Article

Novel Monosulfonated Azo Dyes: Design, Synthesis, and Application as Disperse Dyes on Polyester Fabrics

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ABSTRACT: This study synthesizes and characterizes a series of disperse dyes based on azo Schiff bases, compounds **8−10**. Their structures were identified using various analytical techniques, such as FT-IR, ^{1}H/^{13}C NMR, and mass spectrometry. The study's primary objective was to investigate the behavior of disperse dyes **8−10** when used for dyeing polyester fabrics under different conditions, including variations in time, temperature, shade, and pH. The polyester fabric was chosen for this research due to its wide usage and popularity in the textile industry. By examining the effect of temperature and time on the dyeing process, it was observed that increasing the dyeing temperature from low to high (ranging from 90 to 120 °C) and extending the dyeing time from 10 to 30 min resulted in higher K/S values for the polyester samples dyed with disperse dyes. Additionally, dyes **9** and **10** exhibited the most excellent K/S values among the tested dyes. Furthermore, the study found that dye **8** showed the best dyeing performance as the pH of the dye bath increased to 6.

Application as Disperse Dyes on Polyester Fabrics

NOVEL MONOSULFONATED AZO DYES

Synthesis   Design
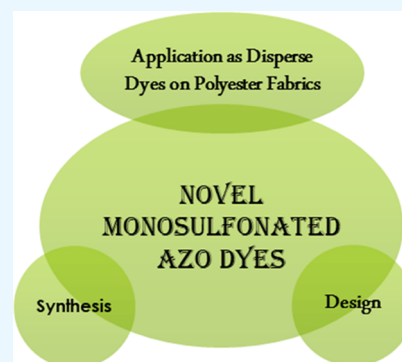
1. INTRODUCTION

Among the diverse applications of industrially synthesized dyes, azo dyes are the most widely used, including dyeing textile fibers, coloring different materials, and advanced applications in organic synthesis.[1−8] Certain compounds contain sulfonated moieties that deserve particular attention because they can be used as disperse dyes and for their wide range of biological and analytical applications.[9]

Extraordinary studies have been conducted on salt forms of monosulfonated azo dyes[10,11] and disulfonated azo dyes.[12] Due to their various elegant characteristics, *e.g.*, color, light fastness, solubility, *etc.*, many sulfonated azo dyes are on the market (Figure 1). However, less attention has been subjected to sulfonated azo dye-free acid forms and their dyeing performance.[11]

In light of the previously mentioned results and supporting an ongoing program of azo dyes,[13−18] this paper presented three new free acid forms of monosulfonated azo Schiff bases starting from cheap materials (salicylaldehyde), having auxochromic groups (−OH and −SO$_3$H) alongside their spectroscopic properties and application in dyeing polyester fabrics.

2. EXPERIMENTAL SECTION

2.1. Synthesis and Spectroscopic Characterization.
2.1.1. Synthesis of 4-((3-Formyl-4-hydroxyphenyl)diazenyl)-benzenesulfonamide (4). A well-stirred solution of 4-amino-benzenesulfonamide **2** (6 mmol, 1.20 g) in concentrated HCl (1.7 mL) and H$_2$O (1.7 mL) was stirred at 0 °C, followed by adding a solution of NaNO$_2$ (6 mmol, 0.41g) in 1 mL of H$_2$O

during 10−15 min; the stirring was continued during the diazotization, and the mixture was kept at 0−5 °C to form 4-sulfamoyl benzene diazonium chloride **3**, which was added dropwise to the solution of **1** (6 mmol, 0.73 g), in the presence of sodium hydroxide (6 mmol, 0.23 g). The solution was continuously swirled for over 4 h at 5 °C. Then, concentrated hydrochloric acid was added slowly and with constant stirring to the cold mixture until the pH of the solution becomes 5.5. The crude product was filtered off and dried to give the corresponding compound **4** as red color powder. Yield: 89% (1.63 g); Mp = 172−174 °C.[19] IR (KBr) ν_{max}/cm^{-1}: 3399 (O−H), 1598 (C=C), 1650 (C=O), 1459 (−N=N−), 1310, 1148 (SO$_2$, SO$_2$NH$_2$); ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.38 (d, 1H), 7.49 (s, 2H), 7.90 (s, 1H), 7.89−8.51 (m, 4H), 7.90 (d, 1H), 10.30 (s, 1H), 12.10 (s, 1H, OH).

