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Fluorescent Dyes Destined for Dyeing High-Visibility Polyester Textile Products

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Abstract
We present an investigation and analysis results of fluorescent disperse dyes used for dying polyester fibres and fabrics to high-visibility colours. The paper describes the results of dye identification tests, the chemical structure of selected dyes, and the results of spectrophotometric colour measurements for yellow fluorescent dyes and their matching with the requirements of the EN 471:1994 standard. The results of the colour tests are presented in the colorimetric system of the three-chromaticity x, y, and Y coordinates, and in the CIE Lab colour differentiation system.

Key words: *fluorescent dyes, disperse dyes, colorimetric measurements, high-visibility textile products.*

Introduction

Many organic substances are characterised by fluorescent ability. In accordance with a generally accepted definition, fluorescent dyes are considered to be those dyes which absorb and emit radiation in the visible spectral range. This limitation, only to the visible spectral range, allows us to eliminate the optical brighteners and dyes characterised by fluorescence in the near-infrared range from the group of substances under consideration. Chemical compounds described as fluorescent substances have been known for a long time. In 1971, Bayer designated the fluorescent dye he discovered as 'fluorescine'. Since this time, several dozen dyeing substances showing fluorescent ability have been recognised. For example, the acridine and xanthene dyes, and the rhodamine and phthalein derivatives can be included into this group. Many chemical structures which are characterised by the phenomenon of fluorescence can be mentioned [1].

The application of fluorescent dyes involves various differentiated fields. Recently, the application of fluorescent dyes in medical diagnostic and biochemical investigations has developed extremely dynamically [2]. Fluorescent dyes have also been used for the construction of lasers, photoelectric cells, and solar batteries.

The application of fluorescent dyes in medium flow control, as well as for testing material defects and equipment damage, has been well known for many years. But one of the major application fields where fluorescent dyes have long been used is for the dyeing of flat materials, especially textiles. The use of fluorescent dyes causes a significant increase in colour brightness, which makes the dyed materials more easily perceptible, or more accurately, have increased visibility. This intensified perceptibility of materials dyed with fluorescent dyes is an advantage in preparing coloured advertisements, information descriptions, road and traffic signs, and to a great extent for manufacturing sports clothing and clothing for special services, such as fire brigades and the police. As the use of every fluorescent dye seemingly causes a desired increase in the depth of colour of the dyed textiles, an independent test was accepted for materials with high visibility to avoid a subjective estimation of the fluorescent dyes' usability. The test is performed in accordance with the EN 471:1994 standard. This standard describes the requirements for textiles dyed with fluorescent dyes which are destined for manufacturing high-visibility 'warning' clothing. Polish Standard PN-EN 471:1997 is identical to the above-mentioned European standard [3].

The problem of fluorescent dyes used in dyeing textile materials has often been described. Source data related to questions connected with this problem can be found in reviews concerned with dyes [1,4,5], as well as in monographs devoted only to fluorescent dyes [6,7]. In the Polish scientific and technical literature concerned with dyeing problems, some papers have also appeared in which the application and estimation of fluorescent dyes have been discussed [8-11]. However, upon analysing the published literature, no information devoted to research into comparison of fluorescent dyes in accordance to the EN 471:1994 standard requirements has been found. This problem is interesting first of all because the above-mentioned standard is connected with the 89/686 EEC European Instructions, which are concerned with individual protective clothing and other protective equipment. In turn, for these object the estimation of the conformity with the requirements is demanded, before the CE-mark, which guarantees the quality and the security of the products, can be granted. It is well known from practical experience that the fulfilment of the requirements of standards at industrial dyeing conditions may cause many problems. Therefore an analysis of the fluorescent dyes offered on the Polish market and the textiles dyed with them seems to be potentially useful, and are the subject of the current paper.

Theoretically, one may assume that in the period of a global market the dyes offered on the Polish market are the same as those on the world market. However, in practice some dye brands exists which, for example, are popular, on the Far East market but are not offered on the European, and by extension the Polish market.

The Structure of Disperse Dyes Used for Dyeing High-Visibility Textile Products

The Institute of Dyes and Organic Products in Zgierz, Poland, which includes in its structure an accredited laboratory for research into materials of high visibility, carried out tests of disperse dyes offered on the Polish market and used for dyeing polyester fibres and textile fabrics which should fulfil the requirements of the EN 471:1994 standard. It should be emphasised here that 'warning' clothing of high visibility is manufactured in only three colours, yellow, orange, and red.

