



THE DIFFERENCE IN COLOUR OF POLYESTER FIBRES
CAUSED BY VARIOUS pH VALUES OF DISPERSE
DYE-LIQUOR p-PHENYLAZOANILINE - PHENOL

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Abstract. The pH value of disperse dye-liquor is one of the factors which affect the reactions in the dye (p-phenylazoaniline-phenol) - substrate (polyester fibres) system as well as the dye exhaustion and fixation. Provided that all other conditions are maintained constant and only the pH value is changed, the colour of polyester fibres will be affected. This effect is manifested through the total difference in colour DE, i.e. in the part of colour hue DH differences, in the part of colour purity DC differences, and in the part of colour lightness DL differences. The colour measurement of the dyed samples is done by reflection spectrophotometry Update Colour Eye 3000 by ICS - TEXICON (England), programme Super MATCH 6.

1. Introduction

The influence of the dyeing solution pH value reflects on the reactions taking place in the dye-substrate system as well as on the exhaustion and fixation of the dye. The increase of the pH of the solution (dye liquor) is followed by the increase of the reaction speed. However, for pH values above 8.00 the decrease of exhaustion is noticed, with consequent lower dye-fixing ability of the fibre. Taking this effect into account, it is necessary to choose such a pH value of the dye-liquor which can provide suitable chemical reactions taking place during the expected dyeing time [1].

According to their chemical composition, disperse dyes fall into three groups: azo compounds, nitrodiphenylamine compounds and anthraquinone compounds.

The group of azo disperse dyes comprises a great number of monazo dyes and a great number of diazo dyes. The distinctive representative of diazo dyes is p-phenylazoaniline-phenol, with the commercial name of S.R.A. Fast Golden Yellow XIII; then Ostacet Yellow E-L5R. S.R.A.,

which is the abbreviation for sulphoricinoleic acid, being the first dispersion agent later substituted by similar synthetic products. In the Colour Index, the p-phenylazoaniline-phenol dye is listed as Disperse Yellow 23 (Reddish Yellow). This dye is produced in Czechoslovakia and its structural formula used in the paper is given in Fig. 1.

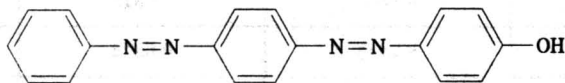


Fig. 1. Structural formula of dye p-phenylazoaniline-phenol (S.R.A. Fast Golden Yellow XIII, Ostacet Yellow E-L5R., C.I. Disperse Yellow 23)

Disperse dyes are used for the dyeing of synthetic fibres such as nylon, triacetate, polyester, acryl. The polyester fibre is the product of the condensation of ethylene glycol and terephthalic acid and its commercial name is terylene or Dacron [2].

2. Experimental

The dyeing of polyester fibres with C.I. Disperse Yellow 23 was done by the exhaustion method on the discontinuous HT pressure dyeing machine AHIBA TEXOMAT model G VII (dyeing at the temperature of 393-408 K, under pressure) [3]. The process was performed in the dye-liquor containing 0.5 - 1 g/dm³ of the dispersion agent SETAMOL WS (BASF, Germany), and the adequate quantity of the acetic acid (30%) or sodiumhydroxide solution for attaining the expected pH value of 3.50 to 13.00. The polyester fibres were treated for a few minutes in this dye-liquor at the temperature of 333 to 353 K, and then the temperature was raised to 403 K and the process of dyeing was continued for 90 minutes. The polyester fibres were finally rinsed and aftertreated. Table I contains all the data related to the preparation of the standard sample dyed at the solution pH value of 4.50 as well as to the preparation of the other test samples. The percentage of the Disperse Yellow 23 dye in the dye-liquor is the same for all of the samples, and amounts to 1% of the undyed substrate mass. The quantity of the dye to be measured for the preparation of the adequate solution is obtained according to the formula:

$$\frac{C \text{ of dye (\%)} \cdot \text{fibre mass (g)}}{100} = \text{quantity of dye to be measured}$$

The solution ratio is 1:30 (1 stands for the mass of the sample to be dyed, while 30 stands for the quantity of the water to be added). This is a constant ratio both for the standard and the test samples.

