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Synthesis and evaluation of changes induced by solvent and substituent in electronic absorption spectra of some azo disperse dyes

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ABSTRACT

Five azo disperse dyes were prepared by diazotizing 4'-aminoacetophenone and *p*-anisidine and coupling with varies *N*-alkylated aromatic amines. Characterization of the dyes was carried out by using UV–vis, FTIR and ¹H NMR spectroscopic techniques. The electronic absorption spectra of dyes are determined at room temperature in fifteen solvents with different polarities. The solvent dependent maximum absorption band shifts, were investigated using dielectric constant (ε), refractive index (*n*) and Kamlet–Taft polarity parameters (hydrogen bond donating ability (α), hydrogen bond accepting ability (β) and dipolarity/polarizability polarity scale (π^*)). Acceptable agreement was found between the maximum absorption band of dyes and solvent polarity parameters especially with π^* . The effect of substituents of coupler and/or diazo component on the color of dyes was investigated. The effects of acid and base on the visible absorption maxima of the dyes are also reported.

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1. Introduction

Having versatile applications in miscellaneous areas such as electrophotography, laser printing, reversible optical storage, nonlinear optical (NLO) devices and liquid crystalline displays (LCDs) as well as biological-medicinal studies, azo compounds have been of utmost importance as colorants [1–8]. The versatile usage of azo compounds especially azobenzene dyes can be attributed to the possession of several advantages such as intensive color, ease to prepare from cheap, readily available starting materials, desirable fastness properties and unique properties arising from reversible conformational changes accompanying light induced cis-trans isomerization [9–11].

Disperse dyes are one of the most important colorants that are poorly soluble in water and are suitable for many applications from traditional dyeing to high and modern technologies, which azo dyes constitute the largest group of disperse dyes [12–15].

Solvatochromism studies are other fields that numerous papers in relation with these disperse dyes have been published. It has long been known that UV–vis/IR absorption spectra of azo dyes influenced by the surrounding medium and solvents can bring about a change in the position, intensity, and shape of absorption bands. However the absorption properties of these dyes are affected by solvents and showed bathochromism (positive solvatochromism) or hypsochromism (negative solvatochromism) upon increasing the polarity of the solvent. Moreover, absorption bands position depends on different types of interactions between solvent and solute. According to complexity of the interactions between solute and solvent, the experimental and theoretical investigations of the structure of liquids are one of the most difficult tasks for both physics and chemistry [16–21].

In the present work, we have synthesized four disperse dyes with donor–acceptor substituents and one dye including donor–donor moieties in their conjugated systems. The solvatochromic behavior of synthesized dyes were investigated in solutions using the empirical solvent polarity parameters, including solvent dipolarity/polarizability (π^*), hydrogen bond donation ability (α) as solvent acidity, and hydrogen bond acceptance ability (β) as solvent basicity, and dielectric constant (ε) and refractive index (n) [22–24]. Also, the effects of substituent and pH on their absorption properties of dyes were evaluated. The chemical structures of the synthesized disperse dyes are presented in Scheme 1.

2. Experimental

2.1. Materials and apparatus

The chemicals used in this study were obtained from Merck and Aldrich Chemical Companies and were used without further purification. All melting points were determined on an electrothermal melting point apparatus and are uncorrected. Elemental analyses were performed using a Heracus CHN-O-Rapid analyzer. Infrared spectra were recorded on a Shimadzu 8400 FT-IR spectrophotometer. The proton nuclear magnetic resonance (¹H NMR) spectra were

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Scheme 1. The chemical structure of synthesized azo dyes.

obtained on a FT-NMR (500 MHz) Brucker apparatus spectrometer, and the chemical shifts are expressed in δ ppm using TMS as an internal standard. The visible spectra were measured using a Pharmacia Biotech Spectrophotometer.