From among many disperse dyes described in the Colour Index, as well as in manufacturers' information, only the dyes listed in Table 1 have found practical application for dyeing fabrics intended to meet the demands of the EN 471:1994 standard.

The data presented in Table 1 shows a situation which is quite typical of the

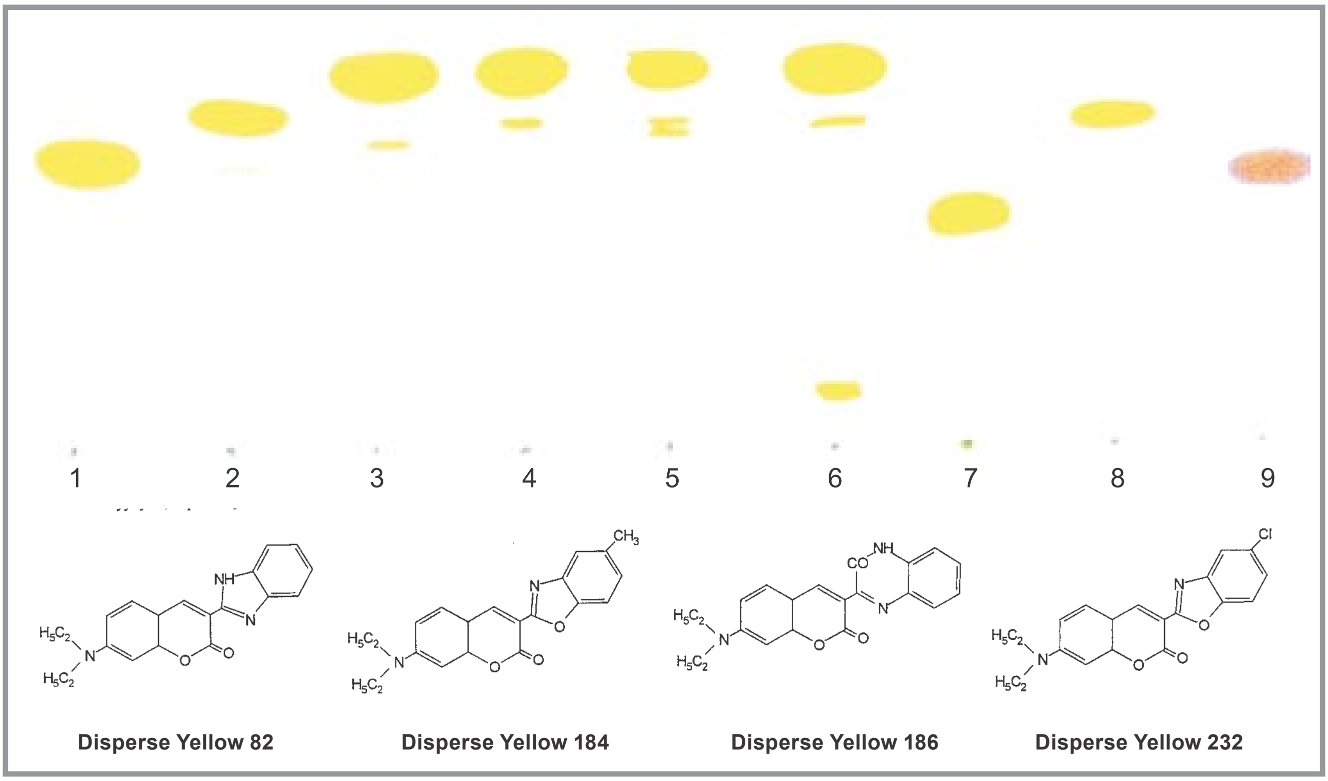

***Figure 1.** Chromatogram of the nine fluorescent dyes listed in Table 1, and the identified chemical structure formulas of four of these dyes. 1 - C.I. Disperse Yellow 82, 2 - C.I. Disperse Yellow 186, 3 - C.I. Disperse Yellow 184:1/A, 4 - C.I. Disperse Yellow 184:1/B, 5 - C.I. Disperse Yellow 184:1/C, 6 - C.I. Disperse Yellow 232, 7 - C.I. Disperse Yellow 199, 8 - C.I. Disperse Yellow 184, 9 - C.I. Disperse Red 277.*

dyes market, where a lack of data allowing the structure of dyes to be identified can often be observed. Considering the strong competitiveness between the individual manufacturers and sellers of dyes, especially from the Far East, the leading dye-manufacturers omitted to publish even the numbers of the C.I Generic Names of dyes, not to mention the numbers of structures (the C.I. Numbers), in its advertising leaflets.

