

Studies in Dyeing. II. Effect of Crystal Modification of Disperse Dyes on Polyester Dyeing at 130°C

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Synopsis

Four commercial disperse dyes were purified and their different crystal forms were prepared by crystallizing from different solvents or precipitating from their solutions in glacial acetic acid by dilution with water. These forms were found to have different melting points. They were dyed on polyester fibers at 130°C to fiber saturation values by changing the dyebath every 15 min. The effect of pretreatment of the dyes in an aqueous environment at different temperatures (60°, 100°, and 130°C) for 15–60 min in the presence and absence of a dispersing agent on the dye uptake values was also studied. Such treatments are shown to reduce the dye uptake. The implication of these treatments in practical dyeing are pointed out.

INTRODUCTION

In an earlier communication,¹ the effect of crystal modification of four disperse dyes on the dyeing of polyester fibers at 100°C and of polyamide fibers at 75° and 100°C was reported. The different crystal modifications were characterized by melting points, x-ray diffraction patterns, and rate as well as extent of dyeing polyester fibers. The crystal modification does not appear to affect the dyeing behavior on polyamide fibers. The present communication deals with the dyeing of polyester fibers at 130°C with different crystal forms of four commercial disperse dyes.

EXPERIMENTAL

Fibers

Terene polyester fiber (1.5 den and staple length, 1.5 in.) manufactured by M/s. Chemicals and Fibres India Ltd., Bombay, was used after Soxhlet extraction with acetone for 8 hr.

Dyes

The following dyes were used: (1) Foron Yellow SE-FL (CI Disperse Yellow 42), (2) Dispersol RedB-3B (CI Disperse Red 11), (3) Dispersol Blue BG (CI Disperse Blue 26), and (4) Tulasteron Fast Yellow 5 RB (made in India by Atic Industries, Ltd.).

The commercial dyes (150 g) were vigorously boiled with distilled water (1000 ml) along with alum (about 100 g) until the dispersion broke. The dyes thus separated were filtered, washed until free from the dispersing agents and electrolytes, and dried at 60° ± 2°C. They were then Soxhlet extracted with ap-

TABLE I
Melting Points of Different Crystal Forms of Disperse Dyes

Dye	Form	Method of preparation	Melting point, °C
Foron Yellow SE-FL	I	benzene-crystallized	157
	II	chlorobenzene-crystallized	157
	III	methanol-crystallized	155
Dispersol Blue B-G	I	dimethylformamide-crystallized	does not melt
	II	precipitated from glacial acetic acid solution	does not melt
Tulasteron Fast Yellow 5RB	I	chlorobenzene-crystallized	151
	II	precipitated from glacial acetic acid solution	149
Dispersol Red B-3B	I	dimethylformamide-crystallized	226
	II	precipitated from glacial acetic acid solution	224

propriate solvents (benzene, chlorobenzene, methanol, and dimethylformamide) and crystallized. The crystallization from the corresponding solvent was repeated till the optical density of the purified dye remained constant. In three cases, the dyes freed from the dispersing agent and electrolytes were dissolved in glacial acetic acid at the boil, cooled, and filtered. The filtrate was slowly added to ice-cold distilled water and the precipitated dye was filtered and washed with distilled water till free from acetic acid.

The different forms of the dyes were dried at 30°C under vacuum. The melting points of these forms were determined in capillary tubes using an electrically heated Toshniwal melting point apparatus. The results are given in Table I.

Preparation of Dye Dispersion and Dyeing Procedure

Dye dispersions were prepared by the method described earlier.¹

The dyeing of polyester fibers was carried out in a laboratory-seal high-temperature beaker dyeing machine manufactured by M/s. Electronic and Engineering Co., Bombay. The machine consists of a pressure tank, a rotor assembly, and stainless-steel dyeing beakers with hinged lids provided with suitable gaskets for leak-proof closing. A pressure gauge and a safety valve are fitted on the tank. Six stainless-steel beakers are placed on the stainless steel rotor assembly in the pressure tank. The rotor assembly is connected to a motor through a gear box and rotated at a speed of 75 rpm. Heating is done by a ring-type electric heater which is clamped at the bottom of the pressure tank. An additional device is provided for regulating the rate of heating. The temperature is controlled automatically in the range of 70°–150°C within an accuracy of $\pm 3^\circ\text{C}$. Cooling of the bath is done by circulating water through water inlet and outlet valves.