2.1.2. General Procedure for the Synthesis of Phenolic Azo Schiff Base Derivatives (8−10). A catalytic amount of AcOH (2 mL) was added to a solution of **4** (6 mmol) and amino sulfonic acid derivatives **5−7** (6 mmol) in EtOH (40 mL). The resulting solution was refluxed and stirred for 6 h. Following the completion of the reaction, as determined by thin-layer chromatography (TLC), the solid precipitate was filtered,
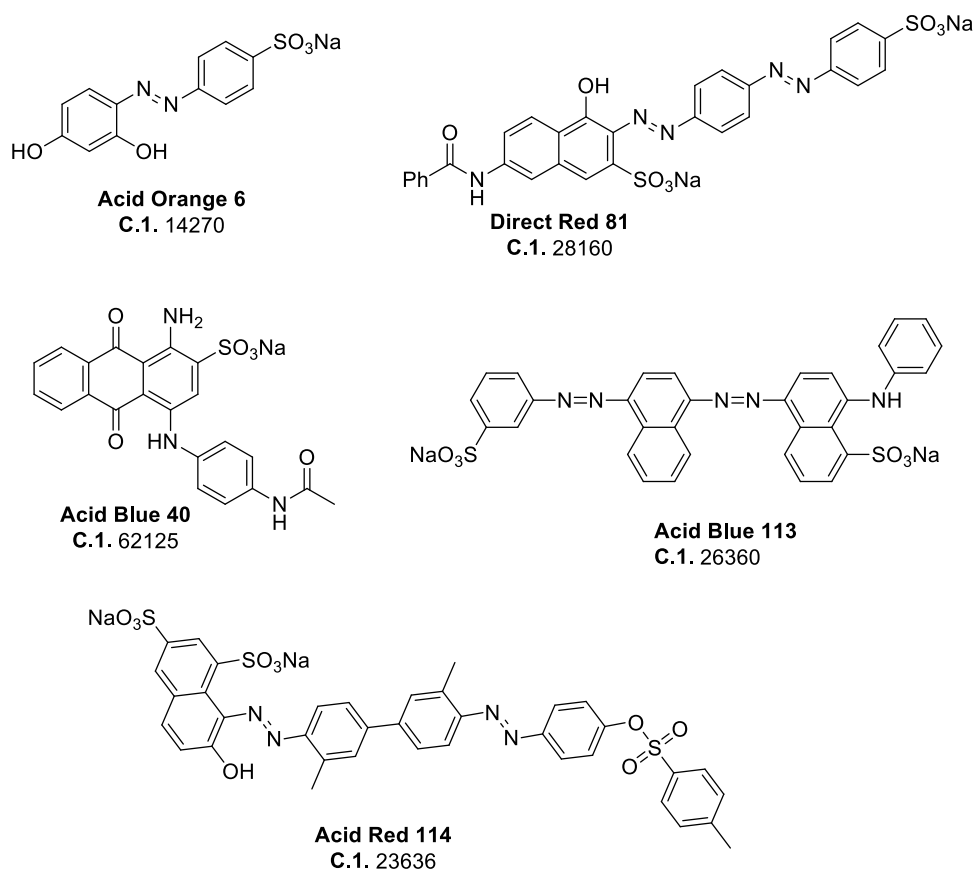
Figure 1. Sulfonated azo dyes on the market.

washed with hot ethanol, dried, and recrystallized from DMF:MeOH to get the desired compounds **8−10**.

2.1.2.1. 2-(((E)-2-Hydroxy-5-((E)-(4-sulfamoylphenyl)-diazenyl)benzylidene)amino)-5-((E)-(4-sulfophenyl)-diazenyl)benzenesulfonic Acid (8). Reddish-brown solid, Yield: 77% (2.97 g); Mp = 285−287 °C; IR (KBr) ν_{max}/cm^{-1}: 3439 (−OH), 3372, 3290 (−NH$_2$), 3060 (Ar−CH), 1624 (−C=N−), 1511 (−N=N), 1333, 1152 (SO$_2$, SO$_2$NH$_2$). ^{1}H NMR (500 MHz, DMSO-d_6), δ 14.52 (s, 2H, 2OH), 9.24 (s, 1H, CH=N), 8.31 (s, 1H, OH), 8.19−7.2 (m, 16H, Ar−H & NH$_2$).^{13}C NMR (126 MHz, DMSO-d_6): δ 168.91, 167.27, 154.30, 152.48, 152.37, 149.79, 146.88, 143.46, 129.19, 127.94, 127.53, 126.89, 124.12, 123.20, 122.89, 117.69, 112.94, 112.50, 101.56. MS m/z (%): 644 (M$^+$, 26.22), 596 (43.35), 484 (95.85), 452 (29.85), 416 (44.16), 388 (100.00), 295 (27.65), 267 (44.77), 218 (58.57), 217 (12.14), 190 (22.57), 99 (29.76), 91 (94.76); Anal. Calcd for C$_{25}$H$_{20}$N$_6$O$_9$S$_3$ (644.05): C, 46.58; H, 3.13; N, 13.04; O, Found: C, 46.45; H, 3.01; N, 12.90%.

