

The influence of pH adjusted with different acids on the dyeability of polyester fabric

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The influence of using formic, oxalic, citric, tartaric, hydrochloric, nitric, sulphuric and phosphoric acid for dyebath pH adjustment was investigated upon the dyeing of polyester fabric with CI Disperse Yellow 60. The positions of colour in CIELab coordinates of the samples dyed with the addition of tested acids were assessed and compared to those dyed with the addition of acetic acid. It was found that the differences in dyeabilities obtained with the addition of citric, oxalic, hydrochloric, nitric and sulphuric acid are entirely acceptable according to both M&S 83A and CMC (2:1) standards in comparison to the dyeability obtained with the addition of acetic acid.

Keywords: polyester, dyeing, disperse dyes, pH, acids.

INTRODUCTION

Polyester fabrics are the most widely used synthetic fabrics due to their excellent textile properties and high chemical stability. Polyester fabrics hydrophobic nature and their highly compact structure^{1,2} causes their use as disperse dyes, at high temperatures (usually in the range of 115–135°C) and high pressure. Disperse dyes are essentially non-ionic, exhibit poor solubility in water and they can be used in the form of water dispersion^{1,3,4}. The polyester fabric dyeing in water dyebath by the exhaustion process is carried out in a slightly acidic medium. Polyester fibres are resistant to dilute aqueous acids and alkaline solutions and pH value does not provide a crucial impact on the dyeing mechanism; however, many disperse dyes undergo degradation if the pH is uncontrolled during aqueous dyeing. Some disperse dyes have hydrolysable groups in their molecules, such as the ester group which makes them particularly sensitive to hydrolysis, especially in alkaline medium⁵. The hydrolyzed form of dye could be of different shade and in some cases of different affinity for polyester fibres compare to unhydrolyzed 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 4.5–5.5⁶.

According to literature data, acetic acid is generally used for adjusting the dyebath pH value, although a buffer system containing formic acid and ammonium sulfate is used as well^{1,3,7,8}. In this paper, the influence of formic, oxalic, citric, tartaric, hydrochloric, nitric, sulphuric and phosphoric acid for the dyebath pH adjustment was investigated upon the dyeing of polyester (100% PET) knitwear, dyed with the disperse dye CI Disperse Yellow 60 (*p*-Aminobenzoic acid, methyl ester → 3-Methyl-1-phenyl-5-pyrazolone). Differences in colour between the dyed samples with the addition of tested acids and the one dyed with the addition of acetic acid were measured using the technique of reflectometry and expressed in CIELab coordinate values and K/S values. For each of the tested acids, difference in colour acceptability was determined comparing to the colour obtained with the addition of acetic acid, according to M&S 83A and CMC (2:1) standards.

EXPERIMENTAL

Polyester (100% PET) knitwear, produced by Nitex, Nis (Serbia) was used (Table 1). A disperse dye, CI Disperse Yellow 60 (*p*-Aminobenzoic acid, Methyl ester → 3-Methyl-1-phenyl-5-pyrazolone) was purchased from Chemapol (Czech Republic) and used without further purification.

Acetic acid (Sinex Laboratory) was used for pH value adjustment of the standard sample and the following acids were used for the test samples: formic, citric, oxalic, tartaric, hydrochloric, nitric, sulphuric and phosphoric (all of them from Sinex Laboratory).

The polyester knitwear was scoured in a bath containing 1 g/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 traces of the scouring liquor were neutralized upon the addition of 0.1 g/L acetic acid during the last rinsing circle.