As an example, the Disperse Yellow 184:1 dye, the dye of this group which is most easy available and popular on the market, has a chemical structure which has not been described until now, in contrary to the Disperse Yellow 184 dye, which although its structure is known, has been eliminated from the market.

Presenting in this paper the investigations we carried out, we accepted the use of deciphered C.I. Generic Name Numbers for dye description, in accordance with the customs honoured by the majority of manufacturers, and not the dye trade marks.

As mentioned earlier, the EN 471:1994 standard takes into account only three colours of high visibility: yellow, orange, and red. Table 1 shows that from the dyer's standpoint, the choice is limited only to yellow dyes. As can be seen, the Disperse Red 277 dye is in practice the only fluorescent red dye. However, as recently as ten years ago, in 1993, Christie [7], while discussing fluorescent red dyes, claimed that the only red fluorescent dye of industrial importance and with a structure described in literature is the Disperse Red 303. Because in dyeing practice the colour orange is obtained by mixing yellow and red dyes, and not by using an individual orange dye, the analysis of the market of fluorescent dyes used for dyeing high-visibility clothing is limited to the analysis of yellow dyes.

***Table 1.** Disperse dyes used for dyeing fabrics which should meet the requirements of the EN-471:1994 standard (*the dye is a blend of two isomers).*

C.I. Generic Name	C.I. Number	CAS	EINECS	Offered on the Polish market
Disperse Yellow 82	55120	27425-55-4	248-451-4	+
Disperse Yellow 184	55164	34564-13-1	252-093-4	-
Disperse Yellow 184:1	-	-	-	+
Disperse Yellow 186	55121	28754-28-1	249-200-1	+
Disperse Yellow 199*	-	35254-10-5 78108-20-0	252-466-1 278-838-3	-
Disperse Yellow 232	55165	35773-43-4	252-722-2	-
Disperse Red 277	-	-	-	+

In Figure 1 a chromatogram of fluorescent dyes approachable on the market is presented, together with the structural formulas only of these dyes for which the formulas have been already officially published. The chromatogram was carried out at the Institute of Dyes and Organic Products.

As can be seen from the structural formulas presented, the yellow dyes of the C.I. numbers 82, 184, 186, and 232 belong to the coumarin group of dyes. Based on the comparison of mass spectra carried out in the Institute of Dyes and Organic Products, and on chromatographic investigation, it can be stated that the Disperse Yellow 184:1 dye is a dye identical with Disperse Yellow 232, and is a coumarine derivative. Also the Disperse Red 277 dye is a coumarine derivative, as has been established by our research [12]. The only dye listed in Table 1 which belongs to another chemical group, is the Disperse Yellow 199 dye. Although its C.I. number is not known, the knowledge of its CAS number allows us to state that it belongs to the group of benzoxanthone derivatives. This dye was present on the Polish market in the past, and although nowadays the European firms do not offer it, it can be found among the offers of Far Eastern producers.

It should be emphasised that the fluorescent dyes we investigated were commercial products, and not raw materials or laboratory preparations. There is a significant differentiation in the case of fluorescent dyes, as the content of the colouring matter in a commercial product, known as the 'typical dye', in general falls within the range of 12-16%; dispersing agents form the rest. This is why it is very troublesome to compare dyeings performed by a non-standardised synthetic preparation with those realised on the basis of a commercial product. For example, a 1% dyeing carried out by means of a non-standardised preparation can be related to a 10% dyeing with the use of a typical commercial product. Considering additionally the effects of the phenomenon of fluorescence quenching with the increase in concentration of the fluorescent substance and the influence of various dispersing agents, it may appear that the estimation of raw dyes do not match those of commercial products.

Research Methodology and Materials Used

The estimation of the fluorescent dyes offered on the Polish market which we carried out was based on the colour analysis of dyeings carried out with the use of the individual dyes and the relation of the results obtained to the requirements of the EN 471:1994 standard for the products destined for high-visibility 'warning' clothing.

The basic parameter which determines the products' usability, in accordance with the standard mentioned above, is the colour characterised by the chromaticity co-ordinates x and y, which determine the chroma saturation and hue, and the luminance factor (determining luminosity) $\beta = Y/Y_0$, where $Y_0 = 100$ is the three-chromatic co-ordinate for a perfect reflecting diffuser, and Y the co-ordinate for the colour tested.

,The luminance factor for fluorescent materials is the sum of two components: the reflective luminance factor and the fluorescent luminance factor.