Table I. Data concerning preparation of dyed polyester fibres samples

No. of samples	Mass of undyed polyester fibres (g)	Mass of dye (g)	Dye-liquor pH value
1. TEST	8.1465	0.0815	3.50
2. STANDARD	8.7413	0.0874	4.50
3. TEST	7.7003	0.0770	6.00
4. TEST	9.4040	0.0940	8.000
5. TEST	9.4080	0.0941	10.00
6. TEST	9.1230	0.0912	13.00

The colorimetry of the dyed polyester fibres was done on the spectrophotometer COLOR EYE 3000, a component part of the equipment also comprising a computer system (Single User System IBM PC) and a computer colour measurement programme by ICS-TEXICON (England).

3. Results and discussion

Reflectance values decrease with the increase of the dye quantity on the fibre, since the dye absorbs more light with the increase of dye concentration. Different colours absorb the light of various wavelengths with different intensity. Using the basic equations (1) and (2) of Kubelka-Munk theory, the log K/S is obtained as dependent on the wavelength in the visible spectrum (400 - 700 nm) for the dyed standard as well as the test samples (Fig. 2). Curve 2 in the Figure refers to the standard sample, while Curves 1, 3, 4, 5 and 6 refer to the dyed test samples. Curves 1, 3 and 4 are very close to Curve 2 in the given coordination system. Consequently, the conclusion is that the values of the log K/S as dependent on λ for the samples dyed at the pH values of 3.5; 6.00; 8.00 are very close to those corresponding to the standard sample dyed at pH 4.50.

$$G = \frac{K}{S} (\text{dyeing}) - \frac{K}{S} (\text{substrate}) = BC \quad (1)$$

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (2)$$

Where

G is the dye absorption for a certain wavelength

K is the absorption coefficient for a certain wavelength

S is the scattering coefficient for a certain wavelength (valid only for the substrate)

R is the measured reflectance value

B is the proportional constant depending on the dye, the wavelength, the substrate and the dyeing conditions; within the bright and the mean region of dyeing, B is also dependent on the dye concentration

C is the quantity of dye.

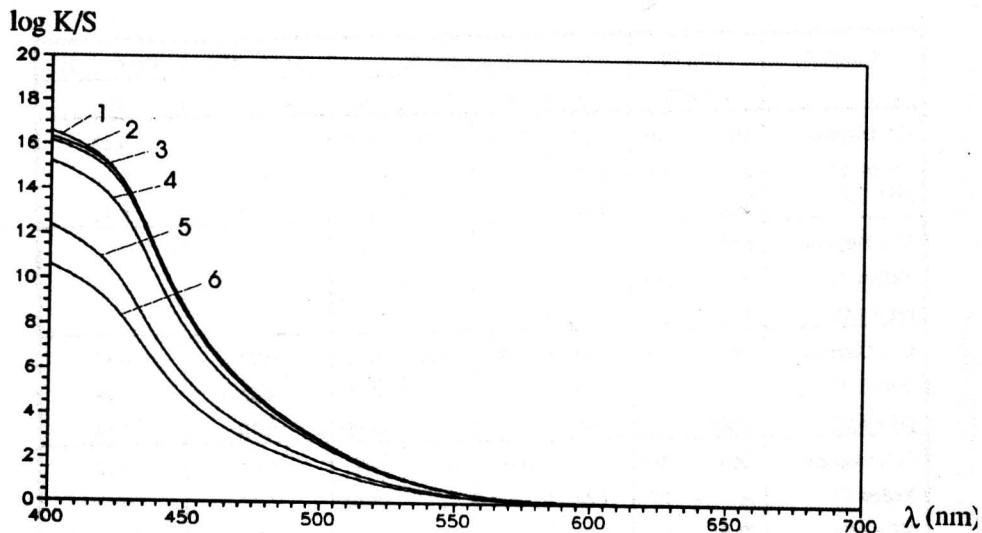


Fig. 2. Dependence log K/S from wavelength in visible region dyed with p-phenylazoaniline-phenol (C.I. Disperse Yellow 23) with dye liquor pH: 1) 3.50; 2) 4.50; 3) 6.00; 4) 8.00; 5) 10.00 and 6) 13.00

The colour coordinate values for all of the dyed samples according to the CIELab 76 measuring system [4], for all of the three types of light (D65 - 10'; A - 10'; TL 84 - 10'), are given in Table II. Provided all other dyeing conditions remained the same, different pH values of the dye-liquor C.I. Disperse Yellow 23 cause different colour coordinate values in the CIELab 76 measuring system. This difference in coordinates also effects the difference in colouring between the standard and the test samples. The difference in colour between the standard and the test samples is given in Table III.