Table 1. Characteristics of the undyed polyester knitwear

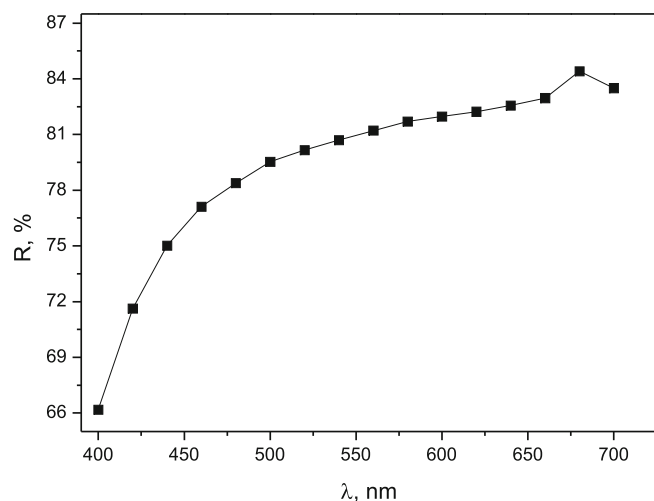
Characteristics	Amount
Horizontal density, [cm ⁻¹]	15
Vertical density, [cm ⁻¹]	18.5
Square meter mass, [gm ⁻²]	130
Shrinking in the process of boiling:	
in length, [%]	1
in width, [%]	1.5

The degree of whiteness of prepared as described polyester knitwear 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. (Fig. 1, Table 2).

Dyeing of polyester knitwear was carried out using an exhaustion process at high temperature and high pressure, with laboratory dyeing machine AHIBA TEXOMAT at a liquor – to – goods ratio of 30:1, in the dyebath containing disperse dye (concentration 2% o.w.f) and 1 g/L Kortamol NNO as a dispersing agent. The pH of the dyebath was adjusted to 4.5 at the beginning of dyeing process recommended by the producers of dyes for polyester knitwear (pH of 4.5 does not cause any degradation neither of fibers nor dyes) using acetic acid for standard sample and formic, citric, oxalic, tartaric, hydrochloric, nitric, sulphuric and phosphoric acid for the

Table 2. CIELab coordinates of the undyed polyester knitwear

Light source	H	C	L	a	b	X	Y	Z
D65-10'	95.99	3.87	92.02	-0.40	3.85	76.36	80.75	81.39
A-10'	79.51	3.87	92.24	0.70	3.80	90.72	81.25	26.89
TL84-10'	90.60	4.24	92.16	-0.04	4.24	84.17	81.05	49.65

**Figure 1.** The reflection curve of the undyed polyester knitwear in the visible spectral range

test samples. Used volumes of acids for pH adjustment are given in Table 3.

The pH was measured using pH meter "RADIOMETER Type PHM 29".

Instead of dyeing profile, we think that the following detailed procedure is better in order to check reproducibility of our results. Dyeing began at the dyebath temperature of 50°C. The dyebath was held at this temperature for 40 min and afterwards, the dyebath temperature was raised to 135°C (it was our choice because the temperature range for dyeing systems under pressure is 125–140°C) and at this temperature dyeing was carried out 60 min. After the dyeing process was finished, the temperature was reduced to 90°C and the knitwear samples were removed and washed out with warm water containing 1 g/L scouring agent (Jugopon 50) at 70°C. Then, the samples were rinsed in warm water, following by washing in cold water until neutral and dried at room temperature. Reduction clearing was performed according to the standard procedure of dyeing

polyesters knitwear with dispersive dyes. This problem was analyzed in details by Baig⁹.

In order to investigate the influence of tested acids on the polyester knitwear, a series of undyed knitwear samples was prepared. The knitwear samples were treated in a 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 bath contained 1 g/L Kortamol NNO (tenside), liquor-to-goods ratio was 30:1, the pH of the bath was 4.5 and it was adjusted the pH using acetic acid for the standard sample and one of the tested acids for the test samples. The treatment of the samples was carried out at 50°C for 40 min, and then at 135°C for 60 min, when the temperature was reduced to 90°C and the knitwear samples were washed with warm water (70°C) containing 1 g/L Jugopon 50, and rinsed in warm, then in cold water and dried at room temperature.

