



## Enaminones-Assisted Synthesis of Disperse Dyes. Part 1: Low Temperature Dyeing of Polyester Fabrics

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**C**ARRIER coloring is a technique for dyeing polyester materials that is utilized when vital. Use of carriers in coloring empowers the coloring of polyester materials at atmospheric pressure. A series of new disperse dyes has been prepared and used in dyeing polyester fabrics at a temperature of 100 °C and a study of the optimum conditions for the use of both the carrier and the dispersing agent. The relationship between the dye concentration used in dyeing polyester fabrics with dispersed dyes and the color strength K/S were studied using three different concentrations of dye weight. Finally, polyester fabrics dyed with disperse dyes have shown that they possess acceptable fastness properties of light and are very good fastness against washing, perspiration and rubbing.

**Keywords:** Disperse dyes, Carrier.

### Introduction

Carriers have been generally used to color polyester strands in open baths in which the temperature cannot be expanded over 100°C [1–3]. Carriers are certain compounds that are added to the dye bath. They have low atomic weights and can be assimilated rapidly and quicken the dyeing rate. Numerous theories have been proposed to clarify the carrier activity. The most significant viewpoints have been contemplated considering the impact that the carrier have on dye absorption are activity inside the fiber and activity in the dye bath [4–6]. Undoubtedly it is basic for the carrier to be absorbed first by the fiber and that the level of action relies upon the amount absorbed. It is likewise realized that the absorption of run of the carrier compounds adjust the viscoelastic properties of the fiber. Various classes of color may show contrasting exercises with a similar carrier, and adjustments of conduct might be observed. It is likely that many mechanisms may work all the while to changing degrees that rely on the dye and the carrier being used [7]. The greater part of the more effective carrier substances have generally low water

solubility and must be utilized as emulsions within the sight of reasonable dyeing agents. In this paper, the dyeing behavior of disperse dyes 1–6 was examined within the presence of a carrier at increasing dye bath concentrations. The dyeing behaviors of the individual dyes are exhibited by utilizing the K/S values as dye uptake of the dyed samples.

### Materials and Methods

#### *General Procedure for the Synthesis of Disperse Dyes 1–6*

The disperse dyes were prepared according to the method that we published in our previous research [8].

#### *Dyeing at 100°C using carrier (low temperature dyeing)*

For the first disperse dye 1 a dispersion of the dye was produced by dissolution of the appropriate amount of dye (1% shade) in 2 ml DMF and then added dropwise with stirring to the dye bath (liquor ration 1:30) containing 1% leveal MDL as anionic dispersing agent (TANATEX chemicals) and different concentration of TANAVOL EP 2007 (0.5, 1, 1.5, 2%) as anionic eco friendly

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carrier (TANATEX chemical) The pH of the dye bath was adjusted to 4.5 with aqueous acetic acid, and the wetted-out polyester fibers (3 gm) were added. We performed dyeing by raising the dye bath temperature to 100°C at a rate of 3 °C/min and holding it at this temperature for 60 min. After they were cooled to 50 °C, the dyed fibers were rinsed with cold water and reduction-cleared (1 g/L sodium hydroxide, 1 g/L sodium hydrosulfite, 10 min, 80 °C). The samples were rinsed with hot and cold water and, finally, air-dried. The same procedure performed for the remained five disperse dyes. After that the above procedures repeated again with different concentration of disperse leveling (0.5, 1, 1.5, 2%) and 1% eco friendly carrier for each dyes.

#### *Color measurements*

The colorimetric parameters of the dyed polyester fabrics were determined on a reflectance spectrophotometer. The color yields of the dyed samples were determined by using the light reflectance technique performed on an UltraScan PRO D65 UV/VIS Spectrophotometer. The color strengths, expressed as K/S values, were determined by applying the Kubelka-Mink equation [9].

$$K/S = (1 - R)^2 / 2R$$

Where, R is the reflectance of colored samples and K and S are the absorption and scattering coefficients, respectively.

