

Dyeing of Polyester Fabric using Nano Disperse Dyes and Improving their Light Fastness using ZnO Nano Powder

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Abstract

In this work a Disperse red 60 and Disperse blue 56 were converted to the nano scale using ultrasonic homogenizer and characterized by transmission electron microscopy. Dyeing of polyester fabrics was carried out in the temperature range of 70–100°C and compared with the polyester fabrics dyed with the same dyes without sonication. Dyeing was carried out using two different carriers: salicylic acid and HC carrier. ZnO nano powder was used to improve the light fastness for the dyed polyester fabrics.

Keywords: Disperse Dyes, Light Fastness and ZnO Nano Powder, Nanotechnology, Polyester Fabric

1. Introduction

Polyester fiber has a highly compact and crystalline structure, and is markedly hydrophobic. For this reason, its aqueous dyeing is carried out at high temperature and high pressure using disperse dyes. The dyeing of polyester can be divided into several parallel processes such as dissolution and redissolution of disperse dye, transfer of dissolved dye from bulk solution to the fiber surface, diffusion and adsorption of dye at the fiber surface, and diffusion from the surface into the interior of the fiber. It is well known that the additives in the dyebath affect the dyeing processes.

Solvent-assisted and carrier dyeing has been widely studied as a means of accelerating dyeing rate, improving dye uptake and lowering dyeing temperature by means of changes in the physical properties of polyester, notably such as Glass transition Temperature (T_g). However, both solvents and carriers have serious problems, namely

toxicity and unpleasant odor, poor light fastness, an adverse effect on the physical properties of the fiber, high costs of waste water treatment and environmental contamination and destruction. Clearly, it seems sensible that efforts should be devoted toward the development of a new dyeing method to accelerate dyeing rate, improve dye uptake and lower dyeing temperature¹.

Nanotechnology is a very complex field combining science (biology, chemistry and physics), technology (computer programming), engineering (electronics and design) and math. Nanotechnology can be applied to many areas in textiles. They are used for producing nano-fibers, nano-composites, nano dyeing and nano-finish. Nano dyeing is the application of reduced size dyes that are smaller than 100nm in textile dyeing⁶.

Comprehensive research efforts have been made in the area of disperse dyes that have been used for the dyeing of hydrophobic fabrics^{2,5}. Consequently, in the last two decades, the research trends focused their efforts on

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the application of new technologies¹⁰ such as nanotechnology, electrochemistry, supercritical carbon dioxide dyeing, plasma, ultrasonic and microwave to modify and develop the dyeability of polyester fabric. Polyester is the most hydrophobic of all common fibers and has a highly compact and crystalline structure. For this reason, its aqueous dyeing is carried out at high temperature and high pressure using disperse dyes¹.

The nano colorants exhibited excellent chromatic properties attributed to the nano scale effects of homogeneous nano colorants⁷. The preparation of nano disperse dye by dry ball milling technique has been studied and applied in a comparative adsorption study onto hydrophobic fabrics¹³.

The purpose of this work was to prepare nano disperse dyes and determine whether nano dyeing could be used to intensify the dyeability of polyester fabric with a traditional disperse dye and its nano scale in a comparative study using chemical auxiliaries carriers.

2. Experimental

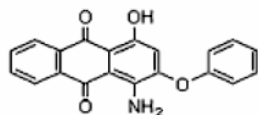
2.1 Materials

2.1.1 Fabric

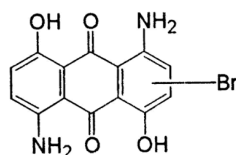
Scoured and bleached 100% polyester fabric (149 g/m²) was supplied by El-Mahalla El-Kobra Company. The fabrics were scoured in aqueous solution with a liquor ratio 1:50 containing 2 g/l nonionic detergent solution (Hostopal, Clariant) and 2 g/l Na₂CO₃ at 50°C for 30 min to remove impurities, then rinsed thoroughly in cold tap water, and dried at room temperature.

2.1.2 Dyes

Disperse red 60 and disperse blue 56 supplied by Dye star Company. Dispersing agents Matexil DA-N was supplied by ICI Company, commercial HC carrier supplied by Egyptian Turkish Co. for auxiliaries, salicylic acid as a carrier, acetone, dimethylformamide (DMF), sodium hydroxide, sodium hydrosulphate and acetic acid were laboratory reagent grade chemicals.