For practical purposes, besides the precise colour definition and the established differences between the standard and the test samples, it is also necessary to define the limits of permitted tolerances. The CIELab 76 system gives a total colour difference DE, but it does not state the precise tolerance limits.

The M&S 83 A system defines the permitted deviations in colour: if the total colour difference of the DE standard and test samples is lower than 1.2, then the deviation is acceptable; the border-line cases are between 1.2 and 1.5, while above 1.5 the difference in colour can not be tolerated.

Table II. Colour coordinates according to CIELab 76 measuring system for colour of polyester fibres samples dyed by dye-liquor of p-phenylazoaniline-phenol (C.I.Disperse Yellow 23) at various pH values

SAMPLE	TYPE OF LIGHT	C I E L ab 76				M&S 83A	CMC (2:1)
		DE	DH	DC	DL	DE	DE
C.I. Disperse Yellow 23 pH = 3,5	D65 - 10'	0,25	-0,14	0,20	-0,06	0,15	0,12
	A - 10'	0,25			-0,04	0,11	0,10
	TL84 - 10'	0,28			-0,05	0,17	0,14
C. I. Disperse Yellow 23 pH = 4,50	D65 - 10'						
	A - 10'						
	TL84 - 10'						
C. I. Disperse Yellow 23 pH = 6,00	D65 - 10'	0,94	-0,67	-0,20	-0,61	0,70	0,55
	A - 10'	0,84			-0,56	0,59	0,48
	TL84 - 10'	0,99			-0,61	0,70	0,54
C. I. Disperse Yellow 23 pH = 8,00	D65 - 10'	1,70	0,00	-1,70	0,00	0,43	0,56
	A - 10'	1,80			-0,10	0,44	0,57
	TL84 - 10'	1,80			0,00	0,42	0,56
C. I. Disperse Yellow 23 pH = 10,00	D65 - 10'	7,90	3,23	-6,80	2,40	4,00	3,40
	A - 10'	8,10			1,70	2,70	2,80
	TL84 - 10'	7,80			2,20	3,80	3,20
C. I. Disperse Yellow 23 pH = 13,00	D65 - 10'	9,90	3,95	-8,10	4,10	4,80	4,20
	A - 10'	9,90			3,30	3,30	3,50
	TL84 - 10'	9,70			3,90	4,50	3,90

According to the CMC (2:1) system, the permitted colour deviation limit is 1.4. If the total colour difference between the standard and the test samples is lower than 1.4, the deviation is acceptable and vice versa [5].

On the basis of the established total colour differences DE, the samples dyed in the C.I.Disperse Yellow 23 dye-liquor at the pH values of 3.50, 6.00 and 8.00 are acceptable according to the M&S 83 A and CMC (2:1) systems, while the samples dyed in dye-liquors at the pH values of 10.00 and 13.00 are rejected. The sample dyed at the pH value of 4.50 is taken as the standard.

In the rectangular coordinate system where (a) is the red-green and (b) yellow-blue coordinate, the graphical position for each of the dyed samples is obtained. The graphical position

for each of the dyed samples is also obtained on the lightness axis L. All the gray tones are on this axis, from white (L=100) to black (L=0).

Table III. The difference in colour of polyester fibres dyed with dye-liquor of p-phenylazoaniline-phenol (C.I.Disperse Yellow 23) at different pH values according to CIELab 76; M&S 83A and CMC (2:1) measuring systems

