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Synthesis and spectral properties of some azo disperse dyes containing a benzothiazole moiety $\overset{\curvearrowleft}{\sim}$



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1. Introduction

Azo dyes with heterocyclic diazo components have been intensively investigated to produce bright and strong color shades ranging from red to greenish blue on synthetic fabrics [1,2]. These results led to commercial products to replace the conventional anthraquinone dyes [3]. Additionally, particularly bathochromic azo dyes that are structurally analogous to the azo benzenes may be obtained by replacement of the carbocyclic acceptor ring with a thiazole or benzothiazole ring [4–6]. These sulfur or sulfur-nitrogen containing heterocyclic azo dyes provide bright and strong shades that range from red through green and blue, which complement the vellow-orange colors of the nitrogen heterocyclic azo dyes to provide a complete coverage of the entire shade range [7–14]. It is also well known that azo dyes including thiazolyl components have been hugely utilized in the fields of nonlinear optics and optical data storage and have shown excellent optical properties in comparison with azo dyes derived from substituted anilines [15–19]. Derivatives of 2-aminobenzothiazole have a long history of use as heterocyclic diazo components for disperse dyes [20]. Some of the best-known representatives of coupling components for thiazole and benzothiazole diazo components are N,N-disubstituted anilines and their derivatives which are among the most widely used compounds for the synthesis of a vast variety of azo dyes and pigments [21–24]. In the present study, we report the synthesis of the four

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ABSTRACT

A known method was employed for the preparation of four substituted benzothiazole amines **I–IV** in moderate yields. These heterocyclic amines were diazotized with nitorsyl sulfuric acid and subsequently coupled with *N*,*N*-diethyl aniline and *N*-phenyl-2, 2'-iminodiethanol to afford heteroarylazo amine dyes **1–8** in satisfactory yields. These dyes were characterized by UV–Visible, FT-IR, ¹H NMR, and elemental analysis techniques. The solvatochromism behavior of the azo compounds is investigated by studying their spectra in pure and mixed organic solvents of different characteristics. The color of the dyes is discussed with respect to the nature of the heterocyclic ring and substituent present therein. In addition, effects of acid and base on the visible absorption maxima of the dyes are also reported.

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substituted 2-aminobenzothiazoles **I–IV** as diazo components (Scheme 1) and the subsequent monoazo disperse dyes **1–8**, starting from *N*,*N*-diethylaniline and *N*-phenyl-2,2'-imino diethanol (Scheme 2). The spectral properties of the prepared dyes were also evaluated in pure and mixed organic solvents of various polarities. The pH effect is also included with the aim of elucidating the absorption behaviors of the compounds under investigation.

2. Experimental

2.1. General

All starting materials were obtained from Merck and Aldrich chemical companies and were used without further purification. IR spectra were recorded on a Shimadzu 8400 FT-IR spectrophotometer. ¹H NMR spectra were obtained by FT-NMR 400 MHz Brucker apparatus in DMSO- d_6 using TMS as internal standard. The absorption spectra of the compounds were run on a Cary UV–Vis double-beam spectrophotometer (Model 100). Mass spectra were recorded on a Micromass Agilent Technology (HP) spectrometer, operating at 70 eV. The elemental analysis was determined on a Leco CHNS-900 analyzer. Melting points were recorded with an Electro-thermal apparatus and uncorrected.

2.2. Synthesis of 2-aminobenzothiazoles I-IV

A solution of bromine (0.26 mL, 10.0 mmol) in acetic acid (5.0 mL) was added over about 30 min to a mixture of aromatic amine (10.0 mmol) and potassium thiocyanate (1.07 g, 11.0 mmol) in acetic acid (20 mL), the temperature being kept between 25 and 35 °C. The

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Scheme 1. Synthetic routes for the prepared 2-aminobenzothiazoles.

slurry was then stirred for 20 h at room temperature. The precipitate was filtered, washed with a little acetic acid, slurried in water, made neutral with aqueous ammonia, and filtered again. The residue was boiled for 20 min with excess of hydrochloric acid (15% v:v) and the hot mixture was filtered from impurities. The filtrate, cooled to 10 °C, was made alkaline with aqueous ammonia and the precipitate was filtered, washed, and dried. The crude product was recrystallized from ethanol–water to afford **I–IV**, respectively. The physical and spectral data of the purified dyes are as follows.