C.I. Disperse red 60



C.I. Disperse blue 56

2.1.3 Sonication of Dyes

The Disperse red 60 and disperse blue 56 were operated to ultrasound homogenizer for different periods of time from 15-90 min by Ultrasonic stirrer (Sonic & Materials, INC, model :VCX750, volts:230VA 50/60HZNOM, U.S.A).

2.1.4 Synthesis of ZnO Nano Particles

Zinc oxide nano particles were synthesized^{8,9}. Synthesis was carried out at a high degree of super saturation in order to achieve a nucleation rate much greater than the growth rate (Kwon et al., 2002) (5.5 g) ZnCl₂ (98%) was dissolved in 200 mL of water at 90°C in an oil bath. Then 16 mL of 5 M NaOH (pellet min.99%) aqueous solution was added drop-wise to the zinc chloride solution with a gentle stirring over a period of 10 min at 90°C. The particles were separated from the supernatant dispersion by sedimentation. The supernatant solution was discarded and the remaining suspension was washed five times with distilled water to lower the concentration of NaCl below 6-10M. Each time, the dilution ratio between the concentrated suspension and the washing solution was about 1:10. The complete removal of NaCl from the suspension was checked using a solution of AgNO₃. The purified particles were then peptized with 2-propanol (98%) in an ultrasonic bath for 10 min at room temperature. The peptization process is necessary to disrupt the micro-agglomerates and to release the nano particles of zinc oxide¹².

The particles were then collected by centrifugation at 6,000 rpm for 15 min. The washing procedure was repeated three times. Thermal treatment of the particles at 250°C for 5 h leads to the formation of ZnO.

2.1.5 Dyeing Process at 130°C

To determine the dyeing process behavior of polyester fabric with DR60 and DB56, 2 g fabric samples were introduced in a flask containing 2% (w.o.f) dye with 1:50 liquor ratio at 130°C and pH 4. At the end of dyeing, the dyed samples were removed, rinsed in warm water and treated in a solution containing sodium hydrosulphite 2g/L, Sodium hydroxide (caustic soda) 2g/L for 10 minutes at 60°C, liquor ratio 1:40, the reduction cleared sample was rinsed thoroughly in water. Rinse well in cold water and neutralize with 1g/L Acetic acid for 5 minutes at 40°C the dyed samples were removed, rinsed in tap water and allowed to dry in the open air.

2.1.6 Dyeing Process with Carriers

Dyeing process was carried out at 60°C, L:R 1:40. Dyeing bath contains 2% (w.o.f) dye, 4% (w.o.f), dispersing agent and 0.5–6% (w.o.f) carrier, pH were adjusted from 3.5 to 4, then the fabric were immersed and the temperature were raised to 70–100°C for 60 min. At the end of dyeing, the dyed samples were rinsed with tap water and allowed to dry in open air.

3. Fabric Treatment with zinc oxide

3.1 Pre-treatment

The fabric samples were soaked for 10 min. in 2-propanol (98%) dispersion of ZnO nano particles (5% w/w) under gentle magnetic stirring. The fabrics were squeezed to remove the excess dispersion and dried in an oven at 130°C for 15 min. under atmospheric pressure (dry heat).

3.2 Simultaneous Treatment

The zinc oxide nano powder was added to the dyeing bath during dyeing.

3.3 Post-treatment

The fabric samples were soaked for 10 min in 2-propanol (98%) dispersion of ZnO nano particles (5% w/w) under gentle magnetic stirring after dyeing. The fabrics were squeezed to remove the excess dispersion and dried in an oven at 130°C for 15 min. under atmospheric pressure (dry heat).

In order to evaluate the nanoparticles adhesion to the textile fibers, the treated fabrics were washed five times as standard method (UNI EN ISO26330:1996). Electrolux automatic laundry machine (internal drum diameter 51.5 cm, internal drum depth 33.5 cm and heating capacity 5.4 kW) was used, and the washing cycles were performed at 30°C with reference detergent without optical brighteners.

The drying step was carried out on a horizontal flat surface. The fabric specimens were tested before and after the washing cycles⁸.