For dyeing, which fulfils the requirements of the standard mentioned above, the chromaticity co-ordinates x and y should be included within the boundaries of the area determined by this standard, and the luminance factor β should be equal to or greater than the value of 0.76. These parameters are determined on the sample before and after illumination.

The illuminations of the dyeings were carried out with the use of a Xenotest apparatus in accordance with the ISO

Table 2. *The values of colour parameters: the chromaticity co-ordinates x and y, and the luminance factor β, together with the colour co-ordinates L, a, and b of the dyeings before illumination.*

Dye designation according to Colour Index	Producer	Dyeing strength, %	x	y	β	a	b	L
Disperse Yellow 82	-	0.6	0.3581	0.5028	0.81	-42.72	68.36	92.13
		0.8	0.3615	0.5133	0.83	-44.76	73.89	93.14
		1.0	0.3653	0.5237	0.84	-46.21	79.29	93.4
Disperse Yellow 184:1	A	0.6	0.3618	0.5432	0.88	-53.65	87.6	95.31
		0.8	0.3662	0.544	0.89	-52.01	89.42	95.01
		1.0	0.3689	0.5475	0.88	-51.97	92.17	95.29
Disperse Yellow 184:1	B	0.6	0.3637	0.5403	0.89	-52.24	87.24	95.55
		0.8	0.3719	0.5431	0.89	-49.43	90.94	94.74
		1.0	0.3728	0.5473	0.88	-50.37	93.62	95.2
Disperse Yellow 184:1	C	0.6	0.3647	0.545	0.86	-52.4	88.48	94.12
		0.8	0.3687	0.5521	0.89	-53.3	94.33	95.37
		1.0	0.3704	0.5534	0.88	-52.95	95.59	95.34
Disperse Yellow 186	-	0.6	0.3632	0.5391	0.77	-49.63	82.4	90.25
		0.8	0.3695	0.5388	0.79	-47.66	85.29	91.24
		1.0	0.3715	0.5479	0.80	-49.43	90.4	91.68
Disperse Yellow 199	-	0.6	0.3514	0.5014	0.83	-43.25	65.95	93.02
		0.8	0.3611	0.5165	0.84	-43.55	69.44	92.72
		1.0	0.3623	0.5212	0.85	-46.39	77.53	93.74

Table 3. *The values of colour parameters: the chromaticity co-ordinates x and y, and the luminance factor β, together with the colour co-ordinates L, a, and b of the dyeings after illumination in accordance with the EN 471:1994 standard.*

Dye designation according to Colour Index	Producer	Dyeing strength, %	x	y	β	a	b	L
Disperse Yellow 82	-	0.6	0.3579	0.4876	0.78	-37.97	62.05	90.69
		0.8	0.3628	0.5026	0.82	-40.94	69.77	92.36
		1.0	0.3663	0.5093	0.81	-41.32	73.08	92.00
Disperse Yellow 184:1	A	0.6	0.3605	0.5211	0.83	-52.23	85.67	94.45
		0.8	0.3636	0.5353	0.88	-52.32	85.54	94.67
		1.0	0.3635	0.5341	0.89	-52.44	84.67	95.23
Disperse Yellow 184:1	B	0.6	0.3632	0.5364	0.89	-51.47	85.51	95.70
		0.8	0.3689	0.5394	0.83	-48.85	86.75	93.03
		1.0	0.3704	0.5464	0.86	-50.68	91.55	94.39
Disperse Yellow 184:1	C	0.6	0.3642	0.5421	0.86	-52.04	87.11	94.35
		0.8	0.3693	0.5483	0.87	-52.05	92.01	94.69
		1.0	0.3714	0.5513	0.91	-52.61	95.28	96.27
Disperse Yellow 186	-	0.6	0.3725	0.5423	0.84	-48.23	81.56	91.12
		0.8	0.3729	0.5439	0.85	-47.11	82.56	91.10
		1.0	0.3732	0.5486	0.85	-46.98	83.34	91.03
Disperse Yellow 199	-	0.6	0.3532	0.4967	0.83	-43.29	65.29	92.88
		0.8	0.3568	0.5057	0.82	-44.16	69.20	92.37
		1.0	0.3615	0.5206	0.85	-47.09	77.27	93.95

105-B02 standard. The samples were illuminated according to the requirements of the EN 471:1994 standard.

