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Scientific paper

INFLUENCE OF APPLYING THE DIFFERENT ACIDS FOR ADJUSTING THE DYEBATH PH IN THE PROCESS OF DYEING THE POLYESTER KNITWEAR WITH DISPERSE YELLOW 3

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Abstract

The influence of using formic, oxalic, citric and tartaric acid for adjusting the dyebath pH upon the dyeability of polyester knitwear dyed with Disperse Yellow 3 was investigated. CIELAB coordinates of the knitwear samples dyed with the addition of tested acids were assessed and compared to those dyed with the addition of acetic acid. The differences in dyeabilities obtained with the addition of citric and oxalic acid are acceptable according to both M&S 83A and CMC (2:1) standard and the difference in dyeability obtained with the addition of tartaric acid is acceptable according to CMC(2:1) standard in comparation to the dyeability obtained with the addition of acetic acid, at dye concentration of 1%. For the dye concentration of 2%, acceptable difference in dyeability, in comparation to the dyeability obtained with the addition of acetic acid, according to both M&S 83A and CMC (2:1) standard, was obtained with the addition of tartaric acid.

Key words:*Polyester, dyeing, disperse dye, pH, acids.*

1. INTRODUCTION

Polyester fabrics are the most widely used sintetic fabrics due to their excellent textile properties and high chemical stability. Polyester fabrics have a hydrophobic nature and a hightly compact structure [1,2] and they are dyed using disperse dyes, at high temperatures (usually in the range of 115 – 135 ºC) and high pressure. Disperse dyes are essentialy non – ionic dyes, exhibiting poor sollubility in water and therefore they are applied in the form of water dispersion [1, 3, 4].

Dyeing of polyester fabric in the water dyebath by the exhaustion process is carried out in a slightly acidic medium. Polyester fibres are resistant to dilute aqueous acids and alkalis and the pH value does not have a crucial impact on the dyeing mechanism, but many disperse dyes degrade if the pH is uncontrolled during aqueous dyeing; some disperse dyes have hydrolysable groups in their molecules, which makes them particulary sensitive to hydrolysis, especially in alkaline medium. The hydrolysed form of the dye could be of different shade and in some cases of different affinity for polyester fibres to the unhydrolysed dye. Thus, in order to minimize the possibility of dye hydrolysis, the dyeing is carried out in slightly acidic medium, usually in the pH range of 4.5 – 5.5.

According to literature data, acetic acid is generally used for adjusting the dyebath pH value, although a buffer sistem containig formic acid and amonnium sulfate is used as well [1,3,5,6].

In this paper, the influence of using formic, oxalic, citric and tartaric acid for adjusting the dyebath pH was investigated upon the dyeability of polyester knitwear, dyed with disperse dye Disperse Yellow 3 (*p*-aminoacetanilide→ *p*-cresol) [7]. Colour differences between the samples dyed with the addition of tested acids and one dyed with the addition of acetic acid were measured using the technique of reflectiometry and expressed in terms of CIELAB coordinate values and K/S values. For each of the tested acids, colour difference acceptability was determined in comparation to the colour obtained with the addition of acetic acid, according to M&S 83A and CMC (2:1) standards.

2. EXPERIMENTAL

Polyester knitwear (100% PET), produced by Nitex Niš (Serbia) was used (Table 1).

Table 1. Characteristics of the undyed polyester knitwear

Polyester knitwear properties	
Horizontal density	15 cm^{-1}
Vertical density	18.5 cm^{-1}
Square meter mass	130 gm^{-2}
Shrinking in the process of boiling:	
in lenght	1 %
in width	1,5 %

A disperse dye, CI Disperse Yellow 3 was purchased from Chemapol (Czech Republic) and used without further purification.

Acetic acid was used for adjusting the dyebath pH value of the standard sample and formic, citric, oxalic and tartaric acid were used for adjusting the dyebath pH of the test samples. All acids were purchased from Sinex Laboratory (Belgrade, Serbia).

The polyester knitwear was scoured in a bath containing 1g/L sodium carbonate, wetting agent and scouring agent (Jugopon 50) at 70°C for 30 min. After scouring, the knitwear was rinsed with cold water. The last tracks of the scouring liquor were neutralised by adding 0.1 g/L acetic acid during the last rinsing circile.

The degree of whiteness of polyester knitwear prepared as described was 62.80% according to C.I.E ’82 system for the light source D65 and 79.20% according to Berger for the light source C2. (Figure 1, Table 2).