SAMPLE (SPECIMEN)	TYPE OF LIGHT	COLOUR COORDINATE ACCORDING TO CIELab 76 SYSTEM									
		X	Y	Z	a	b	L	C	h	x	y
C.I. Disperse Yellow 23 pH = 3,50	D65 - 10' A - 10' TL84 - 10'	47,79 68,89 57,47	41,01 49,96 46,18	6,53 2,43 3,72	26,5 29,6 24,0	69,9 76,7 77,8	70,2 76,0 73,7	74,8 82,2 81,4	69 69 73	0,501 0,568 0,535	0,4302 0,4119 0,4301
C. I. Disperse Yellow 23 pH = 4,50	D65 - 10' A - 10' TL84 - 10'	47,82 68,90 57,46	41,10 50,03 46,26	6,59 2,45 3,75	26,3 29,4 23,8	69,8 76,5 77,6	70,2 76,1 73,7	74,6 82,0 81,2	69 69 73	0,500 0,576 0,534	0,4303 0,4122 0,4304
C. I. Disperse Yellow 23 pH = 6,00	D65 - 10' A - 10' TL84 - 10'	47,09 67,99 56,61	40,24 49,12 45,32	6,44 2,39 3,67	26,8 29,9 24,4	69,4 76,2 77,1	69,6 75,5 73,1	74,4 81,9 80,9	69 69 72	0,502 0,568 0,536	0,4291 0,4110 0,4292
C. I. Disperse Yellow 23 pH = 8,00	D65 - 10' A - 10' TL84 - 10'	47,68 68,57 57,25	41,15 49,94 46,25	7,02 2,60 4,01	25,7 29,0 23,3	68,2 74,8 75,9	70,3 76,0 73,7	72,9 80,2 79,4	69 69 73	0,497 0,566 0,532	0,4293 0,4124 0,4302
C. I. Disperse Yellow 23 pH = 10,00	D65 - 10' A - 10' TL84 - 10'	49,60 70,33 59,23	44,62 52,87 49,69	9,22 3,40 5,26	20,8 24,9 18,6	64,6 70,0 72,2	72,6 77,8 75,9	67,8 74,3 74,5	72 70 76	0,479 0,555 0,518	0,4314 0,4176 0,4352
C. I. Disperse Yellow 23 pH = 13,00	D65 - 10' A - 10' TL84 - 10'	52,03 73,46 62,05	47,30 55,67 52,49	10,55 3,87 6,03	19,8 24,2 17,8	63,5 68,7 71,1	74,4 79,4 77,6	65,5 72,8 73,3	73 71 76	0,473 0,552 0,514	0,4304 0,4186 0,4354

The graphical position in the a/b coordinate system and on the L lightness axis of the sample dyed in the dye-liquor at the pH value of 8.00 is given in Fig. 3.

The position of all dyed samples (of the standard marked as 0 as well as of all the tests) in the CIELab CLOUD PLOT system is given in Fig. 4. This Figure shows that the test samples dyed at pH: 3.50; 6.00 and 8.00 are identical in lightness with the standard sample dyed at pH 4.50. The test samples dyed at pH 10.00 and 13.00 (1 and 2 in Fig. 4) differ in lightness, both when they are compared to each other and when the two of them are compared to the standard sample. Concerning Chroma and Hue, the test samples dyed at pH 3.50 and 6.00 are identical with the standard sample dyed at pH 4.50. The test sample dyed at pH: 8.00; 10.00 and 13.00 (marked as 1, 2 and 3 in Fig. 4) differ in lightness when compared between themselves and when they are all compared to the standard sample marked as 0 according to Chroma and Hue.