4. Testing and Analysis

4.1 ZnO Particle Size

The synthesized powder was determined after appropriate sample dilution with distilled water and freshly

prepared by Transition Electron Microscope (TEM). One drop of each emulsion was mounted on a copper grid covered by a thin film of carbon and after drying, the samples were examined by TEM (Model EM-1230; Jeol, Germany) at high voltage (hV) 100 kV and with resolution ca. 10 Å. The average vesicle size distribution was determined either by volume or number of the particles in the drop.

4.2 Colour Measurements

The color strength expressed as (K/S) values of the dyed samples were measured by using a Hunter Lab Ultra Scan[®] PRO (USA) at the maximum wavelength. The relative color strength (K/S values) was assessed using the Kubelka–Munk equation (1)

$$K/S = \frac{(1-R)^2}{2R}$$

where, R is the reflectance of colored samples and K and S are the absorption and scattering coefficients, respectively. Summation method ($\sum(K/S)\lambda$) was used for an equally spaced selection of wavelength in range of 350–750 nm.

4.3 Light Fastness Improvement

The untreated and treated fabric (pre, simultaneous and post) with ZnO nano powder after dyeing the colored samples were exposed to light irradiation for different time intervals (0–140) hrs. DE for all dyed samples was measured before and after irradiation. The most widely used equation is CIE Lab difference equation.

4.4 CIE Lab Difference

$$\Delta E = L^2 + [(a^2 + b^2)]^{1/2}$$

ΔE : the total difference between the sample and the standard

L: represent white-black axis

a: represent the red-green axis

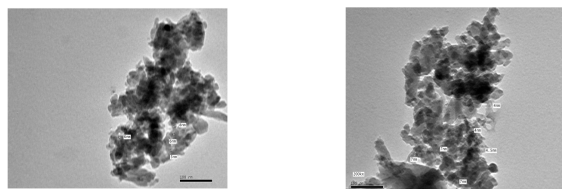
b: the yellow-blue axis

4.5 Fastness Properties

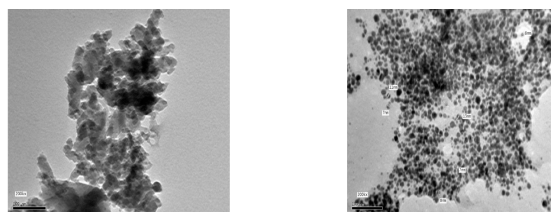
Fastness properties of the dyed samples were determined according to ISO standard methods. The specific tests were ISO 105-X12 (1987), colour fastness to rubbing; ISO 105-C02 (1989), colour fastness to washing and ISO 105-E04 (1989), colour fastness to perspiration.

5. Results and Discussion

Transition Electron Microscope (TEM) for the used dyes



TEM of Disperse blue 56 before sonication TEM of Disperse blue 56 after sonication



TEM of Disperse red 60 before sonication TEM of Disperse red 60 after sonication

The dyeing process is a solid/liquid phase process, which proceeds by the movement of the dye molecules from liquid phase to the solid surface of the fabric by virtue of their affinity, and then diffusion takes place inside the fabric. Therefore, the first process would be a fast adsorption controlled process where the dye molecules get into the fabric, the second slow process which is diffusion controlled, starts to take place. For the dye molecules to diffuse into the fabric, it is expected that the free volume could be formed within the fabric.

To evaluate the relationship between ultrasound treatment of the dye dispersion and the dyeing rate, Color yields (K/S) of the dyed fabrics was determined.

Ultrasonic cavitations are used to disperse nano-size particles into liquids. The application of ultrasonics to nano materials has manifold effects. The most obvious is the dispersing of materials in liquids in order to break particle agglomerates. Another process is the application of ultrasound during particle synthesis or precipitation. Generally, this leads to smaller particles and increased size uniformity. Ultrasonic cavitations improves the material transfer at particle surfaces, too. This effect can be used to improve surface functionalization of materials having a high specific surface area.

5.1 Effect of Time of Sonication on the k/s Value Without using Carriers (at 130°C)

The role of ultrasonication in preparation of Disperse red 60 and disperse blue 56 nanoscale was investigated

with respect to morphological structure with particle size determination. For this purpose, another batch of nanoscale Disperse red 60 and disperse blue 56 was prepared at different time intervals of ultrasonication (15–90 min).