2.2.1. 2-Amino-6-methyl benzothiazole I

White pearly crystals (0.80g, 49%), m.p. 118–120 °C (reported 137 °C [25]); FT-IR (KBr): ν (cm⁻¹): 3370 and 3212 (NH₂), 3075 (Aro.-H), 2950 (Aliph.-H), ¹H NMR (400 MHz, DMSO-*d*₆); δ ppm: 7.45 (1H, d, J = 8.2 Hz), 7.42 (1H, s), 7.15 (1H, dd, J = 1.1, 8.2 Hz), 5.55 (2H, br, NH₂), 2.41 (1H, s, -CH₃).

2.2.2. 2-Amino-6-bromo benzothiazole II

Yellow crystals (1.50 g, 65.5%), m.p187–190 °C; FT-IR (KBr): ν (cm⁻¹): 3375 and 3188 (NH₂), 3070 (Aro.-H), ¹H NMR (400 MHz, DMSO-*d*₆); δ ppm: 7.34 (1H, d, J = 7.9 Hz), 7.18 (1H, s), 7.03 (1H, dd, J = 2.0, 7.9 Hz), 5.45 (2H, br, NH₂).

2.2.3. 2-Amino-6-choloro benzothiazole III

White crystals (0.77 g, 42%), m.p. 208–211 °C (reported 209 °C [25]); FT-IR (KBr): ν (cm⁻¹): 3394 and 3218 (NH₂), 3050 (Aro.-H), ¹H NMR



Scheme 2. Synthetic routes for the preparation of azo dyes 1-8.

(400 MHz, DMSO- d_6); δ ppm: 8.10 (1H, s), 7.97 (1H, d, J = 8.0 Hz), 7.48 (1H, dd, J = 2.1, 8.0 Hz), 5.15 (2H, br, NH₂).

2.2.4. Naphtho [1,2-d]thiazol-2-amine IV

Pale brown crystals (1.23 g, 61.5%), mp > 350 °C; FT-IR (KBr): ν (cm⁻¹): 3414 and 3225 (NH₂), 3075 (Aro.-H), ¹H NMR (400 MHz, DMSO-*d*₆); δ ppm: 8.48 (1H, d, J = 8.4 Hz), 8.46 (1H, s), 8.29 (1H, d, J = 8.4 Hz), 8.05 (1H, s), 7.45 (1H, t, J = 7.0 Hz), 7.70 (1H, t, J = 7.0Hz), 6.25 (2H, br, NH₂), MS: Calcd for C₁₁H₈N₂S (M+) m/e 200.04. Found: 200.30.

2.3. Preparation of benzothiazoleazo dyes 1-8

2-Aminobenzothiazoles (2.0 mmol) were dissolved in glacial acetic acid: propionic acid mixture (2:1, 6.0 mL) and was quickly cooled in an ice-salt bath to 0-5 °C. The liquor was then added in portions during 15 min to a cold solution of nitrosyl sulfuric acid (prepared from sodium nitrite (2.2 mmol, 0.15g) and concentrated sulfuric acid (3 mL at 60 °C)). The mixture was stirred for an additional 3 h at the same temperature. After completion of diazotization procedure, the diazonium salt solution was added dropwise to the solution of coupler compounds (2.0 mmol) in acetic acid. The resulting solution was vigorously stirred at 0-4 °C for 2 h, while the pH of the reaction mixture was maintained at 5–6 by simultaneous addition of sodium hydroxide solution (0.5 M). The progress of the reaction was evaluated by thin layer chromatography (TLC) and then crude dyes were filtered, washed with cold ethanol and purified by recrystallization method. The physical and spectral data of the purified dyes are shown in Table 1.