The reflectance spectra of the dyed and undyed, but treated polyester knitwear samples were recorded with an UPDATE COLOR EYE 3000 spectrophotometer (ICS – TEXICON). For the dyed samples, they were expressed in terms of K/S values in the visible spectral range ($\lambda = 400 - 700$ nm). The colour properties of the samples were expressed in terms of CIELab values. The CIELab coordinates were estimated applying the metric programme "Super Match 6 Supplement".

RESULTS AND DISCUSSION

The influence of 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 (with special emphasis on the wavelength of 400–460 nm). The K/S values are correlated with the reflection coefficient R according to the Kubelka – Munk equation^{10, 11}.

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

Table 3. Used volumes of acids for pH adjustment

Number	Sample	Mass of polyester fibre [g]	Mass of the dye [g]	Concentration of acid [mol dm ⁻³]	Volume of acid, V [cm ³]	V _{bath} [cm ³]
1.	C. I. D. YELLOW 60 II (1)	9.0065	0.1801	acetic 8.9 · 10 ⁻⁵	acetic 5.50	270
2.	C. I. D. YELLOW 60 II (2)	9.4529	0.1890	formic 3.8 · 10 ⁻⁵	formic 2.50	284
3.	C. I. D. YELLOW 60 II (3)	9.3023	0.1860	citric 3.3 · 10 ⁻⁵	citric 9.00	279
4.	C. I. D. YELLOW 60 II (4)	9.4603	0.1892	oxalic 3.2 · 10 ⁻⁵	oxalic 5.00	284
5.	C. I. D. YELLOW 60 II (5)	9.0714	0.1814	tartaric 3.3 · 10 ⁻⁵	tartaric 6.50	272
6.	C. I. D. YELLOW 60 II (6)	9.0492	0.1810	hydrochloric 3.2 · 10 ⁻⁵	hydrochloric 3.00	271
7.	C. I. D. YELLOW 60 II (7)	9.7380	0.1948	nitric 3.2 · 10 ⁻⁵	nitric 2.50	292
8.	C. I. D. YELLOW 60 II (8)	9.0275	0.1805	sulphuric 3.2 · 10 ⁻⁵	sulphuric 1.20	271
9.	C. I. D. YELLOW 60 II (9)	9.0458	0.1809	phosphoric 3.2 · 10 ⁻⁵	phosphoric 3.50	271

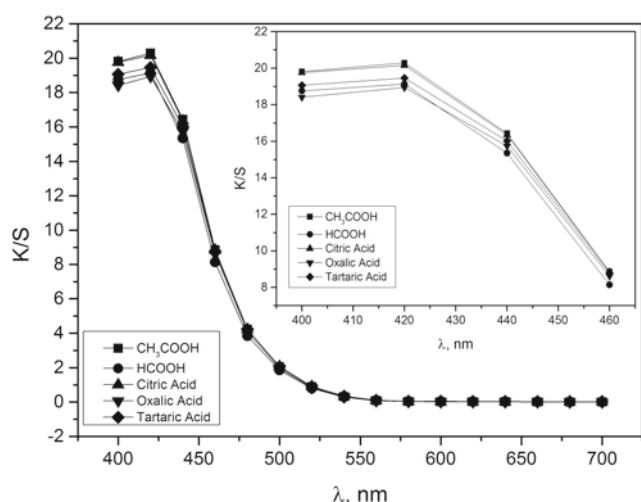


Figure 2. K/S values in the visible spectral range of the polyester knitwear dyed with CI Disperse yellow 60 ($\lambda_{\max} = 420$ nm) and the addition of standard, acetic acid and tested organic acids

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) standard between the test samples and the standard sample are listed in Table 4 (Fig. 2, Fig. 3, Table 4, Table 5).