Polyester fibers were dyed in this machine. The fibers and the dye dispersion (2% dye on the weight of the fiber with a material-to-liquor ratio of 1:50) were added to the beaker, which was introduced in the external water bath contained in the pressure tank kept at the appropriate temperature (60° or 100°C). The amount of the dispersing agent in the dyebath was adjusted to 1 g/l. The temperature of the dyebath was raised to 130°C and the dyeing continued while keeping the beakers rotated. At the end of 15 min, the machine was stopped,

TABLE II
Fiber Saturation Values for the Various Crystal Forms
of Disperse Dyes on Polyester Fibers Dyed at 130°C

Dye	Form	Duration of dyeing, min	Fiber saturation value, g/100 g fiber
Foron Yellow SE-FL	I	135	5.82
	II	150	4.20
	III	105	4.92
	commercial	120	0.46
Dispersol Blue B-G	I	120	2.48
	II	120	4.64
	commercial	150	2.24
Tulasteron Fast Yellow 5 RB	I	135	9.60
	II	90	10.24
	commercial	150	4.50
Dispersol Red B-3B	I	90	3.05
	II	90	3.12
	commercial	90	0.76

cooled quickly, opened, and a small portion of the dyed fiber was cut off and washed with acetone at 0°C till no further bleeding of the dye from the dyed fiber was noticed. This procedure was followed to remove any surface-deposited dye from the fiber. The remainder of the dyed fiber was dyed further with a fresh dye dispersion (2% dye on the weight of the fiber) at $130^{\circ} \pm 3^{\circ}\text{C}$ as before. A fresh dyebath was used every 15 min to avert any possible transition of a metastable form of the dye to a more stable one. The dyeing was continued until the fiber saturation value was reached.

Analysis of the Dyed Fiber

The dyed fibers were washed with acetone at 0°C and dried, followed by conditioning at 65% R.H. at $30^{\circ} \pm 2^{\circ}\text{C}$. An accurately weighted quantity (10–40 mg) of the dyed fiber was extracted with chlorobenzene and the extract was made up to a known volume (10–160 ml). The dye present in the solution was determined using a Hilger Biochem Pattern Absorptiometer using the appropriate filter. The dye content of the fiber is expressed in terms of g dye/100 g dyed fiber.

RESULTS AND DISCUSSION

The different forms of Foron Yellow SE-FL and Tulasteron Fast Yellow 5 RB as well as their commercial forms were dyed on polyester fibers at 130°C, adding the dye and the fiber at 100°C and changing the dye dispersion every 15 min until the fiber saturation value was reached. The rate of dyeing curves for these forms are shown in Figure 1.

Fiber saturation values and the time required to achieve them for all the forms of the dyes studied in the present investigation are given in Table II. It is seen that both the rate and the extent of dyeing vary with the crystal form of the dyes when dyed on polyester fibers at 130°C. Considerable differences are found in

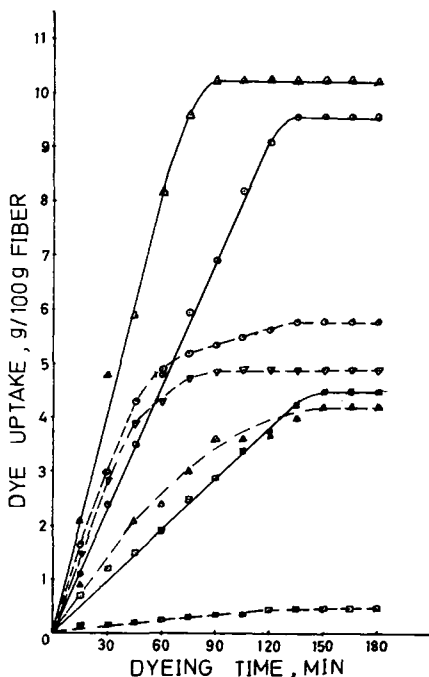


Fig. 1. Rate of Dyeing curves for different forms of disperse dyes on polyester at 130°C: (---) Foron Yellow SE-FL; (—) Tulasteron Fast Yellow 5 RB; (O) Form I; (Δ) Form II; (∇) Form III; (\square) commercial dye.

the fiber saturation values for Dispersol Blue BG. In all the cases, the commercial dyes dye the fiber least when compared with other forms.

Effect of Dispersing Agent and Time of Preheating on Dyeing Behavior

It has been reported by Biedermann² that boiling of a dispersion of a disperse dye in the presence of a dispersing agent for about 1 hr changes the crystal form of the dye to a more stable form which dyes cellulose acetate to a lower extent. In order to see whether such pretreatment of the different forms of the dye affects polyester dyeing, the two forms (I and II) of Dispersol Blue B-G as well as of Tulasteron Fast Yellow 5 RB were heated at 60°, 100°, and 130°C for 15–60 min in the presence and absence of the dispersing agent, and then the dyeing of polyester fibers was carried out at 130°C for 3 hr without interruption. The dye uptake was determined in each case by the method described earlier, and the results are given in Table III.

It is seen that for Form II of Dispersol Blue B-G, appreciable changes in dye uptake occur with increasing time of preheating (15–60 min) of the dye dispersion both in the presence and absence of the dispersing agent. The changes in the dye uptake are not very significant when the temperature of pretreatment was varied (60°–130°C) both in the presence and absence of the dispersing agent. Higher dye uptake values are obtained when the pretreatment is given in the absence of the dispersing agent than in its presence.