2.2. Synthesis and characterization

The mixtures of aniline derivatives (3 mmol), water (5 mL) and concentrated sulfuric acid (7.5–9 mmol) were heated with stirring until a clear solution was obtained. These solutions were cooled to 0-4°C and a solution of sodium nitrite (0.207 g, 3 mmol) in water was added drop wise while the temperature was maintained below 4°C. The resulting mixtures were stirred for 45–60 min in an ice bath. After diazotization was complete, the azo liquor was slowly added to a stirred solution of aromatic amines (3 mmol) dissolved in 10 mL of buffer solution of acetic acid-acetate sodium (pH=5) and 2-5 mL ethanol. The resulting mixture was stirred at 0-4 °C for 2 h in an ice bath. After this stage, the pH of the reaction mixture was maintained at 6.5–7.5 by addition of acetate sodium solution. Then the resulting dyes were filtered and washed with cold water. The crude dyes were purified using recrystallization method with EtOH and DMF/H₂O. The physical and spectral data of the purified dyes are as follows.

2.2.1. 2-(4-(4-Acetylphenylazo)phenylamino)ethanol (dye 1)

Orange dye was recrystallized from EtOH and obtained in a yield of 90%.

Mp: 126–128 °C. UV–vis (EtOH), λ_{max} (nm) 368. IR (KBr): ν 3310–3450 (br, OH and NH, overlapped), 1495 (N=N) cm⁻¹. ¹H NMR (DMSO-d₆) δ 2.4 (1H, t, *J* = 5.3 Hz, OH), 2.47 (3H, s, CH₃), 3.8 (2H, q, *J* = 5.4 Hz, OCH₂), 4.3 (2H, t, *J* = 5.3 Hz, NCH₂), 4.6 (1H, br, s, NH), 6.9 (2H, d, *J* = 8.8 Hz, Aro.–H), 7.94 (2H, d, *J* = 8.9 Hz, Aro.–H), 7.96 (2H, d, *J* = 8.9 Hz, Aro.–H), 8.1 (2H, d, *J* = 8.9 Hz, Aro.–H). Elemental analysis calcd for C₁₆H₁₇N₃O₂: C 67.64%, H 6.22%, N 15.23%. Found: C 67.83%, H 6.05%, N 14.83%.

2.2.2. 2,2'-(4-(4-Acetylphenylazo)phenylamino)diethanol (dye 2)

Orange-red dye with metallic luster was recrystallized from EtOH and obtained in a yield of 94%.

Mp: 156–158 °C. UV–vis (EtOH), λ_{max} (nm) 449. IR (KBr): ν 3280 (br, OH), 1660 (–C=O), 1510 (N=N) cm⁻¹. ¹H NMR (DMSO-d₆) δ 2.51 (s, 3H, CH₃), 3.56 (t, 4H, N–CH₂, *J* = 4.99 Hz), 3.73 (q, 4H, O–CH₂, *J* = 5.19 Hz), 4.59 (t, 2H, OH, *J* = 5.34 Hz), 6.70 (m, 2H, Aro.–H), 7.74 (m, 4H, Aro.–H), 7.92 (m, 2H, Aro.–H). Elemental analysis calcd for C₁₈H₂₁N₃O₃: C 65.82%, H 6.75%, N 14.8%. Found: C 66.04%, H 6.47%, N 12.84%.

2.2.3. 1-(4-(4-(Diethylamino)phenylazo)phenyl)ethanone (dye 3) Red dye with metallic luster was recrystallized from DMF/H₂O and obtained in a vield of 85%.

Mp: 159–161 °C. UV–vis (EtOH), λ_{max} (nm) 461. IR (KBr): ν 2880–2980 (Aro.–H), 1502(N=N). ¹H NMR (CDCl₃) 1.26 (t, 6H, CH₃, *J*=7.1 Hz), 2.5 (s, 3H, CH₃), 3.48 (q, 4H, –N–CH₂, *J*=7.11 Hz), 6.72 (d, 2H, Aro.–H, *J*=9.26 Hz), 7.9 (d, 2H, Aro.–H, *J*=9.26 Hz), 7.96–8.0 (m, 4H, Aro.–H). Elemental analysis calcd for C₁₈H₂₁N₃O: C 73.08%, H 7.09%, N 14.41%. Found: C 73.19%, H 7.17%, N 14.23%.

