disperse Orange 30	
	Synolon Rubine K-GFL	C.I. Disperse Red 167:1	
	Synolon Navy Blue K-GLS	C.I. Disperse Blue 79:1	
SE	Synolon Yellow K-4G	C.I. Disperse Yellow 211	
	Synolon Red K-F3BS	C.I. Disperse Red 343	-
	Synolon Blue K-BR	C.I. Disperse Blue 183:1	

Table 2. Combination of disperse dye concentration in dye mixture

Energy type	Dye concentration (% o.w.f.)			
	Yellow	Red	Blue	Total
S	4	3	3	10
	3	4	3	10
	3	3	4	10
	1	4.5	4.5	10
	4.5	1	4.5	10
	4.5	4.5	1	10
SE	4	3	3	10
	3	4	3	10
	3	3	4	10
	1	4.5	4.5	10
	4.5	1	4.5	10
	4.5	4.5	1	10

2.3 Fastness test

The dyed fabrics were subjected to wash (ISO 105-C06/C1S:1994) fastness test after heat-set at 180 ℃ for 60 s. The shade change together with staining of adjacent fabrics was assessed according to appropriate SDC grey scale. Light fastness (ISO 105-B02:1994) was also evaluated according to blue scale.

3. RESULTS AND DISCUSSION

3.1 Spectral properties of fabric dyed with single and mixture dye

Fig. 1 shows K/S values in the visible range of PET fabric dyed with S-type single dye and mixture of the dyes. In Figure 1a, fabric sample dyed with yellow dye (3% owf) absorbed visible light in the range of 360-640 nm showing maximum absorption (highest K/S values) at 440 or 460 nm. Sample dyed with red dye (3% owf) also absorbed 360-640 nm while maximum absorption was 520 or 540 nm. Blue (4% owf) dyed sample absorbed 360 to 740 nm showing maximum absorption at 600 nm. Fabric dyed with mixture of these three dyes absorbed 360 to 740 nm not showing apparent maximum absorption but exhibiting high K/S values from 440 to 640 nm.

Althought the fabric dyed with mixture dyes showed relatively low K/S values at 360-440 nm and at 640-740 nm, it was black shade which suggested that 440-640 nm is important range for black color. In Figure 1b, generally similar results were obtained with Figure 1a. As low concentration (1% owf) was applied for yellow dye, dyed sample showed lower K/S values but same absorption behavior when compared with the yellow sample in Figure 1a. Red and blue samples showed higher K/S values than those in Figure 1a. Black sample also absorbed 360 to 740 nm exhibiting high K/S values from 440 to 640 nm.

3.2 Effect of combination ratio of dye concentration and dyeing temperature

Fig. 2 shows K/S values in the visible range of black dyed fabrics (dyeing at 130℃) with S-type disperse dyes of three primary color according to the combination ratio of dye concentration (yellow:red:blue). When the combination ratio was 4:3:3, 3:4:3, and