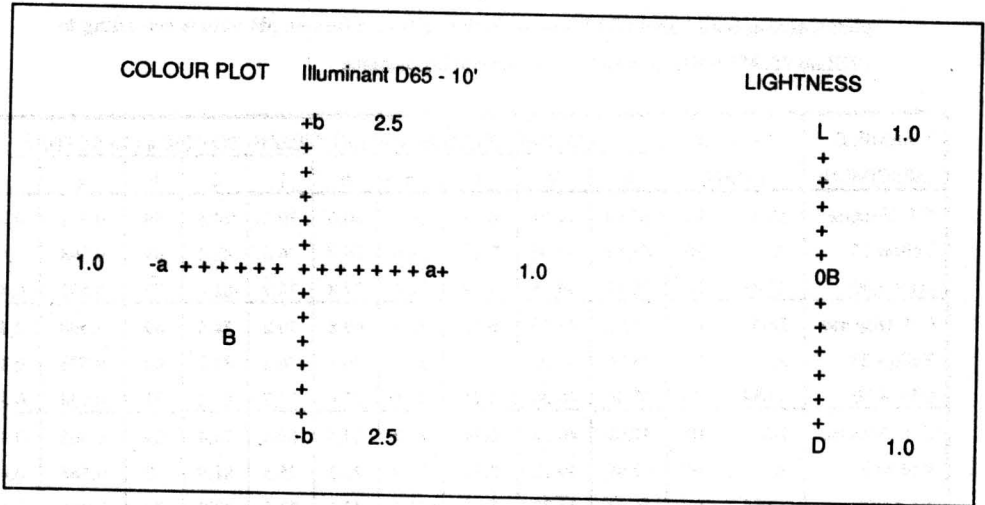
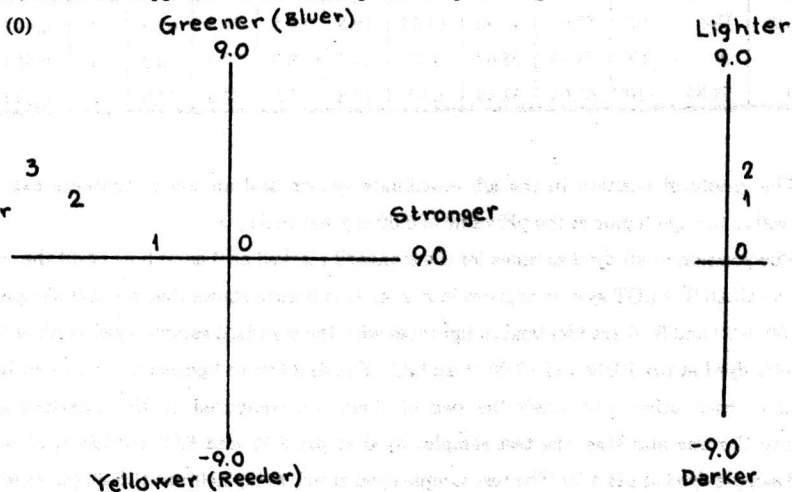


Fig. 3. Test-sample position (B) dyed with dye liquor of p-phenylazoaniline-phenol (C.I. Disperse Yellow 23) where pH is 8.00 at coordination system a/b and on light axis L, where it is apparent that test-sample B is equal in lightness with standard sample



CHROMA AND HUE

LIGHTNESS

Fig. 4. COLOUR CLOUD BATCH DATA

Labels for Lightness display

0	C.I.D. YELLOW	23	pH=4.50	C.I.D. YELLOW	23	pH=3.5 0
	C.I.D. YELLOW	23	pH=8.00	C.I.D. YELLOW	23	pH=6.0 0
1	C.I.D. YELLOW	23	pH=10.00			
2	C.I.D. YELLOW	23	pH=13.00			

Labels for Chroma and Hue display

0	C.I.D. YELLOW	23	pH=4.50	C.I.D. YELLOW	23	pH=3.5 0
				C.I.D. YELLOW	23	pH=6.0 0
1	C.I.D. YELLOW	23	pH=8.00			
2	C.I.D. YELLOW	23	pH=10.0 0			
3	C.I.D. YELLOW	23	pH=13.0 0			

4. Conclusion

The aim of this investigation was to determine the optimal range of the dye-liquor pH value of C.I. Disperse Yellow 23, which would provide appropriate chemical reactions taking place during the expected dyeing time of polyester fibres. For the tested dye p-Phenylazoaniline-Phenol (C.I. Disperse Yellow 23) this range is between pH 3.50 and pH 8.00. The colours obtained within this interval of the dye-liquor pH values and in the concentration of 1% from the substrate weight are acceptable both according to M&S 83 A and CMC (2:1) systems.

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RAZLIKA U OBOJENJU POLIESTARSKIH VLAKANA
IZAZVANA RAZLIČITIM pH VREDNOSTIMA RASTVORA
DISPERZNE BOJE p-FENILAZOANILIN - FENOL

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Izvod. pH vrednost rastvora disperzne boje je jedan od faktora koji se odražava na reakcije koje se odigravaju u sistemu boja (p-fenilazoaniline-fenol) - supstrat (poliestarska vlakna), kao i na iscrpljenje i vezivanje boje. Ako se svi ostali uslovi bojenja održavaju konstantnim, a menja se samo pH vrednost rastvora boje to će se odraziti na obojenje poliestarskih vlakana. Ovaj efekat se manifestuje kroz ukupnu razliku u boji DE, tj. u razlikama u delu tona boje DH, u delu čistoće boje DC i u delu svetline boje DL. Metrika obojenih uzoraka vršena je na refleksionom spektrofotometru Update Colour Eye 3000 firme ICS - TEXICON (England), koji radi po programu Super MATCH 6.