2.2.4. 1-(4-(4-(N-benzyl-N-ethylamino)phenylazo)phenyl) ethanone (dye 4)

Red dye with metallic luster was recrystallized from DMF/H_2O and obtained in a yield of 85%.

Mp: 116–118 °C. UV–vis (EtOH), λ_{max} (nm) 447. IR (KBr): ν 2875–2990 (Aro.– H), 1510 (N=N). ¹H NMR (CDCl₃) 1.3 (t, 3H, CH₃, *J*=7.12 Hz), 3.6 (q, 2H, –N–CH₂, *J*=7.12 Hz), 4.2 (s, 2H, CH₂), 6.7 (d, 2H, *J*=9.1 Hz, Aro.–H), 7.2 (d, 2H, *J*=9.1 Hz, Aro.–H), 7.3 (d, 2H, *J*=8.8 Hz, Aro.–H), 7.7 (d, 2H, *J*=8.8 Hz, Aro.–H). Elemental analysis calcd for C₂₃H₂₃N₃O: C 77.32%, H 6.75%, N 11.82%. Found: C 77.28%, H 6.49%, N 11.76%.

2.2.5. 2,2'-(4-(4-Methoxyphenylazo)phenylamino)diethanol (dye 5)

Golden dye with metallic luster was recrystallized from EtOH and obtained in a yield of 94%.

Mp: 135–137 °C. UV–vis (EtOH), λ_{max} (nm) 409. IR (KBr): ν 3445 (OH), 2960 (Aro.–H), 1504 (N=N) cm⁻¹. ¹H NMR (CDCl₃) δ 3.23 (br, 2H, OH), 3.72 (t, 4H, N–CH₂, *J*=4.74 Hz), 3.91 (s, 3H, CH₃), 3.96 (t, 4H, O–CH₂, *J*=4.88 Hz), 6.79 (m, 2H, Aro.–H), 7.03 (m, 2H, Aro.–H), 7.87 (m, 4H, Aro.–H). Elemental analysis calcd for C₁₇H₂₁N₃O₃: C 64.32%, H 7.1%, N 13.63%. Found: C 64.74%, H 6.71%, N 13.32%.

3. Results and discussion

3.1. The UV–visible spectra and solvatochromic studies of synthesized dyes

In order to study of solvent effects on spectral features of the dyes, we recorded their absorption spectra in various solvents with different polarity at a concentration of $10^{-5}-10^{-6}$ M in the range of 300–700 nm (Table 1), in which the solvents are arranged in the order of increasing polarity. Also, refractive index (*n*), dielectric constant (ε) and the solvatochromic parameters (π^* , α , and β) were taken from the literature [22–24]. As shown in Table 1, the electronic absorption spectra of all studied compounds in different

Table 1	l
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Experimental electronic absorption maxima for investigated dyes and solvent parameters [22-	-24].	•
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Solvents	π^*	α	β	ε	п	λ_{\max} (nm)				
				293 K	293 K	Dye 1	Dye 2	Dye 3	Dye 4	Dye 5
n-Hexane	-0.081	0.00	0.00	1.89	1.3748	359	424	430	426	392
Cyclohexane	0.00	0.00	0.00	2.02	1.4226	362	430	433	429	398
Diethyl ether	0.27	0.00	0.47	4.27	1.3526	364	437	449	440	407
Tetrachloromethane	0.28	0.00	0.00	2.24	1.4601	356	431	455	443	401
Ethyl acetate	0.54	0.00	0.45	6.08	1.3723	366	444	457	445	411
Ethanol	0.54	0.83	0.77	25.33	1.3611	368	449	461	445	409
Dioxane	0.55	0.00	0.37	2.22	1.4224	366	443	447	442	412
Chloroform	0.58	0.44	0.00	4.81	1.4459	359	434	457	443	404
Tetrahydrofuran	0.58	0.00	0.55	7.47	1.4070	361	448	459	445	413
Methanol	0.60	0.93	0.62	33.10	1.3288	365	441	457	439	407
Acetone	0.71	0.08	0.48	21.01	1.3588	367	448	458	445	413
Acetonitrile	0.75	0.19	0.31	37.50	1.3442	368	449	458	446	413
Dichloromethane	0.82	0.30	0.00	9.08	1.4242	365	435	459	448	407
DMF	0.87	0.00	0.69	38.25	1.4305	403	466	477	460	422
DMSO	1.00	0.00	0.76	47.24	1.4770	412	473	485	465	425