The measurement of the parameters of fluorescent colours demands measurement conditions which represents the actual conditions, as the observed chroma and hue of the sample is the result of mutual influence of the radiation reflected and the fluorescence radiation. These conditions are described in the CIE (Commission Internationale de l'Éclairage) standardisation documents. In accordance with the CIE recommendations, the tests of colour of the sample dyeing, dyed with the use of fluorescent dyes, should be carried out with the use of a spectrophotometer or a colorimeter of a 45/0 measuring geometry. This means that the sample illumination should be performed at an angle of 45° to the normal, whereas the observation at the angle of 0° is perpendicular to the sample. The sample illumination should be realised by polychromatic light reproducing the relative power distribution of the D_{65} illuminant radiation, and with the use of the CIE 1931 normal colorimetric system (standard colorimetric observer 2°). The samples are placed in one layer on a black base with a reflection factor smaller than 0.04.

Investigations of the colour parameters of the dyed samples were carried out with the use of a 508c MINOLTA spectrophotometer with the recommended measuring geometry. The investigation results are presented in the x, y, and Y colorimetric system, and in the CIE Lab system which is most often used by colourists. Both of these systems enable the presentation of colour differences of the tested dyeings.

Materials

Dyeings of colour strength of 0.6%, 0.8%, and 1.0% were carried out using a polyester knitted fabric of 140 g/m^2 mass per unit area, with the use of the six following yellow fluorescent dyes:

- C.I. Disperse Yellow 82,
- C.I. Disperse Yellow 184:1A,
- C.I. Disperse Yellow 184:1B,
- C.I. Disperse Yellow 184:1C,
- C.I. Disperse Yellow 186, and
- C.I. Disperse Yellow 199.

(The dyes marked A, B, and C are manufactured by various producers).

The accepted methodology procedure allowed us to analyse the changes of colour parameters of the samples as a function of the dyeing strength, before and after illumination.

Discussion of the Results Obtained

The results of the colour analyses of the dyeings obtained with use of the fluorescent dyes tested are presented in Table 2 for samples before illumination, and in Table 3 for samples after illumination.

Analysing the investigation results obtained and presented in Table 1, an optimum dyeing strength can be observed at which the luminance factor achieves its maximum value.

A further increase in dyeing strength causes a slow quench of the fluorescence. This phenomenon is only visible for the concentrations selected in our investigation for the C.I. Disperse Yellow 184:1 dye; for the rest of the dyes tested, the maximum values of the luminance factor β are achieved at higher concentrations (for example, for the Disperse Yellow 186 dye at a concentration of 1.2%). We can also observe that the colour of all dyeings tested is characterised by a high luminance factor, which is greater than 0.76.

The values of the colour parameters of the dyeings of 1.0% colour strength are presented in Figure 2 in the a-b co-ordinate system (a/-a is the red/green co-ordinate, and +b/-b the yellow/blue co-ordinate).

As can be seen, the colour differences between dyeings carried out with use of the C.I. Disperse Yellow 184:1 dyes from three various producers (the measurement results of samples A, B and C) are very small. Also, the colours of the dyeings carried out by the C.I. Disperse Yellow 82 and the C.I. Disperse Yellow 199 are similar but are characterised by a less green-yellow hue in relation to the rest of the samples tested. In contrast, the colour of the dyeing obtained with the use of the C.I. Disperse Yellow 186 dye has a hue more similar to the dyeings obtained by the C.I. Disperse Yellow 184:1 group of dyes.

So, it can be stated that the dyeings obtained with the use of all tested dyes are characterised by an intensive green-yellow hue which testifies to the high brightness of colour.

In the end, the most essential matter is the question: 'do the dyeings carried out with the use of the dyes tested fulfil the rigorous requirements of the standard?' To obtain an answer to this question, the diagram presented in Figure 3 was designed. The chromaticity positions of the obtained dyeings in the x-y co-ordinates are presented, together with the boundaries of the area (drawn by continues lines) determined by the discussed standard for the yellow colour required. For all dyes tested, the increase in colour strength of the dyeings within the range from 0.6% to 1.0%, causes a drift of the chromaticity position of the individual colours of the dyeings along the x-co-ordinate from left to right, which means in the direction of more saturated colours. All dyeings for which the chromaticity position in the x-y co-ordinates is placed inside the area enclosed by the two continuous lines (within the area of the acute angle) fulfil the requirements of the standard if their luminance factor is no smaller than 0.76. Fabrics with dyeings characterised by colour co-ordinates positioned outside the area marked cannot be applied for the manufacture of high-visibility products.