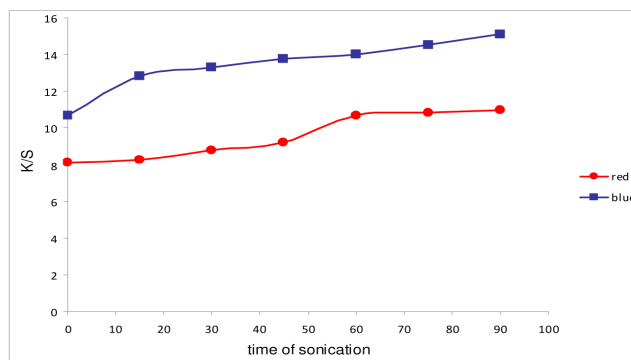
It is apparent from Figure 1 that increasing time of sonication from 15–90 min. leads to high increase in colour strength for polyester dyed with DR60 and/or DB56 compared with the nonsonicated dyes (at 0 time), that is because ultrasound application helped reduce the particle size, generating more particles at ultrasonication for 90 min.

Generated ultrasound waves make similarly-charged particles collide into one another with great force; consequently the problem of agglomeration is greatly reduced. In addition, high energy produced from particle collisions at high temperatures is responsible for breaking down the particles and decreasing particle size which has an impact on dispersion stability of disperse dyes^{3,4}.

In the light of these results the studying of dyeing polyester fabric with both dyes after sonication using two different carriers at low temperature may give better results than expected.

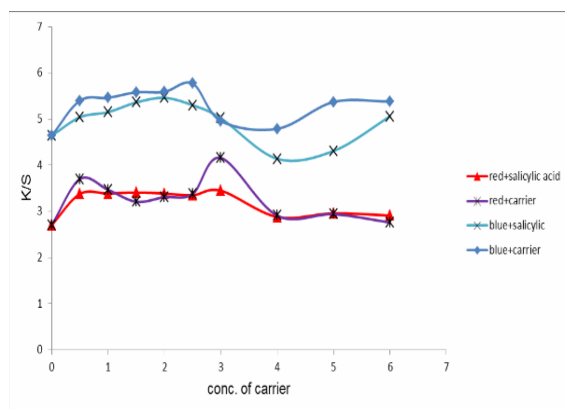
5.2 Effect of Concentration of Carrier without Sonication

Figure 2 shows that in case of the blue dye the increase of the conc. of carrier from 1–6% of the weight of the fabric there is a slightly increase of k/s value till 2.5% carrier concentration. In case of the red dye for both carriers the highest k/s value was obtained at 3% concentration.



Dye 2% at 130°C with 1:50 liquor ratio at pH 4

Figure 1. Effect of time of sonication on the k/s value without using carriers (at 130°C).



Dye 2% (w.o.f), L.R 1:40, pH 3.5-4, temp. at 100°C, dispersing agent 4% (w.o.f).

Figure 2. Effect of the concentration of carriers on the dyeing.

5.3 Effect of Time of Sonication on the k/s Value with using Carriers

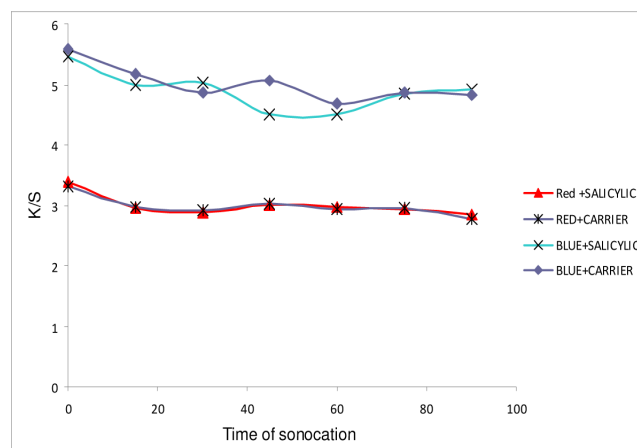
In case of using carriers the dyeing was carried out at 100 °C. Figure 3 shows that better results for both dyes and carriers (salicylic acid and HC carrier) obtained at 0 time of sonication and the value of k/s are very small compared to these of dyeing at 130°C without using carriers. These results revealed that these dyes are in the nano size before sonication, that may be explained the best distribution of the dyes particles at 130°C.