#### *Color fastness to washing*

The color fastness to washing was determined according to the ISO 105-C02:1989 method [10]. The composite specimens were sewed between two pieces of bleached cotton and wool fabrics, and then immersed into an aqueous solution containing 5 g/L of nonionic detergents at a liquor ratio of 1:50. The bath was thermostatically adjusted to 60 °C for 30 min. After the desired time, samples were removed, rinsed twice with occasional hand squeezing, and then dried. Evaluation of the wash fastness was established using the grey scale for color change.

#### *Color fastness to rubbing*

Color fastness to rubbing was determined according to the ISO 105-X12:1987 test method. The test is designed for determining the degree of color that may transfer from the surface of the colored fabrics to another surface by rubbing. The current test can be carried out on dry and wet fabrics.

#### *Dry crocking test*

The test specimen was placed flat on the base of the crockmeter. A white testing cloth was mounted. The covered finger was lowered onto the test specimen and caused to slide back and forth 20 times. The white test sample was then removed for evaluation using the grey scale for staining.

#### *Wet crocking test*

The white test sample was thoroughly (65%) wetted with water. The procedure was run as before. The white test samples were air dried before evaluation.

#### *Color fastness to perspiration*

Two artificial perspiration solutions (acidic and alkaline) were prepared as follows according to the ISO 105-E04:1989 test method. The acidic solution was prepared by dissolving L-histidine monohydrochloride monohydrate (0.5 g), sodium chloride (5 g), and sodium dihydrogen orthophosphate dihydrate (2.2 g) in one liter of distilled water. Then, the pH was finally adjusted to 5.5 using 0.1 N NaOH. To prepare the alkaline solution, L-histidine monohydrochloride monohydrate (0.5 g), sodium chloride (5 g), and disodium hydrogen orthophosphate dihydrate (2.5 g) were all dissolved in one liter of distilled water. The pH was adjusted to 8 using 0.1 N NaOH. The fastness test was performed as follows. The 5 cm × 4 cm colored specimen was sewn between two pieces of uncolored specimens to form a composite specimen. The composite samples were immersed for 15-30 min in both solutions with well agitation and squeezing to ensure complete wetting. The test specimens were placed between two plates of glass or plastic under a force of about 4-5 kg. The plates containing the composite specimens were then held vertically in an oven at 37 ± 2 °C for 4 h. The effect on the color of the tested specimens was expressed and defined by reference to the grey scale for color change.

#### *Color fastness to light*

The light fastness test was carried out in accordance with the ISO 105-B02:1988 test method, using a carbon arc lamp and continuous light for 35 h. The effect on the color of the tested samples was recorded by reference to the blue scale for color change.

### **Results and Discussion**

In our previous research, a number of

new disperse dyes were synthesized by reacting enaminones compounds [10] with arylidenediazonium salts to obtain disperse dyes 1-6 with an excellent yields (Fig. 1). Their chemical composition was verified by scientific methods, for example using IR, proton NMR and Mass spectra [10]. In this study these new disperse dyes were used in dyeing polyester fabrics at a low temperature of 100 °C, in the presence of both the dispersing agent and the carrier, the colors ranged from light yellow to dark yellow.

#### Effect of carrier on K/S

When we dye the polyester fabrics with the new disperse dyes, we used the dispersing agent at a concentration of 1% and we studied the use of the carrier at different concentrations from 0.5 to 2% to study the optimum appropriate concentration giving the best value of color strength K/S which represent the degree of dye uptake. The results set out in Table 1 indicate that the color strength K/S values of the polyester fabrics dyed with the disperse dyes of dyes 1, 2 and 5 increase with an increase in the concentration of the carrier and reach their highest value (10.08, 11.24 and 10.13) at a concentration of 1%. Then they decrease after. The color strength K/S values for the polyester fabrics dyed with the disperse dyes 3 and 6 decrease with an increase in the concentration of the carrier and reach their highest value (6.66

and 10.10) at a concentration of 0.5%. Finally, the K/S values for the polyester fabrics dyed for dye No 4 reaches its highest value (14.04) at 2% concentration of the carrier.