However, the dyeings obtained with the use of the C.I. Disperse Yellow 184:1 fluorescence dyes are characterised by small colour differences (as mentioned before), but nevertheless the chromaticity positions are critical, considering the requirements of the standard. The products of producers A and C give dyeings of chromaticity co-ordinates positioned outside the required area; nevertheless the increase in dyeing colour strength from 0.6% to 1.0% (in comparison to the fluorescent dye from producer B, at concentrations about 0.8% and higher) allow us to obtain a dyeing within the colour parameters required. A similar colour change, with an increase in concentration and a colouristic effect of the same kind, can be achieved using the C.I. Disperse Yellow 186 dye.

The chromaticity co-ordinates of dyeings obtained with the use of the C.I. Disperse Yellow 82 dye with a strength higher than 0.8% also fall within the required area. The colour parameters of dyeings carried out with the use of the C.I. Disperse Yellow 199 dye are positioned outside the area required by the standard for the whole range of colour strengths tested. In general, we can state that the x-y co-ordinates of dyeings obtained with the use of all the fluorescent dyes tested are practically at the boundary of the colours required by the standard. This means that a minimal change in concentration, the quality of the dye or even a change of the raw material of the processed textile fabric may cause undesirable changes in dyeing and a failure to meet the standard requirements for colour. In industrial conditions, the result will be a disqualification of the dyed fabric. This is the reason why tinting the discussed fluorescent dyes by the use of non-fluorescent disperse yellow dyes is applied in the majority of cases [9-11]. The effect of such a procedure is colour shifting within the required area of chromaticity of the dyeing obtained. However, such tinting can simultaneously cause a decrease in luminance which is often below the required value.

The effect of light has a significant influence on the colour parameters. The requirements of the standard for the yellow fluorescent colour comprise only a short illumination described in the standard, and a repeated test of the colour parameters. Great changes in the x-y chromaticity co-ordinates after illumination testify to a low fastness to light. The colour changes we observed for dyeings obtained with the use of the individual fluorescent dyes are listed in Table 3 and presented in Figure 4.

Significant colour changes after illumination are characteristic of dyeings carried out with the use of the C.I. Disperse Yellow 82 and C.I. Disperse Yellow 186 dyes. The colour of these dyeings is shifted in the direction of the red co-ordinate (the value of the chromaticity a-co-ordinate rises). A higher light fastness is displayed by the dyeing obtained by the C.I. Disperse Yellow 184:1A dye. Its colour changes in the direction of the blue co-ordinate (the value of the chromaticity b-co-ordinate decreases). The change in colour of the rest of the dyeings obtained with the use of the C.I. Disperse Yellow 184:1 (B and C) dyes is insignificant, which proves their very high light fastness.

An analysis of the chromaticity x-y co-ordinates of the dyeings obtained in relation to the standard requirements is also of interest; this is presented in Figure 5 for dyeings of 1.0% colour strength. As can be seen in Figure 5, the colour co-ordinates of dyeings obtained with the use of the C.I. Disperse Yellow 82 dye after illumination in the Xenotest apparatus are shifted within the chromaticity area required by the standard for the high-visibility yellow colour. Despite the significant change in the hue caused by the low light fastness, the dyeing still meets the requirements of the standard. The colour of the dyeing obtained by the C.I. Disperse Yellow 186 dye, which is characterised by significantly higher light fastness than the dyeing mentioned earlier, is also shifted within the required area after illumination of the sample. But the direction of the changes indicates that the colour does not lose its saturation, in contrary to the dyeing obtained with the use of the C.I. Disperse Yellow 82 dye.