2.1.2.2. 4-Hydroxy-6-(((E)-2-hydroxy-5-((E)-(4-sulfamoylphenyl)diazenyl)benzylidene)amino)naphthalene-2-sulfonic Acid (9). Brown solid, Yield: 69% (2.18 g); Mp > 350 °C; IR (KBr) ν_{max}/cm^{-1}: 3420 (OH), 3329, 3259 (−NH$_2$), 3066 (Ar−CH), 1609 (−C=N−), 1506 (N=N), 1390, 1170 (SO$_2$). ^{1}H NMR (500 MHz, DMSO-d_6): δ 14.22 (s, 1H, OH), 10.37 (s. 1H, OH), 9.33 (s, 1H, CH=N,), 8.39 (s, 1H, OH), 8.05−7.15 (m, 12H, Ar−H & SO$_2$−NH$_2$). ^{13}C NMR (126 MHz, DMSO-d_6): δ 165.46, 162.80, 154.07, 153.58, 145.90, 145.22, 144.19, 133.12, 130.47, 129.96, 129.34, 127.89, 127.64, 125.35, 123.20, 121.02, 119.99, 118.93, 115.68, 115.51, 114.80, 107.49, 106.88. MS m/z (%): 526 (M$^+$, 24.00), 492 (67.81), 454 (60.28), 436 (23.20), 426 (76.57), 408 (21.92), 390 (59.73),

386 (100.00), 344 (21.05), 303 (25.40), 275 (16.40), 274 (18.70), 241 (24.64), 237 (31.91), 125 (39.44); Anal. Calcd for C$_{23}$H$_{18}$N$_4$O$_7$S$_2$ (526.06): C, 52.47; H, 3.45; N, 10.64; O, Found: C, 52.34; H, 3.31; N, 10.51%.

2.1.2.3. 3-Hydroxy-4-(((E)-2-hydroxy-5-((E)-(4-sulfamoylphenyl)diazenyl)benzylidene)amino)naphthalene-1-sulfonic Acid (10). Deep brown solid, Yield: 74% (2.34 g); Mp = 245−247 °C; IR (KBr) ν_{max}/cm^{-1}: 3498 (−OH), 3356, 3261 (−NH$_2$), 3063 (Ar−CH), 1618 (−C=N), 1578 (N=N), 1333, 1153 (SO$_2$). ^{1}H NMR (500 MHz, DMSO-d_6): δ 13.29 (s, 1H, OH), 10.34 (s. 1H, OH), 9.16 (s, 1H, CH=N), 8.37 (s, 1H, OH), 8.06−7.18 (m, 14H, Ar−H, NH$_2$). ^{13}C NMR (126 MHz, DMSO-d_6): δ 164.64, 154.00, 153.91, 152.47, 151.42, 146.08, 145.22, 142.80, 130.52, 130.47, 128.32, 127.94, 127.68, 127.60, 124.66, 123.29, 123.24, 122.42, 119.16, 112.95. MS m/z (%): 526 (M+, 48.76), 498 (35.47), 471 (39.62), 379 (55.85), 358 (59.10), 287 (32.19), 217 (42.97), 103 (26.81), 56 (100.00); Anal. Calcd for C$_{23}$H$_{18}$N$_4$O$_7$S$_2$ (526.06): C, 52.47; H, 3.45; N, 10.64; O, Found: C, 52.33; H, 3.30; N, 10.52%.

2.2. Dyeing and Fastness Determination. *2.2.1. Preparation of Dye Dispersion.* Polyester fabrics were dyed in a lab setting using infrared dyeing equipment. The dye above dispersion was created in a dye bath of 20 mL, which also contains 2.0 mL/l of a dispersing agent (Kimilev PS based on castor oil 40 ethoxylates from a chemical company, Egypt). The pH of the dye bath was changed with acetic acid to different pH values (2, 4, and 6).

2.2.2. Dyeing of Polyester Fabrics. The polyester fabrics are immersed in a dye bath, and the temperature of the dye bath is increased gradually at a rate of 2 °C per minute. The temperature reaches high levels such as 90, 100, 110, or 120