3. Results and discussion

3.1. Synthesis and characterization

As depicted in Scheme 1, a known method was modified and then employed for the preparation of substituted 2-aminobenzothiazoles I– IV. The known 2-aminobenzothiazoles (I–III) and the new one (IV) were obtained by allowing one-pot reaction of the appropriate aniline derivatives with thiocyanogen generated from bromine and potassium thiocyanate. The crude products were obtained in the satisfactory yields and purified by crystallization from ethanol–water. In addition to comparison with authentic samples, all of the known and new prepared compounds were characterized by the FT-IR and ¹H NMR spectral analysis. For compound IV, the Mass spectrum was measured and confirmed the desired structure. ¹H NMR spectra recorded for the prepared

Table 1	
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Dye	Color	mp	Recrystallization	%C	%H	%N	Yield
		(°C)		Calcd. Found	Calcd. Found	Calcd. Found	(%)
1	Purple	170– 171	DMF/H ₂ O	66.63 67.15	6.21 6.19	17.27 17.88	72
2	Dark red	>250	DMF/H ₂ O	52.45 52.11	4.40 4.47	14.39 14.73	75
3	Bright red	160– 161	DMF/H ₂ O	59.21 59.92	4.97 4.91	16.25 16.87	74
4	Clear red	160– 162	DMF/H ₂ O	69.97 69.21	5.59 5.69	15.54 15.74	65
5	Dark red	187– 189	DMF/H ₂ O	60.65 60.89	5.66 5.61	15.72 15.25	69
6	Dark red	232– 234	DMF/H ₂ O	48.46 48.91	4.07 4.14	13.30 13.02	81
7	Clear red	183– 184	DMF/H ₂ O	54.18 54.92	4.55 4.58	14.87 15.23	63
8	Dark red	179– 180	DMF/H ₂ O	64.27 63.92	5.14 5.19	14.28 13.05	71

Table 2Characterization data of azo dyes 1–8.

Dye	IR (KB	r, cm ⁻¹)				¹ H NMR (DMSO- d_{6} , ppm)		
	$v_0 - H$	V _{AroC} - _H	$v_{AliphC} - H$	$v_c =_c$	$v_N =_N$	AroH	AlipH	0-H
1	-	3090	2980	1590	1495	$\begin{array}{l} 6.92~(d,2H,J{=}8.8Hz),7.35~(dd,1H,J{=}8.2,J{=}1.6Hz),7.82~(d,1H,J{=}1.6Hz),7.84\\ (d,2H,J{=}8.8Hz),7.88~(d,1H,J{=}8.8Hz) \end{array}$	1.20 (t, 6H, CH ₃ , J = 7.2 Hz), 2.46 (s, 3H, CH ₃), 3.56 (q, 4H, CH ₂ I = 7.2 Hz)	-
2	-	3080	2990	1598	1492	$6.95~(d, 2H, J{=}8.8Hz), 7.54~(dd, 1H, J{=}8.4, J{=}2.0Hz), 7.86~(d, 2H, J{=}8.8Hz), 7.98~(d, 1H, J{=}2.0Hz)$	1.20 (t, 6H, CH ₃ , J = 7.2 Hz), 3.58 (q, 4H, CH ₂ , J = 7.2 Hz)	-
3	-	3100	2984	1600	1490	6.94 (d, 2H, J = 9.2 Hz), 7.78 (d, 1H, J = 8.8 Hz), 7.82 (d, 2H, J = 9.2 Hz), 7.89 (d, 1H, J = 8.8 Hz), 8.20 (s, 1H)	1.36 (t, 6H, CH ₃ , J = 7.1 Hz), 3.53 (q, 4H, CH ₂ , J = 7.1 Hz)	-
4	-	3150	2985	1592	1519	7.02 (d, 2H, J = 9.2 Hz), 7.75 (t, 1H, J = 7.2 Hz), 7.84 (t, 1H, AroH, J = 7.2 Hz), 7.90 (d, 2H, J = 9.2 Hz), 8.09 (d, 1H, J = 7.2 Hz), 8.31 (d, 1H, J = 7.2 Hz), 8.48 (s, 1H), 8.78 (s, 1H)	1.28 (t, 6H, CH ₃ , J = 7.1 Hz), 3.53 (q, 4H, CH ₂ , J = 7.1 Hz)	-
5	3400	3085	2920	1595	1493	6.90 (d, 2H, J = 8.8 Hz), 7.52 (d, 1H, J = 8.4 Hz), 7.95 (d, 1H, J = 8.4 Hz), 8.01 (d, 2H, J = 8.8 Hz), 8.04 (s, 1H)	2.50 (s, 3H, CH ₃), 3.35 (s, 8H, CH ₂)	4.88 (br, 2H)
6	3445	3100	2950	1630	1525	7.01 (d, 2H, AroH, J = 8.8 Hz), 7.66 (dd, 1H, AroH, J = 8.8, J = 2.0 Hz), 7.84 (d, 2H. AroH, J = 8.8 Hz), 7.92 (d, 1H, AroH, J = 8.8 Hz), 8.32 (d, 1H, AroH, J = 2.0 Hz)	3.66 (s, 8H, CH ₂)	4.94 (br, 2H)
7	3390	3140	2936	1597	1498	7.07 (d, 2H, AroH, J = 8.9 Hz), 7.54 (dd, 1H, Aro H, J = 8.4, 2.0 Hz), 7.89 (d, 2H, Aro H, J = 8.9 Hz), 7.95 (d, 1H, AroH, J = 8.4 Hz), 8.26 (d, 1H, AroH, J = 2.0 Hz)	3.55 (t, 4H, N-CH ₂ , J = 5.2 Hz), 3.66 (t, 4H, OCH ₂ , J = 5.2 Hz)	5.10 (t, 2H, J = 5.20 Hz)
8	3422	3145	2994	1593	1521		3.69 (s, 8H, CH ₂)	4.92 (br, 2H)