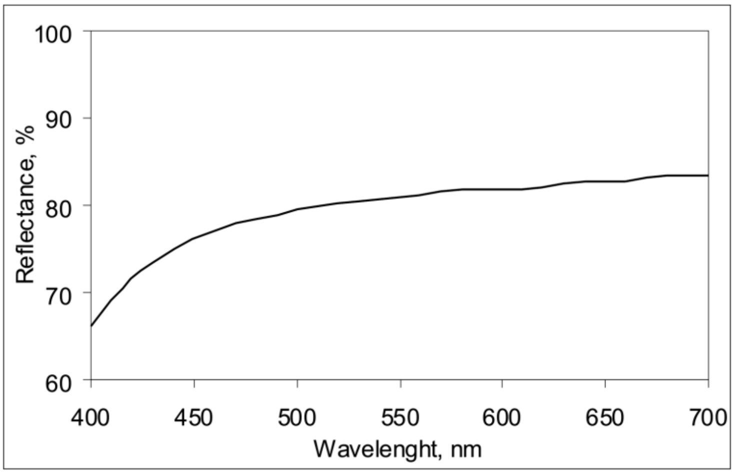

Figure 1. The reflection curve of the undyed polyester knitwear in the visible spectral range (λ = 400 – 700nm).

Table 2. CIELAB coordinates of the undyed polyester knitwear

Light source	L	A	B	C	H	X	Y	Z	R	B	x	y
D65-10’	92.02	-0.40	3.85	3.87	95.99	76.36	80.75	81.39	81.71	75.85	0.3202	0.3386
A-10’	92.24	0.70	3.80	3.87	79.51	90.72	81.25	26.89	82.93	76.38	0.4562	0.4086
TL84-10’	92.16	-0.04	4.24	4.24	90.60	84.17	81.05	49.65	82.38	75.65	0.3917	0.3772

Dyeing of polyester knitwear was carried out by an exhaustion process at high temperature and high pressure, using a laboratory dyeing machine AHIBA TEXOMAT (GVII) at a liquor – to – goods ratio of 30:1, in the dyebath containing disperse dye with the concentration of 1% owf (series 1) and 2% owf (series 2) and 1g/L kortamol NNO as a dispersing agent (for both series). The pH of the dyebath was adjusted to 4.5 using acetic acid for standard sample and formic, citiric, oxalic and tartaric acid for the test samples. The pH was mesured using pH meter “RADIOMETER Type PHM 29” with combined electroce GK 2311C.

Dyeing began at the dyebath temperature of 50°C. The dyebath was held at this temperature for 40 min and then the temperature was raised to 135°C and dyeing was carried out for for 60 min at this temperature. After the dyeing process was finished, the temperature was reduced to 90°C and the knitwear samples were removed and washed with warm water containing 1g/L Jugopon 50 at 70°C. The samples than were rinsed warm and then in cold water until neutral and dried at room temperature.

To investigate the influence of the tested acids on the polyester knitwear, a series of the undyed knitwear samples was prepared. The knitwear samples were treated in the bath containing one of the tested acids at pH 4.5 under the same bath conditions as described above, but this time without the presence of a dye in the bath.

The reflectance spectra of the dyed and undyed, but treated polyester kintwear samples were recorded with an UPDATE COLOR EYE 3000 spectrophotometer (ICS – TEXICON), at the daylight (D65). For the dyed samples, they were expressed in terms of K/S values in the visible spectral range (λ = 400 – 700 nm). The colour properties of the samples were expressed in the terms of CIELAB values and the CIELAB coordinates were estimated applying the metric program“ Super Match 6 Supplement”.

3. RESULTS AND DISCUSSION

The influence of the tested acids on K/S values of the dyed polyester knitwear samples is shown in Figure 2 and Figure 3, where the K/S values of the dyed samples in the visible spectral range are presented. The K/S values are correlated with the reflectance R according to the Kubelka – Munk equation [8, 9]. The color properties of the dyed knitwear samples, as expressed in terms of the CIELAB system are listed in Table 3, and the total colour differences (ΔE units), according to CIELAB, M&S 83A and CMC (2:1) standardbetween the test samples and the standard sample are listed in Table 4.