Scheme 1. Synthesis of Novel Acid Disperse Dye-Based Schiff Base Derivatives 8−10
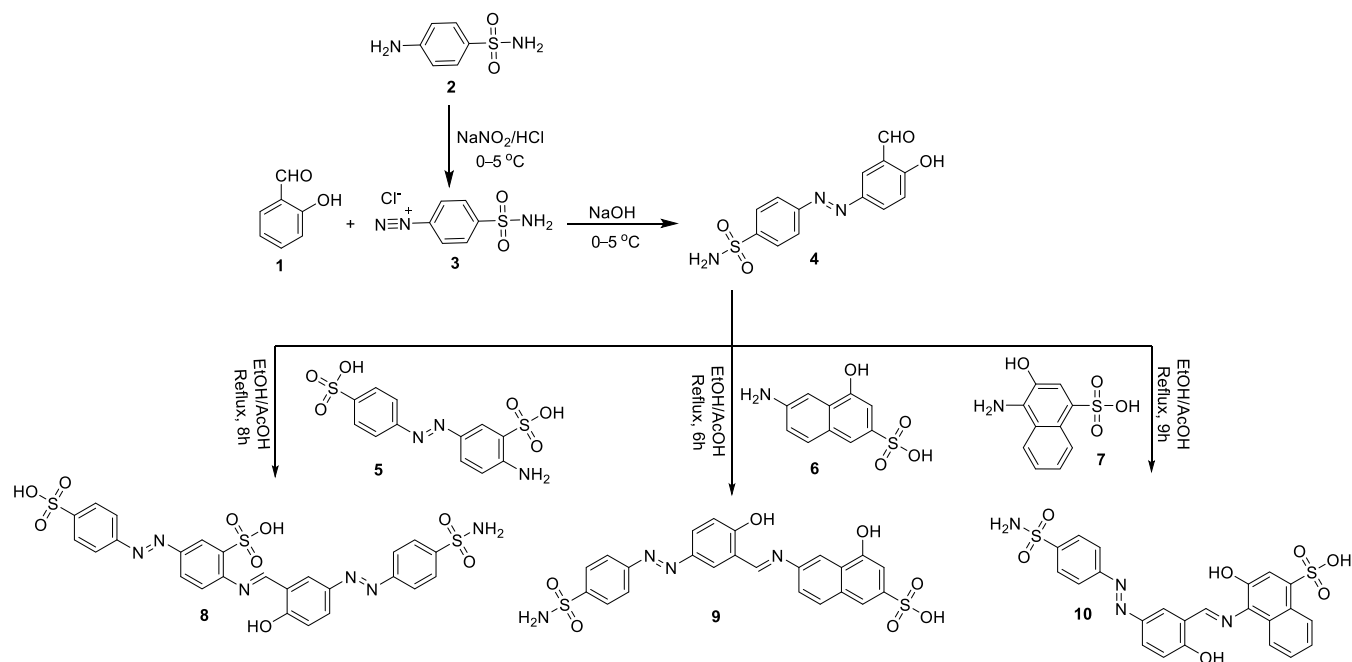
°C. Once the dye bath reaches the desired elevated temperature, it is maintained for various durations, specifically 10, 20, or 30 min.[20] Following the dyeing process, the fabric samples underwent a thorough washing and reduction clearing using 2 g/L sodium hydroxide and 2 g/L sodium dithionite for each liter at 80 °C for 20 min, with a liquor ratio of 1:20. They were then thoroughly cleaned at tap water before being neutralized for 5 min at 60 °C in a separate bath with 1 g/L acetic acid. They were washed in hot, soapy water with 2% nonionic detergent to maximize washing speed. The colored samples were taken from the hot, soapy bath and rinsed in tap water before drying outside.[21,22]

2.2.3. Color Measurements. Using a data color 850 spectrophotometer, CIELAB color coordinates (L^*, a^*, b^*, C^*, and H^o), and color strength values (K/S), colorimetric characteristics of dyed fabric samples were obtained. The Kubelka−Munk equation was used to calculate the color intensity value (K/S) in the visible spectrum (350−700 nm) as follows (eq 1)

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

where K is the absorption coefficient, R is the reflectance of the dyed sample, and S is the scattering coefficient.[23]

The dyed polyester fabric samples' color difference (ΔE) was calculated using the following equation (eq 2)[24]

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

where L indicates the color's lightness and ranges from 100 for white to 0 for black, a measures the color's redness (+ve) or greenness (−ve), b measures the color's yellowness (+ve) and blueness (−ve), and Δ indicates a difference in the values for dyes or colors.[25]

Hue angles (H^o) and the chroma (C^*) hue angles (H^o) were calculated using the following equations (eqs 3 and 4)[25]

$$C^* = (a^2 + b^2)^{1/2} \tag{3}$$

$$H^o = \tan^{-1} b/a \tag{4}$$

The ratio a/b in the CIELAB color space or (L, a, b) coordinates can be used to characterize the colors of dyed materials. When $a/b = 1$ and when $a/b > 1$, the color is "pure" orange and "reddish″ orange, respectively. Additionally, the red contribution increases together with the value of a/b. Similarly, when $a/b = 1$, the color is "yellowish" orange, with the yellow contribution increasing as a/b declines.[26]
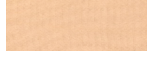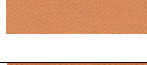
3. RESULTS AND DISCUSSION

3.1. Synthesis of the Precursor Acid Dyes 8−10. The synthetic strategies adopted to obtain precursor acid dyes 8−10 are depicted in Scheme 1. The process starts with the diazotization of 4-aminobenzenesulfonamide 2, followed by the coupling reaction with salicylaldehyde 1. These reactions occur in a basic medium by using 10% sodium hydroxide (NaOH) under stirring. The result of this step is the formation of azosalicyldehyde 4. Diverse acid dye derivatives 8−10 were easily obtained in large quantities from the condensation of 4 and amino sulfonic acid derivatives 5−7 in absolute ethanol with a catalytic amount of acetic acid under refluxing conditions. Different analytical methods, such as FT-IR, ¹H NMR, ¹³C NMR, and mass spectroscopy, were used to confirm the novel acid dyes 8−10 structures (Supporting Information, Figures S1−S12).