#### Effect of dispersing agent on K/S

The polyester fabrics were dyed with the new disperse dyes 1-6 using the carrier at a concentration of 1%, and we studied the use of the dispersing agent at different concentrations from 0.5 to 2% to study the optimum concentration giving the best value of color strength K/S. The results set out in Table 2 indicate that the K/S values of the polyester fabrics dyed with the disperse dyes of the dyes 1, 3, 5 and 6 increase with the increase of the dispersing agent concentration and reach their highest value (10.85, 10.67 13.01 and 7.54) at a concentration of 1.5 %. The K/S values for the polyester fabric dyed with the disperse dyes of the 2 and 4 dyes also increase with increasing dispersing agent concentration and reach their highest value (10.23 and 10.36) at a concentration of 2%. It is clear from the results described in Table 1 and 2 that the most appropriate optimum conditions for the use of the carrier are 1%, and that the most appropriate optimum conditions for the use of the dispersing agent are 1.5%, when dyeing polyester fabrics with new dispersed dyes at a temperature of 100 °C.

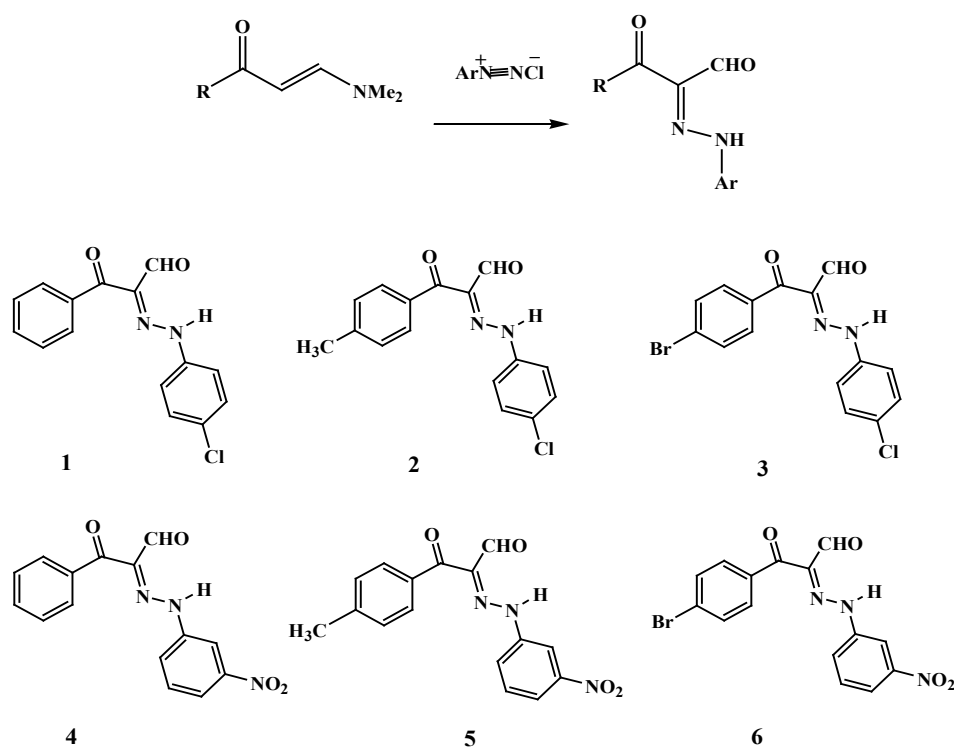


Fig. 1. Chemical structures of the disperse dyes.