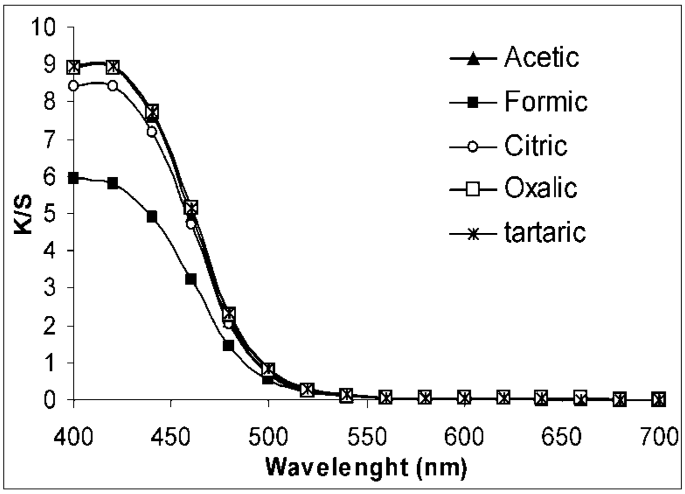

Figure 2. K/S values in the visible spectral range of the polyester knitwear dyed with Disperse yellow 3 with the additon of different acids at the dye concentraion of 1% owf.

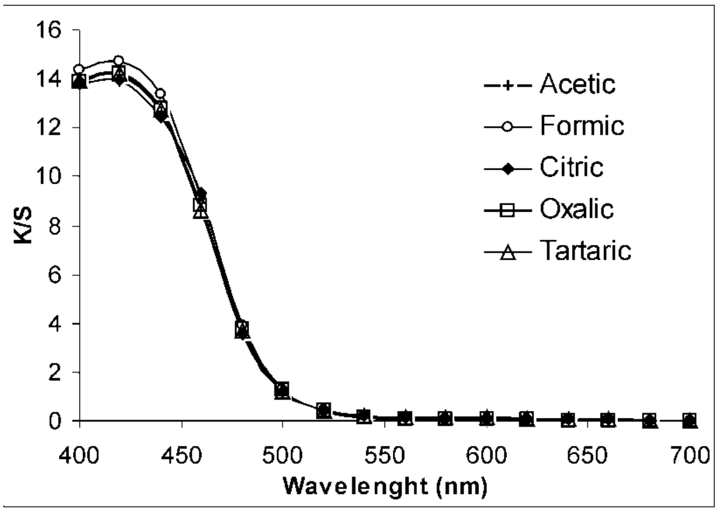

Figure 3. K/S values in the visible spectral range of the polyester knitwear dyed with CI Disperse yellow 3 with the additon of different acids at the dye concentraion of 2% owf.

Table 3. CIELAB coordinates of the samples dyed with Disperse yellow 3 with the addition of different acids

Acid	Dye concentration (% owf)	CIELAB coordinates										
		X	Y	Z	a	b	L	C	h	x	y	R
Acetic	1	58.03	59.67	10.39	3.57	76.55	81.66	76.63	87.33	0.4530	0.4659	74.09
	2	48.38	49.66	6.90	3.58	78.27	75.86	78.35	87.38	0.4610	0.4733	62.17
Formic	1	59.77	62.29	14.01	1.70	69.34	83.07	69.36	88.60	0.4392	0.4578	75.53
	2	50.80	51.08	6.56	6.40	81.11	76.73	81.36	85.49	0.4684	0.4711	65.44
Citric	1	58.20	60.00	10.77	3.24	75.72	81.84	75.79	87.55	0.4513	0.4652	74.23
	2	52.01	52.53	7.05	5.85	80.69	77.60	80.90	85.85	0.4661	0.4708	66.92
Oxalic	1	56.89	58.29	10.06	4.06	76.21	80.90	76.32	86.95	0.4543	0.4654	72.66
	2	54.22	53.88	6.76	8.14	83.16	78.39	83.56	84.41	0.4720	0.4691	69.90
Tartaric	1	57.33	58.04	9.92	5.73	76.39	80.76	76.61	85.71	0.4576	0.4632	73.28
	2	47.66	48.80	6.85	3.90	77.52	75.33	77.61	87.12	0.4613	0.4724	61.23

Table 4. Colour differences according to CIELAB, M&S 83A and CMC (2:1) system between the test samples and standard sample, dyed with the addition of acetic acid