3.2. Dyeing Properties of the Dyes on Polyester Fabrics. *3.2.1. Shade Dyeing.* PET textiles were dyed at different shading levels, specifically at concentrations of 1, 2, 3, and 5% for 10 to 30 min using a dispersing agent at dyeing temperatures between 90 and 120 °C. As a result of the dyeing process, the color tones obtained on the PET textiles ranged from yellow to brownish red.

3.2.2. Color Measurement. Color data were collected and analyzed to evaluate the colored textiles. The color hues were determined using the CIELAB color space, defined by the International Commission on Illumination (CIE) in 1976. This color space uses psychometric coordinates L^*, a^*, and b^* to represent various aspects of color, where a^* stands for the red−green axis, b^* stands for the yellow−blue axis, L^* stands for

Table 1. Effect of Different Shades on the Color Measurement ($a*$, $b*$, $L*$, $C*$, E, H^o, and Color Strength) for Dyes 8−10 (Temperature = 120 °C, pH = 4, and Time = 30 min)

Dye	shade%	L*	a*	b*	C*	H°	E	K/S	R%	Photo of dyeing fiber
8	1%	83.42	5.35	43.28	43.6	82.46	94.13	12.8	5.42	
	2%	80.41	9.47	48.65	49.56	78.91	94.45	18.8	3.58	
	3%	76.28	12.8	51.8	53.36	76.11	93.08	20.2	2.77	
	5%	71.53	16.59	52.25	54.82	72.04	90.12	24.4	2.49	
9	1%	79.07	6.98	31.32	32.09	77.44	85.33	6.3	9.57	
	2%	78.45	7.37	35.09	35.86	78.14	86.25	8.4	7.28	
	3%	72.15	10.22	38.84	40.16	75.26	82.57	15.5	4.36	
	5%	66.85	11.63	34.21	36.13	71.24	75.99	16.6	3.78	
10	1%	76.26	12.37	38.69	40.62	72.26	86.35	7.4	8.34	
	2%	68.12	15.91	40.7	43.7	68.64	80.93	12	5.27	
	3%	58.97	16.83	39.96	43.36	67.16	73.19	17	3.30	
	5%	53.01	23.04	44.08	49.74	62.40	72.69	21.6	2.58	

lightness, $C*$ stands for chroma, and H^o stands for the hue angle from 0 to 360°. Positive $L*$ values showed that the disperse dyes **8−10** were lighter, and a lack of both (shade degree, duration, and temperature) progressively raised the values of the $L*$ coordinate (Table 1). The favorable $C*$ estimations also substantiate this fact. It is usual to practice using K/S values at the wavelength of minimal reflectance or at the wavelength of maximum absorbance to describe dye accumulation behavior on textile surfaces.

3.2.3. Effect of Dye Shade on Color Strength (K/S) and Reflectance (R). The results of measuring color strength (K/S values) and reflectance values for dyed polyester fabrics with different shades of disperse dyes **8−10** (Figure 2 and Table 1) are as follows:

Increasing the dye shade (1, 2, 3, and 5% o.w.f) on dyeing polyester resulted in better K/S values, indicating higher color strength. However, the reflectance values decreased as the dye shade increased for all three dyes. The difference in the K/S value between shades 3 and 5% for dye **9** was very small (1.1 in value). This could be because the pores of the polyester fabrics became saturated with dye particles, limiting further absorption.

Therefore, the optimal shade for dye **9** was 5% based on the high K/S value achieved.

The highest K/S values obtained for disperse dye **8** were 12.8, 18.8, 20.2, and 24.4 for shades 1, 2, 3, and 5% o.w.f, respectively. These values were achieved at pH 4 and a dyeing time of 30 min. The reflectance values of the samples dyed with shade 1% in water were greater than those dyed with shades 2, 3, and 5% o.w.f for all three dyes. This indicates that the increased concentration of dye particles inside the polyester resulted in higher absorption and reduced reflectivity.