As can be seen from Table 5, polyester knitwear dyed in CI Disperse Yellow 60 (concentration 2%) with the addition of citric, oxalic, hydrochloric, nitric, sulphuric

and phosphoric acid produces dyeabilities in a daylight (D65) which are completely acceptable according to M&S 83A standard (tolerance limits are 1.2–1.5) and CMC (2:1) standard (tolerance limit is 1.4) compared to the standard sample, dyed with the addition of acetic acid. Dyeability obtained with the addition of formic acid is within the tolerance limit according to M&S 83A standard and acceptable according to CMC (2:1) standard. The lowest colour difference (ΔE units) in comparison to the standard sample was obtained with the addition of phosphoric acid and the highest was achieved with the addition of tartaric acid (they cause changes in structures owing to formation of oligomers-changes of polyesters knitwear as a substrate caused by the different anions of used acids); this colour difference is not acceptable either according to M&S 83A, or CMC (2:1) standard.

The total colour differences between the standard sample and the test samples are very small and they are results of the slight differences in values of hue angle from 0° to 360° (ΔH), chroma (ΔC) and lightness (ΔL) of the standard and test samples.

According to data given in Table 5, the values of ΔH were positive and the values of ΔC were negative for all the test samples comparing to standard sample, while ΔL values were positive for samples dyed with the addition of formic and oxalic acid, and negative for citric, tartaric, hydrochloric, nitric, sulphuric and phosphoric acid compared to the standard sample (according to both CIELab and CMC (2:1) system). This means that for all tested acids, the colour hues of CI Disperse Yellow 60

Table 4. CIELab coordinates of the polyester knitwear dyed with CI Disperse Yellow 60 and some acids

Acid	Light source	CIELab coordinates							
		H	C	L	a	b	X	Y	Z
Acetic	D65-10'	76.62	88.84	77.95	20.56	86.43	58.45	53.13	5.79
	A-10'	75.10	94.21	83.57	24.22	91.04	82.87	63.24	2.31
	TL84-10'	80.09	96.84	81.73	16.67	95.39	69.79	59.80	3.21

Table 5. Colour differences according to CIELab, M&S 83A and CMC (2:1) system between test samples dyed with CI Disperse Yellow 60 and some acids

Acid	Light source	CIELab				M&S 83A			CMC (2:1)			
		ΔE	ΔH	ΔC	ΔL	Δa	Δb	ΔE	ΔE	ΔH	ΔC	ΔL
Acetic (standard)	D65-10'	–	–	–	–	–	–	–	–	–	–	–
	A-10'	–	–	–	–	–	–	–	–	–	–	–
	TL84-10'	–	–	–	–	–	–	–	–	–	–	–
Formic	D65-10'	2.0	1.47	-1.3	0.4	-1.7	-1.0	1.3	0.95	0.84	-0.42	0.16
	A-10'	2.0	1.01	-1.7	0.3	-1.3	-1.5	0.9	0.76	0.54	-0.53	0.09
	TL84-10'	1.9	1.42	-1.2	0.4	-1.6	-1.0	1.2	0.87	0.77	-0.37	0.15
Citric	D65-10'	1.7	0.66	-1.4	-0.7	-0.9	-1.3	0.66	0.64	0.37	-0.45	-0.27
	A-10'	1.9	0.49	-1.6	-0.9	-0.8	-1.5	0.51	0.64	0.26	-0.49	-0.31
	TL84-10'	1.8	0.59	-1.5	-0.8	-0.9	-1.4	0.67	0.66	0.39	-0.45	-0.29
Oxalic	D65-10'	1.3	1.11	-0.6	0.3	-1.2	-0.3	0.95	0.68	0.65	-0.19	0.10
	A-10'	1.2	0.89	-0.8	0.1	-1.0	-0.6	0.71	0.55	0.49	-0.25	0.05
	TL84-10'	1.4	1.27	-0.5	0.3	-1.3	-0.3	1.01	0.70	0.68	-0.16	0.10
Tartaric	D65-10'	5.3	2.37	-4.3	-2.0	-3.3	-3.6	2.5	2.1	1.5	-1.3	-0.7
	A-10'	5.8	1.97	-4.9	-2.4	-2.9	-4.4	1.9	2.0	1.1	-1.5	-0.9
	TL84-10'	5.8	2.92	-4.5	-2.2	-3.6	-4.0	2.7	2.3	1.7	-1.3	-0.8
Hydrochloric	D65-10'	2.2	1.11	-1.8	-0.6	-1.5	-1.6	1.1	0.90	0.65	-0.57	-0.24
	A-10'	2.4	0.53	-2.2	-0.8	-1.2	-1.9	0.7	0.84	0.42	-0.66	-0.30
	TL84-10'	2.3	1.25	-1.8	-0.7	-1.4	-1.6	1.0	0.88	0.64	-0.55	-0.25
Nitric	D65-10'	0.75	0.26	-0.6	-0.37	-0.13	-0.64	0.17	0.24	0.01	-0.20	-0.14
	A-10'	0.76	0.28	-0.6	-0.40	-0.15	-0.62	0.15	0.24	0.01	-0.19	-0.15
	TL84-10'	0.82	0.17	-0.7	-0.39	-0.13	-0.71	0.17	0.26	–	-0.21	-0.14
Sulphuric	D65-10'	0.95	0.42	-0.8	-0.30	-0.55	-0.71	0.38	0.36	0.23	-0.25	-0.11
	A-10'	1.04	0.36	-0.9	-0.38	-0.50	-0.82	0.30	0.35	0.16	-0.28	-0.14
	TL84-10'	0.99	0.48	-0.8	-0.33	-0.53	-0.77	0.37	0.36	0.22	-0.25	-0.12
Phosphoric	D65-10'	0.63	0.17	-0.6	-0.09	-0.15	-0.61	0.14	0.19	–	-0.19	-0.03
	A-10'	0.60	–	-0.6	-0.12	-0.15	-0.57	0.12	0.18	–	-0.18	-0.04
	TL84-10'	0.70	–	-0.7	-0.10	-0.14	-0.67	0.14	0.21	0.01	-0.20	-0.04