**TABLE 1. Carrier effects on the dyeing process of disperse dyes by using 1% dispersing agent.**

Dye No	% carrier	L*	a*	b*	C*	h*	K/S
1	0.5%	81.35	-10.33	53.63	54.61	100.91	9.07
	1.0%	81.24	-10.18	56.44	57.35	100.22	10.08
	1.5%	81.54	-10.42	55.70	56.76	100.60	9.82
	2.0%	81.38	-9.94	57.43	58.29	99.82	8.70
2	0.5%	81.67	-11.52	54.20	55.41	102.00	10.51
	1.0%	81.49	-11.41	54.85	56.03	101.76	11.24
	1.5%	82.25	-12.81	52.10	53.65	103.81	10.19
	2.0%	82.79	-13.29	53.19	54.82	104.02	10.43
3	0.5%	83.18	-12.91	44.47	46.30	106.18	6.66
	1.0%	83.02	-12.75	45.50	47.25	105.66	6.53
	1.5%	82.96	-12.30	45.54	47.17	105.11	6.22
	2.0%	82.97	-12.25	45.82	47.43	104.97	6.40
4	0.5%	80.33	-5.64	40.41	40.80	97.94	12.95
	1.0%	81.35	-6.52	38.62	39.17	99.58	10.08
	1.5%	80.62	-6.62	38.87	39.43	99.66	11.48
	2.0%	81.09	-6.33	40.58	41.07	98.86	14.04
5	0.5%	81.28	-6.06	37.91	38.39	99.08	8.48
	1.0%	81.61	-6.33	36.91	37.45	99.73	10.13
	1.5%	82.11	-7.04	36.08	36.76	101.04	7.94
	2.0%	81.13	-6.07	38.83	39.30	98.89	8.81
6	0.5%	81.79	-6.92	37.76	38.39	100.38	10.10
	1.0%	81.32	-6.75	39.03	39.61	99.82	7.24
	1.5%	81.32	-6.63	39.63	40.18	99.50	6.96
	2.0%	81.38	-7.26	38.60	39.28	100.65	7.41

**TABLE 2. Dispersing agent effects on the dyeing process of disperse dyes by using 1% carrier.**

Dye No	% Dispersing agent	L*	a*	b*	C*	h*	K/S
1	0.5%	81.14	-10.13	57.18	58.07	100.05	9.46
	1%	81.77	-10.41	57.08	58.02	100.34	9.78
	1.5%	80.78	-10.39	57.13	58.07	100.31	10.85
	2%	80.99	-10.37	57.26	58.19	100.26	10.36
2	0.5%	82.24	-13.61	51.69	53.71	104.68	9.41
	1%	82.20	-13.54	50.77	52.54	104.93	9.89
	1.5%	82.75	-13.10	50.97	52.62	104.41	8.48
	2%	80.08	-12.98	50.44	52.08	104.44	10.23
3	0.5%	81.71	-12.30	54.77	56.14	102.66	9.29
	1%	82.74	-13.64	51.44	53.17	104.67	9.31
	1.5%	82.43	-13.01	51.32	52.95	104.23	10.67
	2%	82.13	-13.11	51.89	53.52	104.18	10.24
4	0.5%	80.82	-6.12	38.11	38.60	99.12	9.37
	1%	81.03	-5.87	36.92	37.38	99.03	7.88
	1.5%	80.69	-6.33	37.43	37.97	99.59	7.15
	2%	81.36	-6.54	36.42	37.00	100.18	10.36
5	0.5%	81.06	-6.67	36.84	37.44	100.27	8.64
	1%	82.17	-7.01	36.80	37.46	100.78	9.61
	1.5%	81.95	-6.82	35.82	36.46	100.79	13.01
	2%	81.50	-6.64	38.91	39.47	99.68	9.89
6	0.5%	82.23	-6.72	33.42	34.09	102.15	4.44
	1%	82.30	-7.26	33.73	34.51	101.12	6.62
	1.5%	82.05	-6.91	34.70	35.38	101.26	7.54
	2%	81.76	-7.32	37.22	37.94	101.12	7.29