3.2.4. Effect of Dyeing Time on Color Strength (K/S) and Reflectance (R). According to Table 2:

- **Dye 8**: The dyeing process took longer than 10 min. Each time the process took longer, the color strength increased by 0.2. The best time for dyeing with it was 20 min, resulting in a color strength value of 19.4. Increasing the time to 30 min caused the color strength to decrease by 3.2 (Supporting Information, Figure S13). This decrease is likely due to an unfavorable impact causing the dye to leak out of the fiber. As time passes, the value of reflection increases and becomes almost intangible (Supporting Information, Figure S14).
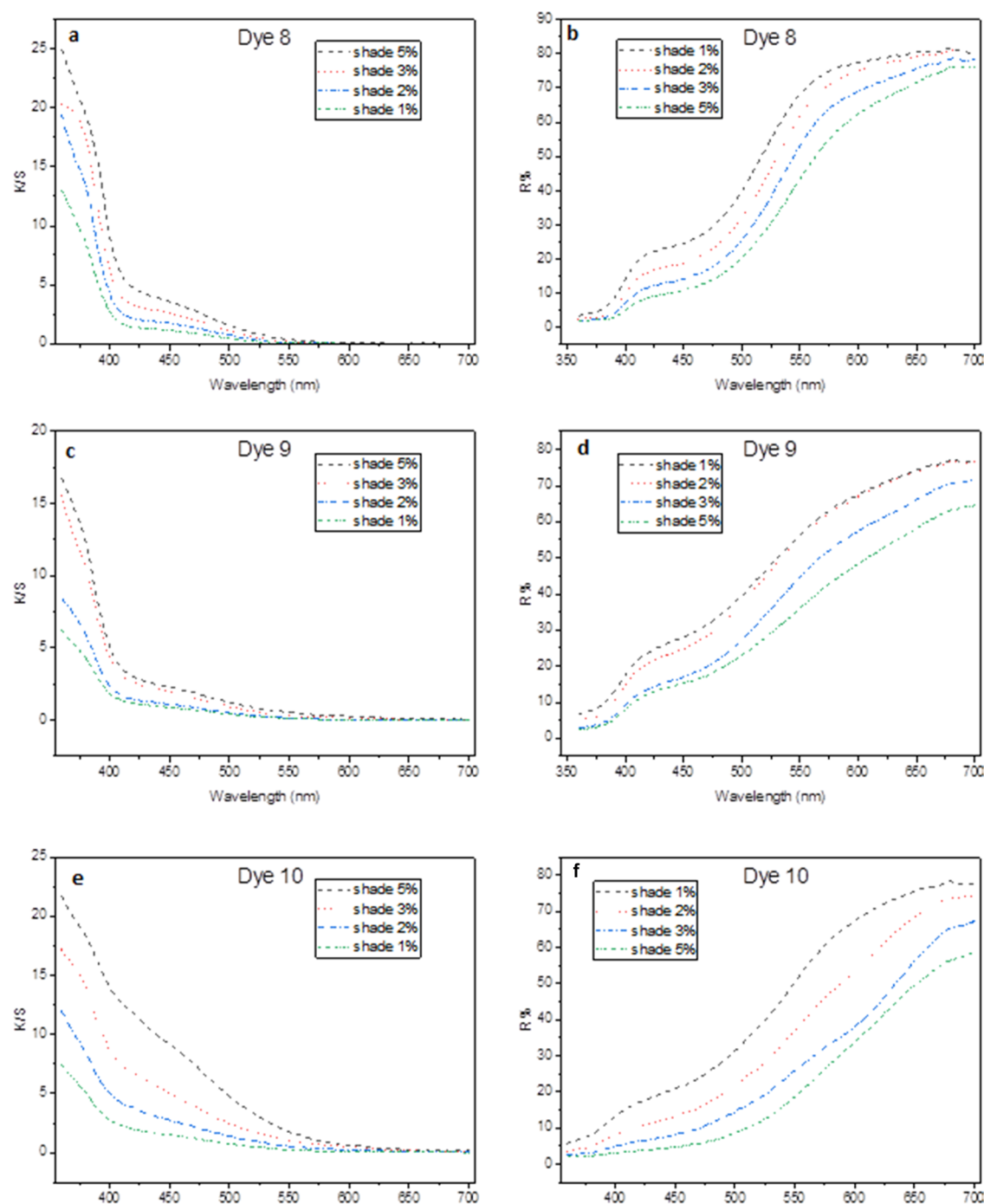
Figure 2. (a−f) Effect of dyeing shade (1, 2, 3, and 5%) on color strength and reflectance (where dyeing pH is 4, dyeing temperature is 120 °C, and dyeing time is 30 min) for dyes **8−10**.

- **Dye 9:** The color strength of dye **9** decreased when the duration was extended from 10 to 20 min (Supporting Information, Figure S15). The ideal period for dyeing with dye **9** was 30 min. The highest reflection value was observed at 20 min (Supporting Information, Figure S16). If the time was prolonged to 30 min, the color strength rose to 11.7.

- **Dye 10:** It exhibited a rise in color intensity from 12.9 to 16.4, increasing from 10 to 30 min (Supporting Information, Figure S17). Equilibrium between the dye molecules in the dyeing solution and those inside the fiber occurred at 30 min. The optimal time for coloring with

dye **10** is within this brief time frame. The value of reflection did not significantly change between a time difference of 10 to 30 min (Supporting Information, Figure S18).