Acid	Dye conc. (% owf)	CIELAB							M&S 83A	CMC (2:1)			
		ΔE	ΔH	ΔC	ΔL	Δa	Δb	Δh	ΔE	ΔE	ΔH	ΔC	ΔL
Formic	1	7.6	1.90	7.2	1.4	-1.9	-7.2	1.3	2.5	2.6	0.9	-2.4	0.5
	2	4.1	-2.53	3.1	0.9	2.8	2.8	-2.4	2.6	1.8	1.5	1.0	0.3
Citric	1	0.91	0.39	-0.8	0.18	-0.33	-0.82	0.7	0.36	0.33	0.17	-0.27	0.06
	2	3.7	-2.00	2.6	1.7	2.3	2.4	-1.4	2.1	1.6	1.2	0.8	0.7
Oxalic	1	0.96	-0.50	-0.3	-0.76	0.49	-0.33	-0.3	0.53	0.41	0.29	-0.1	-0.28
	2	7.2	-4.18	5.3	2.5	4.6	4.9	-3.4	4.1	3.0	2.4	1.7	1.0
Tarataric	1	2.3	-2.10	0.0	-0.9	2.2	-0.2	-1.3	2.1	1.3	1.2	0.3	-0.3
	2	0.98	-0.42	-0.7	-0.54	0.32	-0.75	-0.4	0.41	0.37	0.20	-0.24	-0.20

According to data from Table 4, acceptable total colour differences (expresed in ΔE units) compared to the standard sample, according to both M&S 83A (tolerance limits are 1.2 - 1.5) and CMS(2:1) standard (tolerance limit is 1.4), are obtained with the additon of citric and oxalic acid, when the dye concentration was 1%. The colour difference obtained with the addition of tartaric acid was only acceptable according to CMC (2:1) standard. When the dye concentration was 2%, acceptable colour difference was obtained only with the addition of tartaric acid, according to both M&S 83A and CMC (2:1) standard. As we can see, no tested acid, used for adjusting the dyebath pH, gave the acceptable colour difference at both dye concentrations, according to both of the standards. Therefore, their usage for this purpose is limited. In the present of formic acid, there was no acceptable colour difference for any of the examined dye concentrations. It can also be seen that at the dye concentration of 1% the hightest colour difference in comparation to the standard, was obtained with the adition of formic acid, and at the dye concentration of 2%, it was obtained with the additon of oxalic acid. Appearently, the colour differences of the polyester knitwear dyed with Disperse Yellow 3, depended on the acid present in the dyebath, as well as of the dye concentration. The total colour differences (ΔE) between the standard sample and the test samples are generaly very small and they are the result of the slight differences in the values of the hue angle from 0° to

360° (ΔH), chroma (ΔC) and lightness (ΔL) of the standard and the test samples [10]. Combination of those coordinates's differences give the total colour diference, ΔE. In the Table 4 we can see how the particular coordinate of the dyed samples is changed when the different acids are added to the dyebath during the dyeing process.The reasons for these diffences are not completely clear, but it is evident that the presence of different acids in the dyebath does have an effect upon them, since all of the other dyeing conditions were the same for all the samples. One possible explenation is that the presence of different anions in each of the baths slightly affected the dye molecule chromophore absorption. It is posible that some slight changes on the fibre surface took place in the presence of different acids: for example, some dye molecules may have migrated out from the interior of the fibres and were then deposited on their surface and grouped in a different way in the presence of different acids [11]; a certain amount of oligomers originating from the polyester fabric may also have deposited on the fibres surface in some way [1], depending on the presence of a particular acid.Small differences in CIELAB coordinates, reflectance curves and degree of whiteness occurred between the undyed polyester knitwear samples treted with the addition of the different acids as well.

Table 5. CIELAB coordinates of the undyed polyester knitwear treated with different acids

Acid	CIELAB coordinates									
	X	Y	Z	a	b	L	C	h	x	y
Acetic	77.22	81.67	82.08	-0.44	4.04	92.43	4.07	96.26	0.3203	0.3389
Formic	71.65	76.44	79.87	-1.74	1.61	90.06	2.37	137.18	0.3143	0.3353
Citric	76.19	81.25	82.90	-1.72	3.11	92.24	3.56	118.96	0.3170	0.3381
Oxalic	75.58	80.28	82.74	-1.09	2.48	91.81	2.71	113.68	0.3168	0.3365
Tartaric	72.82	76.97	75.80	-0.33	5.17	90.31	5.18	93.61	0.3228	0.3412