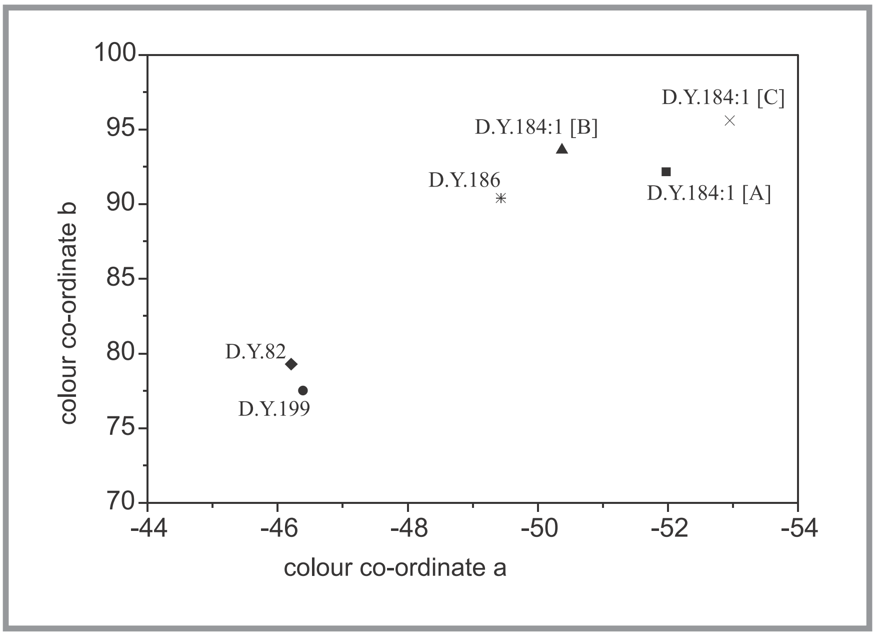

Figure 2. The colours of dyeings carried out with the use of fluorescent disperse dyes presented in the CIE chromaticity diagram in the a-b rectangular co-ordinates.

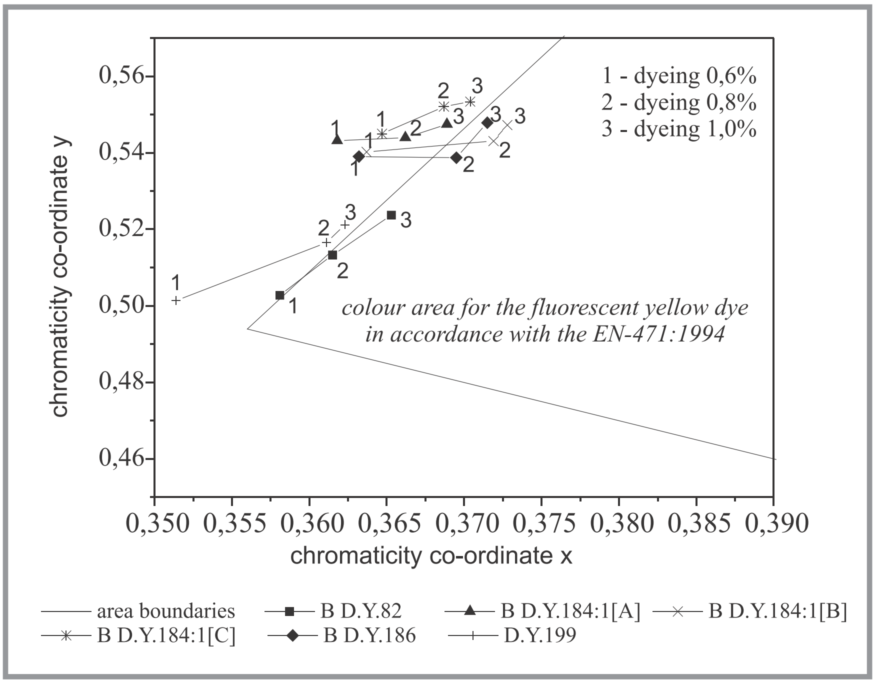

Figure 3. The chromaticity parameters of the dyeings of 0.6%, 0.8%, and 1.0% colour strength presented in the x-y chromaticity co-ordinates, in relation to the area boundaries for the fluorescent yellow dye in accordance with the EN-471:1994 standard.

Conclusions

- The fluorescent yellow dyes tested belong to the coumarin group of dyes, with the exception of the C.I. Disperse Yellow 199 dye, which belongs to the benzoxanthone derivatives group.
- All the dyes tested allow dyeings of a very bright colour of yellow-green hue and high luminance factor within the range of β from 0.77 to 0.89 to be