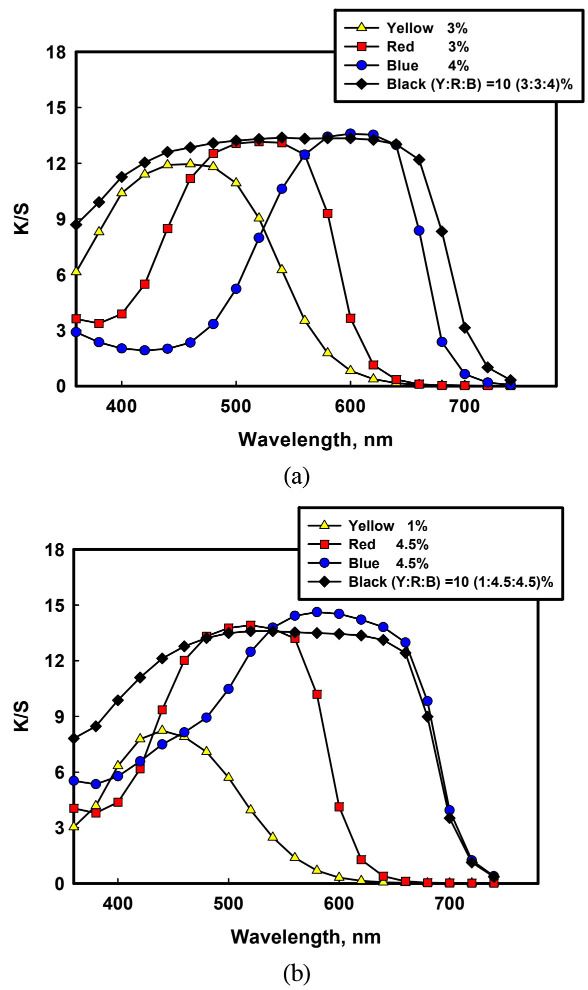
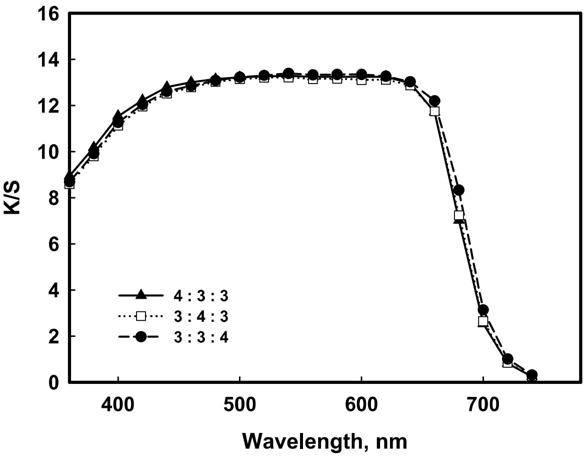
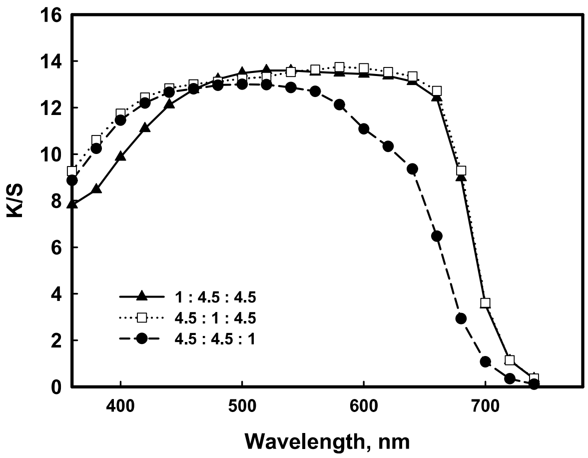


Fig. 1. K/S values in the visible range of PET fabric dyed with S-type single dye (yellow, red, and blue) and mixture of the dyes (black). (a) dye concentraion : yellow 3%, red 4%, and blue 4% owf; (b) dye concentraion : yellow 1%, red 4.5%, and blue 4.5% owf.



(a)



(b)

Fig. 2. K/S values in the visible range of PET fabric dyed at 130℃ with mixture of S-type disperse dyes of three primary color. (a) combination ratio of dye concentraion [yellow:red:blue], 4:3:3, 3:4:4, and 3:3:4% owf; (b) combination ratio of dye concentraion [yellow:red:blue], 1:4.5:4.5, 4.5:1:4.5, and 4.5:4.5:1% owf.

Table 3. The effect of dyeing temperature and combination ratio of dye concentration on color properties of PET fabric dyed with S-type (high energy level range) disperse dyes

Energy type	Dyeing temp. (℃)	Combination ratio of dye concentration (o.w.f.) [Y:R:B][a]	CIE $L^*a^*b^*$ values			ISCC-NBS Color name
			L^*	a^*	b^*	
S	130	4 : 3 : 3	22.1	0.8	-0.5	Black
		3 : 4 : 3	22.2	0.9	-0.7	Black
		3 : 3 : 4	22.0	0.8	-0.9	Black
		1 : 4.5 : 4.5	21.9	1.3	-1.7	Black
		4.5 : 1 : 4.5	21.8	0.4	-0.9	Black
		4.5 : 4.5 : 1	23.1	4.0	1.1	Dark grayish black
	120	4 : 3 : 3	22.8	1.8	0.0	Black
		3 : 4 : 3	22.6	2.0	-0.5	Black
		3 : 3 : 4	22.7	1.3	-0.9	Black
		1 : 4.5 : 4.5	21.9	2.6	-3.3	Dark grayish purple
		4.5 : 1 : 4.5	22.3	0.2	-0.7	Black
		4.5 : 4.5 : 1	24.8	9.3	3.8	Dark grayish reddish brown