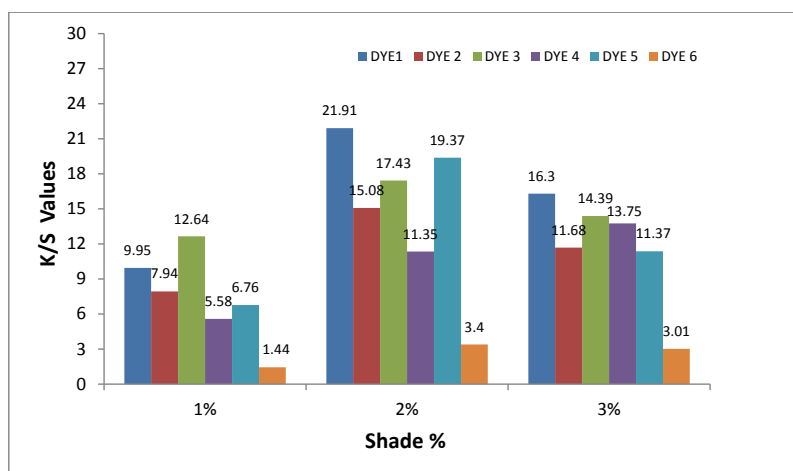
*Relation between dye concentrations of the disperse dyes and K/S*

From the appraisal of K/S esteems, it is presumed that distinctive shade of disperse dyes show changing behavior because of increasing dye bath concentrations. Regularly carrier is added to the dye at atmospheric pressure at 100°C and advance the shading at this degree. It tends to be seen that carrier addition changes the shading esteems relying upon hue of the dye, dye bath concentration. In an attempt to find the relationship between the concentration of the dye used in dyeing polyester fabrics with the new dispersed dyes and the intensity of the color resulting from that dyeing process on the dyed

fabrics, the dyeing process was done by using three concentrations of each dye 1, 2 and 3%, and the color strength was measured and results are presented in Table 3. Figure 2 and Table 3 indicate that the color strength K/S values for the polyester fabrics dyed with the disperse dyes of the dyes 1, 2, 3, 5 and 6 increase with increasing the dye concentration and reach their highest value (21.91, 15.08, 17.43, 19.37 and 3.40) at dye concentration 2%. As for the color strength K/S values of the polyester fabric dyed with disperse dyes No. 4, it increases with increasing the dye concentration and reaches its highest value (13.75) at the dye concentration 3%.

**TABLE 3. Effect of the dye shades used in dyeing process at 100 °C and K/S of dyed fabrics.**

Dye No	% shade	L*	a*	b*	C*	h*	K/S
1	1%	87.88	-5.65	67.75	67.98	94.77	9.95
	2%	85.63	-2.60	81.84	81.88	91.82	21.91
	3%	83.94	-1.03	83.13	83.14	90.71	16.30
2	1%	87.44	-9.58	60.92	61.67	98.94	7.94
	2%	88.07	-8.19	71.01	71.49	96.58	15.08
	3%	86.45	-8.60	67.57	68.11	97.26	11.68
3	1%	88.46	-9.35	68.29	68.93	97.80	12.64
	2%	85.09	-6.96	71.83	71.72	95.57	17.43
	3%	87.12	-3.89	78.98	79.08	92.82	14.39
4	1%	87.68	-1.11	47.54	47.55	91.33	5.58
	2%	83.19	-1.20	53.54	53.55	91.29	11.35
	3%	81.17	-0.23	51.38	51.38	90.26	13.75
5	1%	83.22	-3.31	45.18	45.30	94.19	6.76
	2%	75.14	-1.59	48.83	48.86	91.87	19.37
	3%	83.62	-1.38	50.11	50.12	91.57	11.37
6	1%	88.59	-4.68	26.43	26.84	100.03	1.44
	2%	86.77	-3.98	36.28	36.50	96.26	3.40
	3%	85.30	-2.29	37.78	37.85	93.47	3.01



**Fig. 2. Relation between dye concentrations and shades of the disperse dyes.**

*Fastness properties*

The fastness properties of polyester fabrics dyed with the new disperse dyes have been done at shades from 1% to 3%, and the results set in Tables 4-6 show the fastness against washing gave very good results and that the shade of 2% was better than both shades 1% and 3%.

The shade 2% was like 3% with respect to rubbing fastness, but shade 1% was better than them, and this is self-evident since the color strength of shade 1% less than that in shades 2% or 3%, and as color strength of shade 2% greater than shade 3%, thus shade 2% was better than shade 3%, and also the results of rubbing fastness were very good.