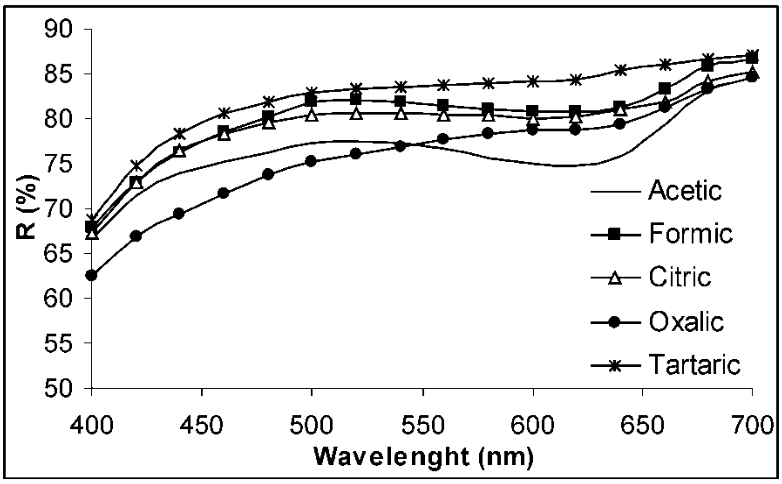

Figure 4. Reflectance curves in the visible spectral range of the undyed polyester knitwear samples treated in the bath with the adition of different acids (R: reflectance,%)

Table 6.The degree of whiteness values according to C.I.E '82 and Berger of the undyed polyesyer knitwear samples treated with different acids

Acid	The degree of whiteness according to C.I.E '82 in % (light souce D65-10')	The degree of whiteness according to Berger in % (liht source C 2)
Acetic	62.87	79.32
Formic	68.71	87.85
Citric	66.68	86.14
Oxalic	68.65	86.81
Tartaric	52.46	68.79

It is evident that they also occurred due to the presence of a different acid in each of the baths. On the basis of their values, it can be concluded that the reasons for their appearance are not exactly the same as for the colour differences between the dyed samples; perharps some small differences on the fibres surface occurred in this case as well: some molecules of dispersing agent could adsorb on the fibres surface in the absence of a dye and aggregate in a different way in the presence of different acids; the aggregation of oligomers originating from the polyester fabric on the fibres surface could take place as well; in any case, the usage of the different acids for adjusting the dyebath pH did have an effect upon the dyed polyester knitwear, but it also affected the undyed, but treated knitwear. However, since those differences are quite small, the effect of different acids used in the dyeing process is not huge neither, and it is related to only small differences in the fibres surface.

4. CONCLUSION

For dyeing polyester knitwear with Disperse Yellow 3 by an exhaustion process, acetic acid can be adequately replaced with citric and oxalic acid for adjusting the dyebath pH value at the dye concentration of 1% under the investigation conditions. For the dye concentration of 2%, acetic acid can be replaced with tartaric acid.

The color differences obtained due to the precence of different acids in the dyebath were small, which means that the influence of the tested acids used for adjusting the optimal dyebath pH value for dyeing polyester knitwear with Disperse Yellow 3 is not significantly different when compared to that of acetic acid. It can be assumed that above acids do not bring about any changes in the mechanism of dye fixing, compared to the one already known in literature on dyeing with the addition of acetic acid.

REFERENCES

[1] Burkinshaw S.M. *Chemical Principles of Sinthetic Fibre Dyeing*, Blackie Academic & Professional: London, 1995, pp 1 - 35

[2] Perepelkin K.E. *Fibre Chem*. 2009, 41, 9–21.

[3] Aspland, J.R. *Textile Dyeing and Coloration*, American Association of Textile Chemists and Colorists: Research Triangle Park, 1997, pp 193 - 211

[4] Ferus-Comelo M., Clark M. and Parker S. *Color. Technol*. 2005, 121, 255–257.

[5] Lee J.J., Han N.K., Lee W.J., Choi J.H. and Kim J.P. *Color. Technol*. 2002, 118, 154–158.

[6] Lee W.J. and Kim J.P, *J.S.D.C.* 1999, 115, 270–273.

[7] Society of Dyers and Colorists (Ed.). *Color Index (3th ed.)*, American Association of Textile Chemists and Colorists: Bradford, 1971, pp 4040.

[8] Mirabella FM.(Ed.). *Modern Techniques in Applied Molecular Spectroscopy*, John Wiley & Sons: New York, 1998, pp 189 - 208

[9] Zarubina N.P., Zavadskaya L.K.and Telegin F.Y. *Fibre Chem*. 2004, 36, 278–282.

[10] Schanda J. (Ed.). *Colorymetry: Understanding the CIE system*, John Wiley & Sons: New York, 2007, pp 1 – 91.

[11] Qian H.F. and Song X.Y. *Color. Technol*. 2009, 125, 141–145.