[a] Y: Yellow, R: Red, B: Blue

3:3:4, absorption behaviors of all the samples were similar showing high K/S values in the range of 440 to 640 nm. Lightness (L^*) of all the three samples were similar (22.0-22.2) and ISCC-NBS color names were black as shown in Table 3. When the combination ratio was 1:4.5:4.5, 4.5:1:4.5, and 4.5:4.5:1, absorption behaviors were markedly different to each other because difference among the dye concentrations became larger. Sample for 1:4.5:4.5 showed low K/S values at 360-440 nm while K/S values of the sample for 4.5:4.5:1 were low at 580-720 nm. In Table 3, samples for 1:4.5:4.5 and 4.5:1:4.5 were black color showing lower L^* values (21.8-21.9) than those for 4:3:3, 3:4:3, and 3:3:4. On the other hand, sample for 4.5:4.5:1 in which small amount (1% owf) of blue dye was used, showed high L^* value (23.1) and color was not neat black but grayish black. From these results, it was found that lightness on PET fabric was dependent upon the combination ratio of disperse dye concentration used and that, in order to obtain black color, blue dye should be added enough.

From the color values (L^*, a^*, and b^*) and color name in Table 3, dyeing temperature was also found to be important for black dyeing. All the Samples dyed at 120℃ showed higher L^* values (whiter) than those dyed at 130℃ except the sample for 1:4.5:4.5 which

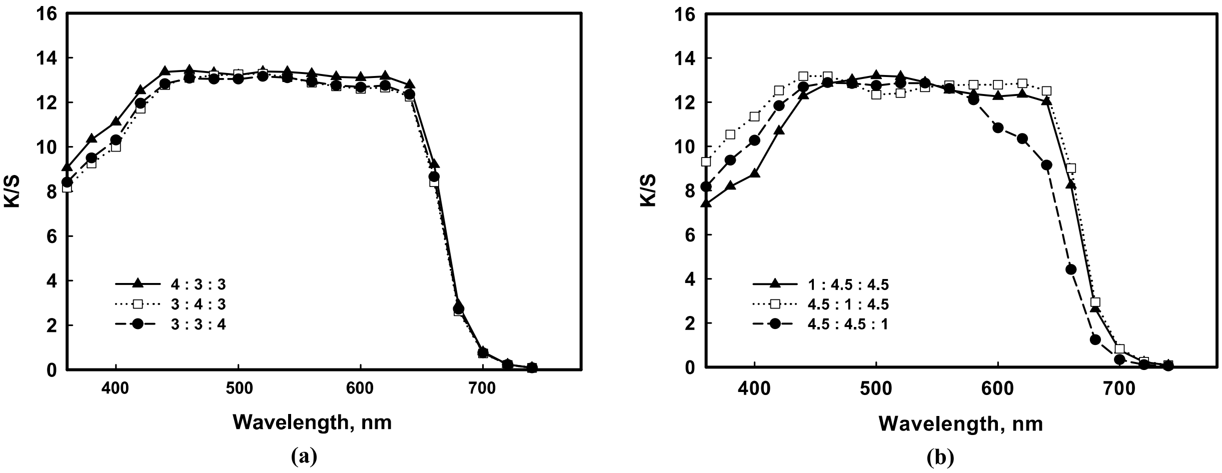


Fig. 3. K/S values in the visible range of PET fabric dyed at 130℃ with mixture of SE-type disperse dyes of three primary color. (a) combination ratio of dye concentraion [yellow:red:blue], 4:3:3, 3:4:4, and 3:3:4% owf; (b) combination ratio of dye concentraion [yellow:red:blue], 1:4.5:4.5, 4.5:1:4.5, and 4.5:4.5:1% owf.