solvents exhibit maximum absorption band (λ_{max}) in the range of 361–485 nm which can be attributed to $n \rightarrow \pi^*$ and/or $\pi - \pi^*$ electronic transitions of azo chromophores [25]. The dyes displayed a single main absorption peak without a shoulder in all solvents. The λ_{max} of all compounds were found to show a strong solvent dependency, denoting bathochromic effect (positive solvatochromism) in more polar solvents, except for chlorinated solvents. For chlorinated solvents the deviation may be due to unique interactions between the polychlorinated solvents and the solute molecules. Such interactions can be accounted using another specific solvent polarity scale named as delta (δ) that generally is used in multi-parameter correlation. The spectral shift is mainly due to solute–solvent interactions that give rise to a better stabilization of the π^* orbital as compared to the π orbital in polar solvents (Fig. 1).

It is well known that the position and shape of absorption spectra changes with solvent nature. The changes induced by solvents were evaluated using solvatochromic parameters such as $(n \text{ and } \varepsilon)$ and $(\pi^*, \alpha \text{ and } \beta)$. In order to the more studies of solvatochromic properties of synthesized dyes, we were obtained the correlation coefficient values between maximum wavelength (λ_{max}) of dyes and solvent parameters (Table 2). The investigations of data in Table 2 indicate that there was regular change between the λ_{max} values of the compounds and π^* , ε and β values of studied solvents. On the other hand, all studied dyes, exhibit a bathochromic shift (red shift) as a result of an increase in the solvent polarity scale (π^*), hydrogen bond accepting ability (β). Therefore, it can be concluded that the most important contributions to the solvatochromism shift arise from π^* and β terms of solvent parameters. This effect was attributed to the interaction of solvents such as DMF and DMSO with non-bonding electron pair on nitrogen atom in coupler



Fig. 1. Electronic absorption spectra of dye 4 in different solvents.

Table 2

Correlation coefficient (R) of dyes in studied solvents.

Solvent parameters	π^*		α	β	ε	п
Dye no.	R	R ₁ ^a	R	R	R	R
Dye 1	0.40	0.47	0.027	0.37	0.58	0.13
Dye 2	0.63	0.85	0.002	0.66	0.67	0.03
Dye 3	0.80	0.87	0.011	0.42	0.56	0.09
Dye 4	0.80	0.85	0.004	0.35	0.47	0.16
Dye 5	0.77	0.91	0.008	0.58	0.51	0.03

^a Correlation coefficient (R_1) obtained without chlorinated solvents.

component of synthesized dyes, which increases the electron density at the nitrogen atom. Also, no linear correlation was obtained when solvent parameters n and α were applied. Thus, the correlation coefficient values obtained for synthesized disperse dyes show that a major contribution to the solvatochromism is due to the solvent dipolarity/polarizability (Fig. 2), hydrogen bond accepting ability (Fig. 3) and dielectric constant (Fig. 4).