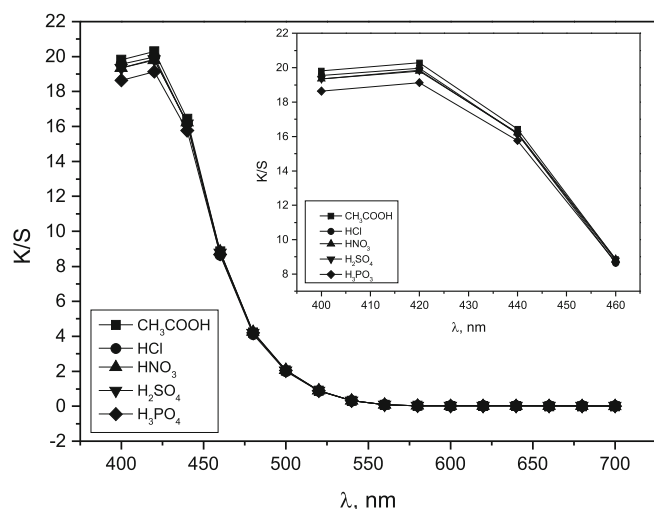


Figure 3. K/S values in the visible spectral range of the polyester knitwear dyed with CI Disperse yellow 60 ($\lambda_{\max} = 420$ nm) and the addition of standard, acetic acid and the tested inorganic acids

on polyester knitwear were slightly shifted to greenish according to both CIELab and CMC (2:1) system (the sample dyed with the addition of phosphoric acid showed no difference in the hue value compared to standard in CMC (2:1) system, but gave the positive ΔH value in CIELab system). The shades of all the test samples were darker than the standard sample. The samples dyed with the addition of formic and oxalic acid were lighter, whereas those dyed with the addition of citric, tartaric, hydrochloric, nitric, sulphuric and phosphoric acid were darker than the standard sample, dyed with the addition of acetic acid. The dyeability obtained with the addition of tartaric acid was the greenest, the darkest than samples obtained with the addition of acetic acid which was the yellowest and the brightest; those obtained with the addition of formic acid was the lightest. For all three coordinates (ΔH , ΔC and ΔL), the highest difference related to the standard sample was obtained with the addition of tartaric acid, and the lowest with the addition of phosphoric acid.