5.4 Effect of Dyeing Temp

Figure 4 shows that as the temperature of dyeing increases from 70-100°C the k/s values increases for both dyes and for both carriers this is attributed to the increase of the kinetic energy of the dye molecules and the high distribution rate.

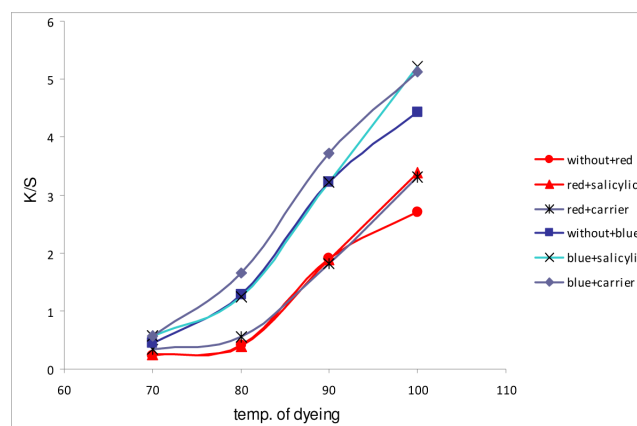
5.5 The Total Color Difference (DE)

We have measured ΔE for all dyed samples before irradiation and after irradiation from 0 to 140 hours according to CMC 2:1 equation which gives more accurate results, the results indicates that the results of ΔE which represent the color different rate for the exposed dyed fabrics with or without ZnO nano powder for the two different dyes and the two different carriers compared to the sonicated and not sonicated dyes at 100°C. The light fastness of the dyed samples at 130°C are very good (6-7 in the blue scale) and there is no need to use ZnO.



Dye 2% (w.o.f), L.R 1:40, pH 3.5-4, temp. at 100°C, conc. of carrier 2% (w.o.f), dispersing agent 4% (w.o.f).

Figure 3. Effect of time of sonication on the k/s value of dyeing polyester fabrics with DR60 and DB56 using two different carriers.



Dye 2% (w.o.f).L.R 1:40, pH 3.5-4, dispersing agent 4% (w.o.f).conc. of carrier 2% (w.o.f).

Figure 4. Effect of dyeing temp.

The comparison between the sonicated and non sonicated dyes shows that sonicated one gives better results than that of not sonicated in presence of carriers. Using of ZnO nano powder becomes more effective in case of post treatment than that of simultaneous and the pre-treated fabric.

Table 2 shows that The fastness properties (washing-rubbing-perspiration and light) for the dyed fabrics using the sonicated D-R60 dye All results give very good except for the light fastness which give good results (4 in the blue scale) then increases to very good by increasing the sonication time.

Table 1. Color data for the dyed fabrics

Hours	Colour Data	Disperse red60+carrier Hc								Disperse red60+salicylic acid							
		Conventional dyeing				dyeing after sonication				Conventional dyeing				dyeing after sonication			
		W	B	S	A	W	B	S	A	W	B	S	A	W	B	S	A
35	ΔE	73.29	73.73	74.96	74.47	75.23	73.79	73.77	75.16	73.91	73.42	73.69	73.89	73.30	73.25	71.77	74.20
70	ΔE	72.84	72.94	74.07	73.47	74.62	73.25	73.14	73.92	73.87	72.44	72.66	73.19	74.09	72.79	71.79	74.02
105	ΔE	72.66	72.74	74.05	73.33	74.42	73.11	72.98	74.32	74.59	72.04	73.18	73.26	73.58	72.26	71.48	73.13
140	ΔE	72.41	72.76	73.37	73.09	74.14	72.96	73.03	74.18	73.19	71.82	72.75	73.04	73.18	71.30	72.39	73.59