In the case of Form I of the dye, which is more stable than Form II, time, temperature, and presence of the dispersing agent does not appear to significantly

TABLE III
Effect of Preheating Dye Dispersion on Dye Uptake

Dura- tion of pre- heating min	Dye uptake ^a g/100 g fiber											
	Form I						Form II					
	In presence of dispersing agent		In absence of dispersing agent		In presence of dispersing agent		In absence of dispersing agent		In presence of dispersing agent		In absence of dispersing agent	
	60°C ^a	100°C	130°C	60°C	100°C	130°C	60°C	100°C	130°C	60°C	100°C	130°C
15	1.04	1.04	1.02	1.09	1.09	1.08	Dispersol Blue B-G					
30	1.04	1.04	1.00	1.09	1.09	1.08	1.46	1.42	1.38	1.53	1.50	1.48
45	1.04	1.02	0.96	1.09	1.08	1.04	1.42	1.38	1.34	1.50	1.46	1.46
60	1.02	1.00	0.92	1.06	1.04	0.96	1.36	1.34	1.32	1.46	1.42	1.38
15	0.96	0.84	0.84	1.02	0.94	0.94	Tulasteron Fast Yellow 5 RB					
30	0.84	0.76	0.68	0.89	0.88	0.76	1.40	1.36	1.02	1.58	1.56	1.36
45	0.72	0.60	0.46	0.80	0.76	0.60	1.28	1.28	0.84	1.52	1.48	1.22
60	0.64	0.54	0.32	0.72	0.68	0.49	1.20	1.20	0.76	1.40	1.44	1.02

^a Preheating temperature.

TABLE IV
Effect of Changing the Temperature of Dyestuff Addition on Dye Uptake

Initial dyebath tempera- ture, °C	Dye uptake, g/100 g fiber		
	Form I	Form II	Form III
	Foron Yellow SE-FL		
60	1.64	1.58	1.60
100	1.66	1.66	1.66
	Dispersol Blue BG		
60	1.04	1.50	—
100	1.09	1.64	—
	Tulasteron Fast Yellow 5 RB		
60	0.94	1.60	—
100	1.10	1.78	—
	Dispersol Red B-3B		
60	1.30	1.74	—
100	1.38	1.78	—

affect the dye uptake, except pretreatment at 130°C for 1 hr in the presence of the dispersing agent, which produces the least dye uptake value.

It is seen that in the case of Tulasteron Fast Yellow 5 RB, both the time and temperature of preheating considerably affect the dye uptake both in the presence and absence of the dispersing agent. Thus, with the Form II, the dye uptake of 1.48 g/100 g fiber when preheated at 60°C for 15 min in the presence of the dispersing agent drops to 0.76 g/100 g fiber when preheated at 130°C for 1 hr. The corresponding dye uptake values in the absence of the dispersing agent are 1.68 and 1.02, respectively.

Similar dyeing experiments with the different forms of Dispersol Red B-3B and Foron Yellow SE-FL showed that the variations in time, temperature, and presence or absence of the dispersing agent in the preheating bath brought about marginal changes in the dye uptake values.

To see whether the temperature at which the dye is added to the dyebath affects the dye uptake, the dyeings were carried out with different forms of the dyes at 130°C by adding the dyes at 60°C and 100°C and then raising the temperature of the dyebath to 130°C (the former took about 22 min and the latter, 12 min to reach 130°C). In both cases, the dyeing was continued for 3 hr at 130°C without interruption. The dye uptake values were determined as before, and the results are given in Table IV. It is seen that, except for Foron Yellow SE-FL, the different forms dye to different extents when dyed on polyester fibers at 130°C for 3 hr without changing the dyebath. Further, when the dye was added at a higher temperature (100°C), increased dye uptake was achieved for both forms of Tulasteron Fast Yellow 5 RB. For other dyes, differences due to variation in the temperature of dyestuff addition are marginal or nonexistent.

Thus, it may be concluded that the pretreatment of disperse dyes with a dispersing agent in an aqueous environment over the temperature range of 60°–130°C for 15–60 min affects the dye uptake values of different disperse dyes to different extents. This is of utmost importance in beam dyeing of polyester fabrics (or its blends with cellulosic fibers) at 130°C. It is the usual mill practice to add the disperse dye and the dispersing agent at about 60°C and then raising

the temperature of the dyebath gradually to 130°C in 30–45 min and dye at this temperature for 60–90 min. It is likely that since with most of the dyes, the dyeing rarely starts below 100°C, the dye is in contact with the dispersing agent in the aqueous medium at 60°–100°C for a fairly prolonged period (20–40 min), during which time crystal modification of the dye (to a more stable form, which lowers the dye uptake as well as the dyeing rate) can take place. In such cases, it is worthwhile to add the dye to the dyeing machine at about 100°C and then raise the temperature to 130°C in as short a time as possible (provided this does not lead to the production of uneven dyeings) so as to retain the metastable form of the dye and to get a better color value (dyeing yield) in practical dyeing in textile mills. In any case, such dyeing data on the effect of time, temperature, and dispersing agent have to be obtained in the case of all disperse dyes by the dyer himself to get the maximum color value in the dyehouse.

References

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