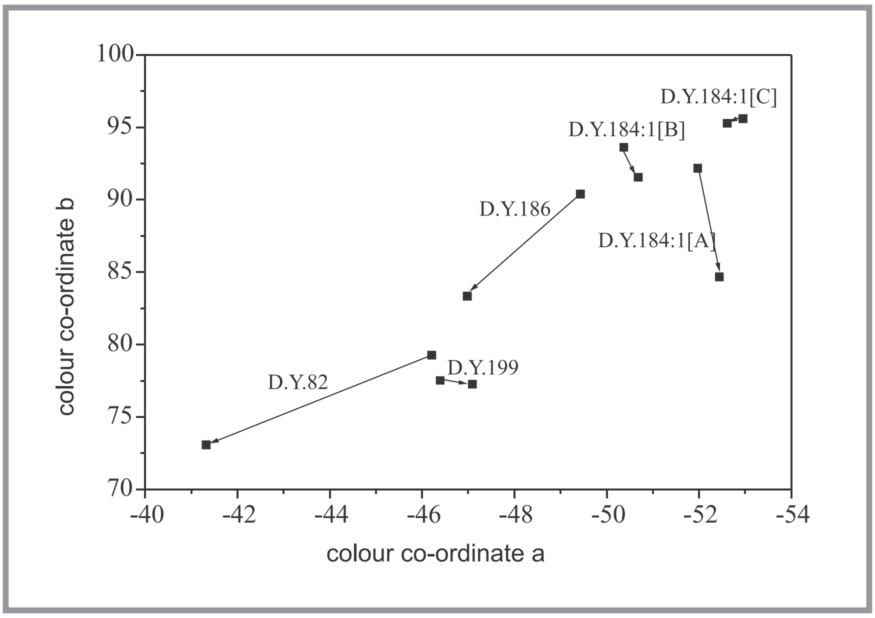

***Figure 4**. Colour differences of dyeings carried out with the use of the dyes tested before and after illumination of the samples in the Xenotest apparatus, presented in the a-b colour co-ordinates; *the arrows indicate the directions of the position displacements for the individual sample colours within the a-b co-ordinates after sample illumination.*

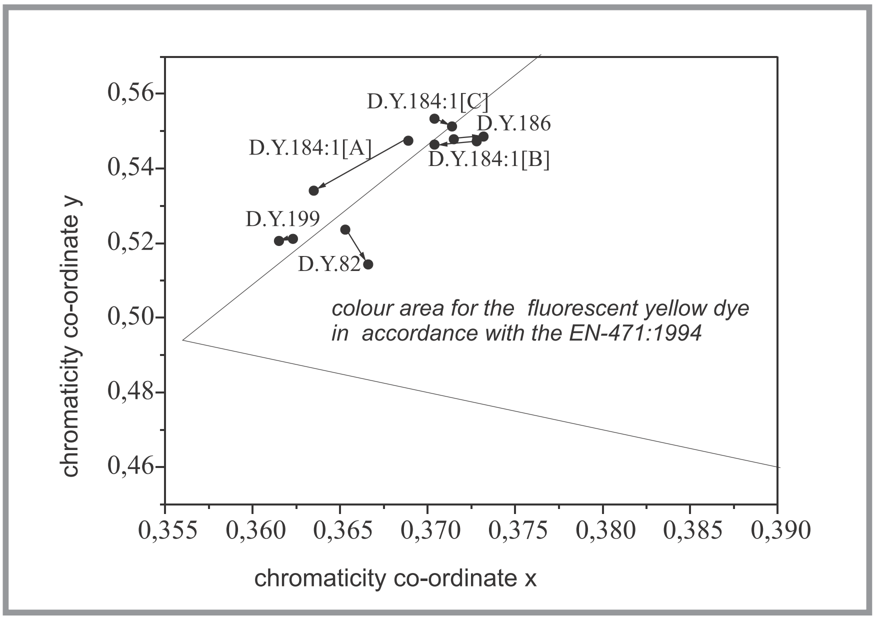

***Figure 5.** Colour differences of dyeings carried out with the use of the dyes tested before and after illumination of the samples in the Xenotest apparatus, presented in the x-y colour co-ordinates.*

obtained on a polyester knitted fabric. The colour chromaticities of the dyeings obtained with the use of all fluorescent dyes tested are positioned near the boundary of the area within the chromaticity x-y co-ordinates required by the standard for the fluorescent yellow colour. This statement is true of the dyeings which fulfil the standard requirements, as well as of those which do not meet these requirements.

- The yellow fluorescent dyes tested, when applied alone, do not guarantee the manufacturers that the polyester knitted fabrics dyed for high-visibility products will meet the requirements of the EN 471:1944 standard for the yellow colour.
- The commercial C.I. Disperse Yellow 184:1 dyes permit dyeings which either meet or do not meet the EN 471:1994 standard requirements, in relation to the dye producer. The compatibility in the chemical structure of these dyes does not ensure compatibility with the standard requirements.
- Dyeings whose hue after illumination shifts in the direction of red (increase in the a-parameter) achieve a colour of chromaticity parameters which is more similar to the requirements of the standard, despite their low light fastness.
- An analysis of the investigation carried out allows us to state that the requirements of the EN 471:1944 standard for chromaticity of the fluorescence yellow colours force the manufacturers of textile fabrics to apply tinting with the use of non-fluorescent dyes.

Editorial note:

The problems discussed in this paper were partially presented at the 19th Seminar of the Polish Colourists in Piła, Poland, 17-20 September, 2003.

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