The reasons for these differences are not clear, but it is evident that the presence of different acids in dyebaths influences the dyeing process, since remaining dyeing conditions were the same for all samples. One possible explanation is that the presence of different anions in baths slightly affect the adsorption of dyechromophore of dye molecule (changes of polyester knitwear caused by the different anions of used acids). It is possible that some slight changes on fibre surface (SEM evidences are available, but in this paper we did not provide them (they can be gotten upon the request) took place in the presence of different acids: for example, some of dye molecules may have migrated out from the interior of the fibres and were then deposited on their surface and grouped in a different manner in the presence of different acids¹² a certain amount of oligomers originating from the polyester fabric may also have deposited on the fibres surface¹, depending on the presence of acid. RC washing would be helpful in eliminating these oligomers⁹.

Small differences in CIELab coordinates, reflectance curves and degree of whiteness occurred between the undyed samples treated with the addition of the different acids as well (Table 6, Table 7, Fig. 4). It is

evident that they also occurred due to the presence of different acid in baths. On the basis of their values, it can be concluded that the reasons for appearance of small differences are not exactly the same as for the colour differences between the dyed samples; perhaps some small differences on fibres surface occurred as well: some molecules of dispersing agent could adsorb on fibres surface in the absence of dye and aggregate in a different way in the presence of different acids; the aggregation of oligomers originating from the polyester fabric on fibres surface could take place in this case. The degree of whiteness of the undyed fibres is generally lower with the addition of stronger acids.

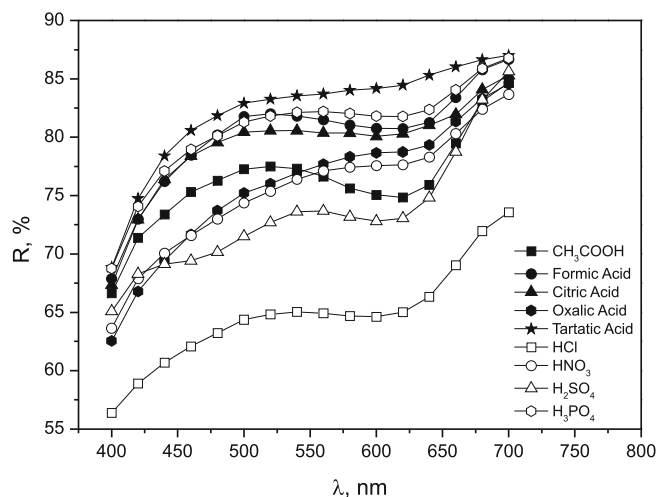


Figure 4. The reflectance curves in the visible spectral range of the undyed polyester knitwear samples treated in the bath with acids

CONCLUSION

Formic, citric, oxalic, tartaric, hydrochloric, nitric, sulphuric and phosphoric acid were used for the dyebath pH adjustment in the process of dyeing the polyester knitwear with disperse dye CI Disperse Yellow 60. Colour differences were investigated between the polyester knitwear samples dyed with the addition of acids and the standard sample, dyed with the addition of acetic acid. Small differences in colour appeared between dyed samples with the addition of tested acids and the sample dyed with the addition of acetic acid.

For dyeing polyester knitwear with CI Disperse Yellow 60 by an exhaustion process, acetic acid can be adequately replaced with citric, oxalic, hydrochloric, nitric, sulphuric and phosphoric acid for the dyebath pH value adjustment under the conditions of the investigation

The influence of the tested acids used for the optimal dyebath pH value adjustment in dyeing polyester knitwear with CI Disperse Yellow 60 is not significantly different compared to acetic acid. The above acids do not cause any changes in the mechanism of dye fixing, compared to already known in literature. We did not control the pH change at the end of dyeing.