Table 4. The effect of dyeing temperature and combination ratio of dye concentration on color properties of PET fabric dyed with SE-type (medium energy level range) disperse dyes

Energy type	Dyeing temp. (℃)	Combination ratio of dye concentration (o.w.f.) [Y:R:B][a]	CIE $L^*a^*b^*$ values			ISCC-NBS Color name
			L^*	a^*	b^*	
SE	130	4 : 3 : 3	22.2	1.6	0.2	Black
		3 : 4 : 3	22.5	2.3	0.0	Black
		3 : 3 : 4	22.5	2.0	0.1	Black
		1 : 4.5 : 4.5	22.7	2.6	-0.3	Black
		4.5 : 1 : 4.5	22.7	0.8	0.7	Black
		4.5 : 4.5 : 1	23.4	5.2	1.4	Dark grayish black
	120	4 : 3 : 3	23.9	3.7	2.1	Dark grayish reddish brown
		3 : 4 : 3	23.5	5.9	0.0	Dark grayish purple
		3 : 3 : 4	23.4	4.4	0.8	Dark grayish red
		1 : 4.5 : 4.5	23.3	5.8	-0.6	Dark grayish purple
		4.5 : 1 : 4.5	24.1	1.6	2.7	Dark grayish brown
		4.5 : 4.5 : 1	24.5	7.9	2.8	Dark grayish red

[a] Y: Yellow, R: Red, B: Blue

showed the same L^* value but color of the sample at 120℃ was grayish purple not black as a^* and b^* values were big. Therefore, it will be desirable to dye PET fabric at high temperature for deep black color.

Fig. 3 shows K/S values in the visible range of black dyed fabrics and Table 4 shows their color values and color name for SE-type disperse dyes. In Fig. 3, the trend of absorption behavior was similar to that for S-type disperse dyes (Fig. 2). In table 4, similarly, lightness was dependent upon the combination ratio of dye concentration and sample dyed at 130℃ with small amount of blue dye (4.5:4.5:1) did not show black shade. When compared to the samples dyed with S-type dye, L^* values of the samples dyed with SE-type dye were generally higher and all the samples dyed at 120℃ were not neutral black color. From these results, it can be concluded that S-type disperse dyes should be chosen rather than SE-type ones for ideal black color.

3.3 Compatibility of dyes

In practical dyeing, dye mixtures rather than single dyes are commonly used and it is important to select dyes having similar properties or showing good compatibility in order to produce uniform dyeing[9]. Thus, dyeing compatibility of the disperse dyes used was evaluated by measuring dyeing rate of each single dye. It is well known that dyeing behavior of one dye is not affected by the presence of the other dye when two kinds of disperse dyes participate in dyeing[10].

Therefore, we can consider dyeing rate of single dye as dyeing rate of the dye in mixture. Fig.4 and 5 show relative exhaustion of S-type and SE-type dyes of three primary color on PET fabric according to dyeing time or dyeing temperature.

As high concentration of disperse dyes is used for black dyeing, the effect of dye concentration on dyeing