In the next part of the investigation, the pH values of the synthesized dyes were also determined spectrophotometrically in 80% (v/v) ethanol–water mixtures (aqueous hydrochloric acid and sodium hydroxide) at $25 \pm 2 \,^{\circ}$ C. A digital pH meter Genway model 3505 was employed for determination of pH. The instrument was accurate to ± 0.01 pH unit (Table 3). It was calibrated using two standard Genway buffer solutions at pH=4.01 and 7.00 and used relation of Van Uitert and Haas [26]. Comparison of absorption maxima of the dyes revealed a red shift in λ_{max} of the main visible band in acidic solutions. This shift can be explained on the basis that the dyes especially dye 5 with donor–donor substituent groups exist in



Fig. 2. Variation of λ_{max} (nm) of dye 4 as a function of π^* .

Table 3
Electronic absorption spectral data of compounds 1-5 in acidic and basic solution.

Dye no.	$\lambda_{\max}(nm)$									
	pH = 1	pH=3	pH=5	pH=6	pH = 7	pH = 8	pH=9	pH=11	pH=13	
1	454	411	386	372	369	369	372	373	377	
2	504	492	475	466	448	449	449	451	454	
3	511	503	488	472	461	460	462	463	468	
4	521	518	471	451	446	446	447	447	449	
5	553	408, 560	408, 555	408, 562	400	409	410	410	416	



Scheme 2. Acid-base equilibrium of dye 5.

2.00

1.80

1.60

Abs



pH=3 1.40 pH=5 1.20 1.00 pH=6 0.80 pH=7 0.60 pH=8 0.40 pH=9 0.20 pH=13 0.00 325 375 425 475 525 575 625 675 725 λ (nm)

Fig. 3. Variation of λ_{max} (nm) of dye 4 as a function of β .

acid–base equilibrium (Scheme 2). Considering the λ_{max} in Table 3, these dyes in acidic solutions shifts to higher wavelengths, which are probably related to the prominence of hydrazone forms in acidic medium, while in neutral and basic solutions the dyes exists in azo forms. The UV–vis spectroscopic study on dye 5 clearly indicates that the present dye exists in acid–base equilibrium forms in solution with pH = 3, 5 and 6 as shown in Fig. 5.

It was also observed that the absorption curves of the dyes were not significantly sensitive to base solutions.

3.2. Substituent effects

In our previous work, we have demonstrated that absorption maxima of azobenzene dyes are strongly influenced with substituents in para-position in contrast to ortho- and metapositions [27]. As expected, the donor-acceptor polarization



Fig. 4. Variation of λ_{max} (nm) of dye 4 as a function of ε .

Fig. 5. Absorption spectra of dye 5 in acidic and basic solutions.

of synthesized dyes 1–4 are increased by introducing an electron-accepting acyl group into the para-position of benzenoid system, caused bathochromic shifts relative to similar dye 5 including an electron-donating methoxy group. Therefore dyes 1–4 showed bathochromic shift due to extended resonance system in comparison with dye 5 in all studied solvents as shown in Table 1. In addition, the introduction of electron donating alkyl groups in the coupler components are affected in absorption band shifts and resulted in bathochromic shifts in all solvents (e.g. for dye 1, λ_{max} = 368 nm relative to dye 2, λ_{max} = 449 nm or dye 3, λ_{max} = 461 nm in ethanol).

4. Conclusions

In summary, we have synthesized five disperse azo dyes 1–5 in this paper. The structures of prepared dyes were confirmed by ¹H NMR, FTIR and UV–vis spectra. The electronic absorption spectra of disperse dyes were recorded in solvents with different physical–chemical properties. A large bathochromic shift (positive solvatochromism) of these compounds was observed upon increasing the solvent polarity. The influence of the pH changes on the dyes considered by comparison of the absorption maxima of these dyes in solutions with different pH, this fact directed us to the conclusion that the dye 5 having donor–donor substituent groups exist in acid–base equilibrium forms. Also, introduction of acyl group in the diazo component ring or alkyl amino group in the coupling component ring resulted in the bathochromic effect in studied solvents.

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pH=1

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