ACKNOWLEDGMENT

This work was supported by the Project No. 34020 of Ministry of Education and Science, Republic of Serbia.

Table 6. CIELab coordinates of the undyed polyester knitwear (treated with acids)

Acid	Light source	CIELab coordinates							
		H	C	L	a	b	X	Y	Z
–	D65-10'	96.26	4.07	92.43	-0.44	4.04	77.22	81.67	82.08
	A-10'	79.50	4.06	92.67	0.74	3.99	91.80	82.21	27.13
	TL84-10'	90.90	4.46	92.57	-0.07	4.46	85.13	82.00	50.06
Acetic	D65-10'	137.18	2.37	90.06	-1.74	1.61	71.65	76.44	79.87
	A-10'	128.45	1.50	89.99	-0.93	1.17	84.27	76.29	26.34
	TL84-10'	136.48	2.31	90.00	-1.68	1.59	78.38	76.30	48.77
Formic	D65-10'	118.96	3.56	92.24	-1.72	3.11	76.19	81.25	82.90
	A-10'	101.54	2.75	92.27	-0.55	2.69	90.06	81.32	27.40
	TL84-10'	111.41	3.55	92.24	-1.30	3.30	83.69	81.24	50.54
Citric	D65-10'	113.68	2.71	91.81	-1.09	2.48	75.58	80.28	82.74
	A-10'	93.53	2.18	91.87	-0.13	2.18	89.29	80.41	27.32
	TL84-10'	106.20	2.74	91.82	-0.77	2.63	83.00	80.30	50.49
Oxalic	D65-10'	93.61	5.18	90.31	-0.33	5.17	72.82	76.97	75.80
	A-10'	78.08	5.35	90.64	1.11	5.24	86.97	77.69	25.08
	TL84-10'	90.58	5.75	90.50	-0.06	5.75	80.36	77.39	46.17
Tartaric	D65-10'	101.09	3.33	93.23	-0.64	3.27	78.84	83.49	85.01
	A-10'	81.78	3.15	93.39	0.45	3.12	93.48	83.86	28.08
	TL84-10'	94.66	3.57	93.31	-0.29	3.56	86.75	83.67	51.86
Hydrochloric	D65-10'	104.43	3.08	84.32	-0.77	2.98	61.00	64.68	65.88
	A-10'	83.23	2.89	84.45	0.34	2.87	72.35	64.94	21.74
	TL84-10'	101.94	3.30	84.37	-0.68	3.23	66.96	64.78	40.18
Nitric	D65-10'	92.66	4.44	89.97	-0.21	4.43	72.19	76.24	76.00
	A-10'	78.20	4.65	90.26	0.95	4.55	85.98	76.88	25.09
	TL84-10'	91.58	4.90	90.16	-0.14	4.90	79.55	76.66	46.37
Sulphuric	D65-10'	96.90	2.99	88.40	-0.36	2.97	68.94	72.89	74.40
	A-10'	80.55	3.14	88.58	0.52	3.10	81.72	73.28	24.49
	TL84-10'	102.99	3.27	88.51	-0.73	3.18	75.60	73.14	45.50
Phosphoric	D65-10'	108.92	3.19	92.46	-1.03	3.01	76.98	81.73	83.53
	A-10'	91.16	2.80	92.56	-0.06	2.80	91.06	81.96	27.57
	TL84-10'	104.10	3.35	92.51	-0.82	3.25	84.58	81.86	50.97

Table 7. Values of the degree of whiteness according to C.I.E '82 and Berger of the undyed polyester knitwear samples

Acid	The degree of whiteness according to C.I.E '82 (light source D65-10')	The degree of whiteness according to Berger (light source C 2)
–	62.87	79.32
Acetic	68.71	87.85
Formic	66.68	86.14
Citric	68.65	86.81
Oxalic	52.46	68.79
Tartaric	68.40	85.48
Hydrochloric	49.69	66.41
Nitric	55.17	71.17
Sulphuric	58.54	74.41
Phosphoric	67.68	85.63

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