Hours	Colour Data	Disperse blue56+carrier HC								Disperse blue56+salicylic acid							
		Conventional dyeing				dyeing after sonication				Conventional dyeing				dyeing after sonication			
		W	B	S	A	W	B	S	A	W	B	S	A	W	B	S	A
35	ΔE	60.63	57.83	58.70	60.48	57.68	59.99	59.78	60.83	62.27	59.89	61.73	60.26	61.04	62.10	58.94	61.61
70	ΔE	60.45	57.21	48.41	59.52	57.25	59.80	59.75	60.50	61.44	58.63	60.52	59.84	60.64	61.57	58.87	62.22
105	ΔE	60.52	57.16	88.55	59.67	57.16	59.85	60.63	60.30	60.72	58.60	60.36	60.01	60.73	51.55	58.98	62.06
140	ΔE	60.52	57.03	58.41	59.70	57.30	59.31	60.40	60.61	60.34	58.79	60.57	60.27	60.74	61.58	59.25	62.10

W=blank , B= treated with zinc oxide before dyeing , S= simultaneous treatment during dyeing, A=treated with zinc oxide after dyeing

Table 2. Fastness Properties of dyed polyester with D-R60 at different time of sonication at 130°C.

Sonication time	K/S	croaking		perspiration								washing				light
		dry	wet	acid				alkali				A	C	P	W	
				A	C	P	W	A	C	P	W					
0 min. of son.	8.13	4	4	4	4	4	4	4	4	4	4	4	4	4	4-5	4
15 min. of son.	8.26	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4-5
30 min. of son.	8.78	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
45 min. of son.	9.21	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
60 min. of son.	10.67	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6
75 min. of son.	10.81	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6-7
90 min. of son.	10.97	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6-7

A=alternation, C= staining on cotton, P=staining on polyester, W= staining on wool

Table 3 shows that The fastness properties (washing-rubbing-perspiration and light) for the dyed fabrics using the sonicated D-B56dye All results give very good except for the light fastness which give good results (4–5 in the blue scale) then increases to very good by increasing the sonication time for all samples dyed at 130°C.

Table 4 provides the fastness properties (washing-rubbing-perspiration and light) for the dyed fabrics using the sonicated D-R60 dye. All results give 'very good' except for the light fastness which gave 'less than good' (4 in the blue scale), so we try to increase its light fastness by using Zinc oxide nano powder.

6. Conclusions

The sonication of the dyes results in increased fastness which depends on the length of sonication. The nano ZnO powder used in (pre, simultaneous and post treatment) the fabrics has improved the light fastness in the sonicated dyes better than in the non sonicated ones. Also in case of post treatment, better results were obtained with it for the light fastness which increased the UV protection and decreased the rate of fading. The dyeing of the polyester samples using two disperse dyes has greatly increased in the value of k's and also the fastness properties when dyed at 130°C. This may be due to the distribution of the sonicated dyes properly in the dyeing bath.

Table 3. Fastness Properties of dyed polyester with D-B56 at different time of sonication at 130°C

Sonication time	K/S	croaking		perspiration								washing				light
		dry	wet	acid				alkali				A	C	P	W	
				A	C	P	W	A	C	P	W					
0 min. of son.	10.68	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4-5
15 min. of son.	12.80	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4-5
30 min. of son.	13.28	4	4	4	4	4	4	4	4	4	4	4	4	4	4	5
45 min. of son.	13.78	4	4	4	4	4	4	4	4	4	4	4	4	4	4-5	5
60 min. of son.	13.99	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6
75 min. of son.	14.51	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6-7
90 min. of son.	15.11	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6-7

A=alternation, C= staining on cotton, P=staining on polyester, W= staining on wool

Table 4. Fastness Properties of dyed polyester with D-R60 and D-B56 at 100°C using two carriers:

dyestuff	Dyeing process	K/S	croaking		perspiration								washing				light
			dry	wet	acid				alkali				A	C	P	W	
					A	C	P	W	A	C	P	W					
Disperse red 60 + carrier HC	Conventional dyeing	2.40	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Dyeing after sonication	3.31	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Disperse red 60 +salicylic acid	Conventional dyeing	2.22	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Dyeing after sonication	2.91	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
Disperse Blue 56 + carrier HC	Conventional dyeing	3.99	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	3-4
	Dyeing after sonication	5.98	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
Disperse Blue 56 + salicylic acid	Conventional dyeing	4.40	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
	Dyeing after sonication	4.68	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	3-4

A=alternation, C= staining on cotton, P=staining on polyester, W= staining on wool

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