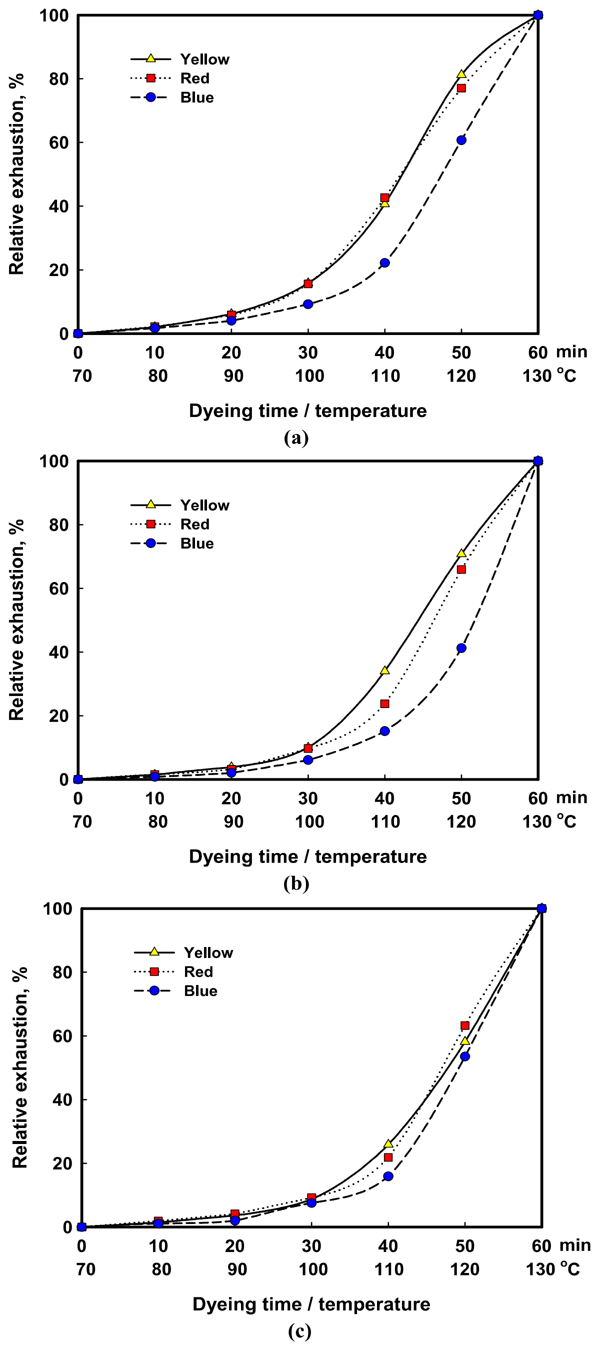


Fig. 4. Relative dyeing rate and compatibility of S-type disperse dyes of three primary color on PET fabric. (a) dye concentraion : yellow 1%, red 1%, and blue 1% owf; (b) yellow 2%, red 2%, and blue 2% owf; (c) yellow 3%, red 3%, and blue 3% owf.

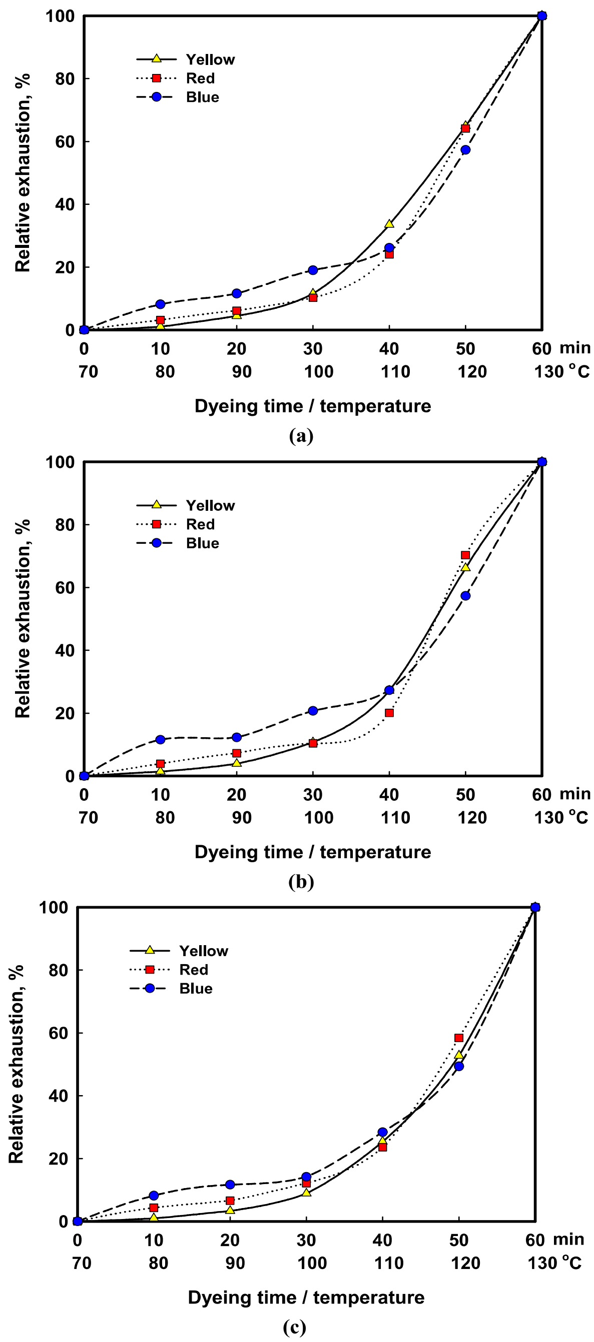


Fig. 5. Relative dyeing rate and compatibility of SE-type disperse dyes of three primary color on PET fabric. (a) dye concentraion : yellow 1%, red 1%, and blue 1% owf; (b) yellow 2%, red 2%, and blue 2% owf; (c) yellow 3%, red 3%, and blue 3% owf.