3.2.5. Effect of Dyeing Temperature on Color Strength (K/S) and Reflectance (R). The dyeing process with a disperse dye on polyester samples was carried out at different temperatures ranging from 90 to 120 °C. The K/S values, which indicate color strength, increased as the temperature increased from low to high (90, 100, 110, and 120 °C). This suggests that higher temperatures promoted better dye absorption by the polyester samples, leading to higher color strength (Supporting

Table 2. Effect of Dyeing Time on the Color Measurement (*a**, *b**, *L**, *C**, *E*, *H*º, *R%*, and Color Strength) for Dyes 8−10 (Temperature = 120 °C, pH = 4, and 3% Shade)
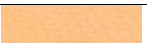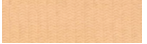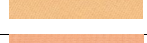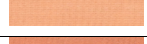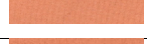
Dye	Time	L*	a*	b*	c*	Hº	E	K/S	R%	Photo of dyeing fabrics
8	10 min	79.04	9.35	48.11	49.01	79	93.00	19.2	3.5	
	20 min	75.56	10.87	47.15	48.39	77.02	89.72	19.4	3.5	
	30 min	61.56	11.91	33.64	35.68	70.5	71.15	16.2	4	
9	10 min	76.9	8.07	37.69	38.55	77.91	86.01	11.2	4.3	
	20 min	77.28	8.14	37.13	38.02	77.63	86.12	9.2	5	
	30 min	76.51	8.26	37.74	38.63	77.65	85.71	11.7	4.4	
10	10 min	59.66	25.36	38.62	46.21	56.71	75.37	12.9	3	
	20 min	58.64	27.05	39.29	47.7	55.45	75.59	14.6	2.9	
	30 min	56.15	24.78	38.04	45.39	56.92	72.20	16.4	2.9	

Table 3. Effect of Dyeing Temperatures on the Color Measurement (*a**, *b**, *L**, *C**, *E*, *H*º, *R%*, and Color Strength) for Dyes 8−10 (Time = 30 min, pH = 4, and 4% Shade)
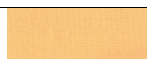
Dye	Temperature (°C)	L*	a*	b*	C*	Hº	E	K/S	*R %*	Photo of dyeing fabrics
8	90	83.5	4.9	43.9	44.3	80.6	94.5	7.2	7	
	100	77.1	14.6	50.4	52.5	73.8	93.3	18.5	3	
	110	75.4	17.3	50.5	53.4	71.2	92.4	21.4	2.9	
	120	72.2	21.1	45.6	50.3	65.2	87.9	17.5	3.1	
9	90	81.9	5.2	40.1	40.4	82.6	91.3	6.9	7	
	100	80.4	6.3	41.4	41.9	81.4	90.6	11.2	4	
	110	78.5	7.6	43	43.7	79.9	89.8	14.4	3.1	
	120	78.5	7.9	42.1	42.8	79.4	89.4	14.6	3	
10	90	72.2	16.0	35.1	38.6	65.5	81.8	7.4	6	
	100	62.3	22.2	33.0	39.8	56.1	73.9	10.7	5	
	110	60.7	24.6	34.2	42.2	54.3	73.9	9.9	5.2	
	120	57.8	17.5	34.4	38.6	63.1	69.5	13.5	4	

Information, Figures S19−S21). On the other hand, the reflectance (*R%*) of the dyed polyester samples decreased as the temperature increased. Higher temperatures resulted in lower light reflectance from the dyed samples (Figures S22−S24). Lower reflectance can be associated with increased dye absorption and reduced light scattering.

Specifically, for dye **8**, the best color strength value was observed at 110 °C. However, when the temperature was raised to 120 °C, the color strength value started to decline. At 100 °C, the color strength value was 18.4; at 120 °C, it dropped by 3.9 units. This indicates that temperatures above 110 °C diminished the dye's effectiveness, leading to lower color strength. The data also suggest that temperature fluctuation between 110 and 120 °C may fluctuate whenever the dye concentration in the solution exceeds that of the dye in the dye bath.

Table 4. Effect of Different Dyeing pH on the Color Measurement (*a**, *b**, *L**, *c**, *E*, *H°*, *R%*, and Color Strength) for Dyes 8−10 (Time = 30 min, Temperature = 120 °C, and 4% Shade)

Dye	pH	L*	a*	b*	C*	H°	E	K/S	R %	Photo of dyeing fabrics
6	PH 2	79.07	12.28	50.91	52.37	76.44	94.84	**25.6**	2.71	
	PH 4	80.94	10.96	50.93	52.10	77.86	96.25	22.0	3.03	
	PH 6	81.43	10.02	49.70	50.70	78.60	95.92	19.9	3.32	
7	PH 2	71.57	10.11	33.13	34.64	73.04	79.51	12.5	5.58	
	PH 4	72.87	10.14	34.01	35.49	73.40	81.05	12.4	5.32	
	PH 6	73.26	10.36	34.52	46.04	73.30	81.64	**12.8**	5.23	
8	PH 2	59.88	15.57	41.24	44.08	69.31	74.35	19.2	3.27	
	PH 4	60.57	12.77	35.19	37.44	70.06	71.20	15.8	3.99	
	PH 6	57.01	15.21	38.23	41.14	68.31	70.30	**19.5**	3.04	

Further details and observations regarding this fluctuation can be found in Figure S19 and Table 3. In contrast to the color strength, the optimum reflectance value was observed at 90 °C (Figure S22 and Table 3). This means the dyed samples exhibited the highest light reflectance at a lower temperature of 90 °C.