**TABLE 4. Fastness properties of disperse dyes on polyester fabrics at shade 1%**

Dye No	Washing fastness			Rubbing fastness		Perspiration fastness						Light fastness
						Acidic			Alkaline			
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	
1	4	4	4	4-5	4-5	4-5	4	4	4-5	4-5	4	3
2	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	3
3	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
4	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	3
5	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4-5	4	3
6	4-5	4	4-5	4	4-5	4	4	4	4-5	4	4	2-3

**TABLE 5. Fastness properties of disperse dyes on polyester fabrics at shade 2%**

Dye No	Washing fastness			Rubbing fastness		Perspiration fastness						Light fastness
						Acidic			Alkaline			
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	
1	4-5	4	4-5	4-5	4	4-5	4-5	4	4-5	4-5	4	2-3
2	4-5	4-5	4-5	4-5	4	4-5	4	4	4-5	4	4	3
3	4-5	4	4-5	4	4	4	4	4	4-5	4-5	4	2-3
4	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
5	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
6	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	3-4

**TABLE 6. Fastness properties of disperse dyes on polyester fabrics at shade 3%**

Dye No	Washing fastness			Rubbing fastness		Perspiration fastness						Light fastness
						Acidic			Alkaline			
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	
1	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
2	4-5	4	4-5	4-5	4	4-5	4-5	4	4-5	4	4	2-3
3	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
4	4-5	4	4-5	4	4	4	4	4	4-5	4-5	4	2-3
5	4-5	4	4-5	4-5	4	4-5	4-5	4	4-5	4	4	2-3
6	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4-5	4	3-4

When looking at fastness against perspiration, it gave very good results, and the three shades 1%, 2% and 3% were almost identical, and however, shade 3% was a little better in acidic perspiration than the rest. Finally, the fastness properties against light gave acceptable results, and the results of shade 1% were slightly better than both shades 2% and 3%, and also shade 2% was slightly better than shade 3%. In general, and given that the K/S values of shade 2% is greater than both of shade 1% and 3%, the fastness properties of the polyester fabric dyed with the new disperse dyes 1-6 at shade 2% is preferable one if we compare it with the rest shades.

### Conclusion

This study can be summarized in preparing some new disperse dyes and using them in dyeing polyester fabrics at a temperature of 100 °C, and the optimum conditions for using the carrier were 1%, and the most appropriate optimum conditions for using the dispersed agent is 1.5%. When studying the relationship between the dye concentration used in dyeing polyester fabrics with the new disperse dyes and the resulting color strength K/S, this study showed that the K/S values of dyed polyester fabrics with the disperse dyes 1, 2, 3, 5 and 6 increase with increasing dye concentration and reach up to the top its values at the dye concentration 2%. As for the disperse dyes No 4, the dye concentration was 3%. When studying the fastness properties of these dyes, the results showed that the fastness properties of polyester fabric dyed with disperse were the best at shade 2% and were very good for fastness against washing, perspiration and rubbing, and acceptable for light fastness

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## تشبيد بعض الصبغات المنتشرة بمساعدة الإينامينونات. جزء ١: صباغة أقمشة البولي استر في درجة حرارة منخفضة

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الصباغة باستخدام العامل الحامل هو تقنية حيوية لصباغة مواد البولي استر. استخدام تلك المواد الحاملة في التلوين تمكن تلوين مواد البولي استر في الضغط الجوي العادى. تم إعداد سلسلة من الأصباغ الجديدة المنتشرة واستخدامها في صباغة أقمشة البولي استر عند درجة حرارة 100 درجة مئوية ودراسة الظروف المثلى لاستخدام كل من العامل الحامل وعامل التثبيت. تمت دراسة العلاقة بين تركيز الصبغة المستخدمة في صباغة أقمشة البولي استر وشدة اللون باستخدام ثلاثة تركيزات مختلفة من وزن الصبغة. أخيراً ، أظهرت أقمشة البولي استر المصبوغة بالأصباغ المنتشرة أنها تمتلك خصائص ثبات مقبولة للضوء وخصائص ثبات جيد للغسيل والعرق والاحتكاك.