rate and, subsequently, compatibility of the dyes was also investigated. In Fig. 4, relative dyeing rate of blue dye was slower than that of yellow or red dye when dye concentration is not so high. However, when high concentration (3% owf) of dye was used, relative dyeing rate became similar to one another. This can be explained by that, as dye concentration increases, dye exhausted at higher temperature which suggests relative dyeing rate should be slower[10]. Relative dyeing rate of yellow and red dye became slower as their concentration increased while that of blue dye maintained, which led the relative dyeing rate of all the dyes becoming similar. In Fig. 5, relatvie dyeing rate of SE-type disperse dyes at high concentration was also similar to one another. From these results, disperse dyes of three primary color with both type (S, and SE) were found to show generally good compatibility at high concentration of dye.

3.4 Fastness properties

Table 5 shows the wash and light fastness of black dyed PET fabric dyed with mixture of three primary color. In case of washfastness, color change and staining to cotton, polyester, and acryl were good to excellent for all the samples. However, staining to acetate and nylon was fair to moderate (2-3 to 3 grade) and staining to wool was moderate for S-type dyes. Samples from SE-type dyes showed worse wash-fastness than those from S-type dyes in acetate, nylon, and wool staining by 0.5 to 1 grade with some exceptions. Lightfastness was generally moderate.

4. CONCLUSIONS

When PET fabric was dyed in black shade with the mixture of disperse dyes of three primary color, fabric showed high K/S values from 440 to 640 nm.

Lightness of black dyed fabric was found to be dependent upon the combination ratio of dye concentration. Amoung the three primary color, blue dye should be used in large amount for black color, while red or yellow dye could be added in small amount on condition that other two dyes would be used enough. It was also found that S-type disperse dyes were more appropriate than SE-type dye and 130℃ dyeing was desirable to obtain deep black color.

S- and SE-type disperse dyes of three primary color showed generally good dyeing compatibility especially at high dye concentration. In washfastness, staining to acetate, nylon, and wool fabric was fair to moderate and samples dyed with S-type dyes showed slightly better washfastness than those dyed with SE-type dyes. Lightfastness was generally moderate.

Although washfastness was fair to moderate and lightfastness was moderate, this level can be accepted

Table 5. The wash and light fastness of black dyed PET fabric(dyeing at 130℃ for 60 min)

Energy type	Combination ratio of dye concentration (o.w.f.) [Y:R:B][a]	Wash fastness							Light fastness
		Change	Staining						
			Acetate	Cotton	Nylon	Polyester	Acryl	Wool	
S	4 : 3 : 3	4-5	2-3	5	2-3	4-5	5	3	4
	3 : 4 : 3	4-5	2-3	5	2-3	4-5	5	3	4
	3 : 3 : 4	4-5	2-3	5	2-3	4	5	3	4
	1 : 4.5 : 4.5	4-5	2-3	5	2-3	4	5	3	4
	4.5 : 1 : 4.5	4-5	2-3	5	3	3-4	5	3	4
	4.5 : 4.5 : 1	4-5	2-3	5	2-3	3-4	5	3	4
SE	4 : 3 : 3	4-5	2	5	2	4-5	5	3	4
	3 : 4 : 3	4-5	2	5	1-2	4	5	2-3	4
	3 : 3 : 4	4-5	2	5	2	4-5	5	3	4
	1 : 4.5 : 4.5	4-5	1-2	5	1-2	4	5	2-3	3-4
	4.5 : 1 : 4.5	4-5	3	5	2-3	4-5	5	4	4
	4.5 : 4.5 : 1	4-5	2-3	5	2	4-5	5	3	3-4

commercially once the dyed fabric shows deep black color. Thus, from this study, we could find optimum black dyeing condition using minimum amount of dyes in commercial dyeing process. It is expected that we can save large amount of dyes and also minimize the discharge of unfixed dye into dyehouse effluent.

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