Concerning dye **9** exhibited optimal color strength at a temperature of 120 °C, with a small surplus in the value at 110 °C (Supporting Information, Figure S20 and Table 3). A slight increase in temperature led to a change in color strength. At 100 °C, the color strength value was 11.2; at 90 °C, it reduced to 6.9. This suggests that the most effective temperature for dispersing this dye in water in the presence of a dispersing agent is the limiting temperature of 120 °C, where equilibrium occurs. However, as expected, the highest value of reflection was observed at a temperature of approximately 90 °C. This means the dyed samples exhibited higher light reflectance at lower temperatures, around 90 °C (Supporting Information, Figure S23 and Table 3).

Dye **10** displayed distinct behavior compared to the other dyes **8** and **9** in the study. The color strength increased with temperature, similar to the trend observed for the other dyes. At 120 °C, the color strength value was 13.5 (Supporting Information, Figure S21 and Table 3). In contrast, the reflection value decreased with an increasing temperature. Higher temperatures resulted in lower reflection values, with a reflection value of 4 at 120 °C (Supporting Information, Figure S24 and Table 3).

3.2.6. Effect of Dyeing pH on Color Strength (K/S) and Reflectance (R). According to supplementary Figures S25−S27 for dyes **8−10**, respectively, dyeing got roughly the same *K/S* throughout the range of acidic pH, indicating that the change in dyeing pH has no discernible impact on the dyeing behavior for the dyes tested. The reflectance values of dyed polyester fabrics were measured with different dyeing bath pH of dispersed dyes **8−10**, and the results are shown in Figures S28−S30 and Table

4. The obtained findings demonstrated that raising *K/S* values and lowering reflectance values in the case of dye **8** was caused by reducing the dyeing pH in dyeing polyester (Supporting Information, Figures S25 and S28). Naturally, decreasing the dyeing pH to 2 produced higher *K/S* values; as a result, dyeing pH 2 is considered the ideal pH. In terms of the value, it was discovered that pH 2 had the strongest color strength for dye **8**, followed by pH 4 and pH 6 (25.6, 22, and 19.9, respectively). Contrarily, in terms of reflectance, compared to dye **9**, there is very little change in color intensity *K/S*. According to the value (12.8), its optimum value is at pH 6, followed by pH 2 and 4, as seen in Supporting Information, Figure S26. Contrarily, it is clear that there is not much of a difference when it comes to reflectance, also in Supporting Information, Figure S29. The best color strength for dye **10** is at pH 6, followed by pH 2 and 4 (19.5, 19.2, and 15.8, respectively), as shown in supplementary, Figure S27. Contrarily, the difference in reflectance is fairly straightforward (Supporting Information, Figure S30).

Based on Figures S25−S27, it appears that for dyes **8−10**, the dyeing pH had little to no discernible impact on the dyeing behavior, as indicated by the relatively consistent *K/S* values across the range of acidic pH. This suggests that variations in the dyeing pH did not significantly affect the dye absorption or color strength for the tested dyes.

4. CONCLUSIONS

In the investigation, novel dispersed dyes based on azo Schiff bases **8−10** were synthesized and then applied to polyester fabrics using low-, medium-, and high-temperature dyeing techniques. The results obtained regarding color levelness and shade depth were satisfactory, producing a range of colors, including beige, orange, and reddish brown with various shades. For disperse dye **8**, the optimal dyeing parameters for achieving high color strength were 30 min, a pH of 2, and a temperature of 120 °C. These specific conditions resulted in the desired color intensity for dye **8**. Similarly, for dispersion dyes **9** and **10**, the

best dyeing conditions for obtaining satisfactory color results were 30 min, a pH of 6, and a temperature of 120 °C. These parameters yielded the desired color levelness and shade depth for dyes **9** and **10**. It is important to note that the information provided highlights the optimal dyeing parameters for achieving satisfactory color results for the mentioned dispersed dyes on polyester fabrics. Other factors such as dye concentration, fabric pretreatment, and specific dyeing techniques may also play a role in achieving desired color outcomes.

■ ASSOCIATED CONTENT

⑤ Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.3c05905.

> FT-IR spectra, ^{1}H NMR spectra, ^{13}C NMR spectra, mass spectra of compounds **8**, **9**, and **10**; effects of different times on the color strength and reflectance for dyes **8**, **9**, and **10**; effects of different temperatures on the color strength and reflectance for dyes **8**, **9**, and **10**; and effects of different pH on the color strength and reflectance for dyes **8**, **9**, and **10** (PDF)

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Notes

The authors declare no competing financial interest.

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■ DEDICATION

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