compounds clearly supported the proposed structures. The protons belonging to the aromatic system were observed at the expected chemical shifts and integral values.

In order to synthesize the aminoazo dyes **1–8**, 2-aminobenzothiazoles **I–IV** were diazotised with nitrosylsulfuric acid in a mixture of acetic and propionic acids. Coupling reaction was carried out by adding the diazonium solution to a solution of *N*,*N*-disubstituted anilines in acetic acid (Scheme 2). The chemical structures of these dyes were confirmed by some spectroscopic methods and elemental analysis. The physical and spectral data of the dyes are summarized in Tables 1 and 2. Spectral measurements of the synthesized azo dyes revealed good agreement with proposed structures. FT-IR spectrum of compounds **1–8** showed aromatic and aliphatic C– H bands at 3080–3150 cm⁻¹ and 2936–2990 cm⁻¹, respectively. The dyes also revealed the aromatic C=C bands at 1590– 1630 cm⁻¹ and the azo N=N peaks at 1490–1525 cm⁻¹ in their IRspectral data.

The aromatic protons of the dyes were observed from 8.78 to 6.92 ppm in the ¹H NMR spectra measured in DMSO-d₆ at 25 °C. Dyes **1–4** indicated a quartet peak at $\delta = 3.58-3.53$ ppm and a triplet peak about $\delta = 1.36-1.20$ ppm. These signals were attributed to the adjacent methylene and methyl protons, respectively. Other significant signals that were detected in ¹H NMR spectrum of dyes **5–8**, are two distinct quartets at 3.69–3.35 ppm which can be ascribed to methylene protons connected to nitrogen and oxygen atoms. In addition, the singlet and triplet of hydroxyl group were observed at $\delta = 5.10$ –4.88 ppm for these dyes.

3.2. UV–Visible study of the prepared aminoazo dyes

The absorption spectra of aminoazo dyes **1–8** were measured in six organic solvents with different polarities (chloroform, ethanol, acetic

 Table 3

 Absorption maxima (in nm) of dyes 1–8 in different solvents.

Dye	DMSO	DMF	CH ₃ CN	CHCl ₃	EtOH	CH₃COOH
1	526	520	512	516	508	566, 602
2	540	528	520	518	524	560, 596
3	536	528	522	512	528	558, 592
4	556	548	540	540	544	560
5	528	520	508	498	510	576, 608
6	538	530	518	506	520	542, 602
7	540	530	520	512	522	538, 604
8	556	548	536	528	538	542

acid, acetonitrile, dimethyl formamide and dimethyl sulfoxide) in the wavelength rang 350–700 nm at a concentration $\sim 10^{-5} \text{ mol} \cdot \text{L}^{-1}$. The results are given in Table 3. The dyes have low solubility in some of the used solvents but are completely soluble in DMSO. Therefore, stock solutions of each dye with a concentration of $\sim 10^{-3} \text{ mol} \cdot \text{L}^{-1}$ were accurately prepared in DMSO and dilutions of these stocks were used for absorption measurements.

As an illustrative example, the spectral shifts of dye **6** in various solvents are shown in Fig. 1. The maximum absorption of dye **6** showed a significant shift in CH₃COOH and DMSO with respect to the maximum absorption in other solvents (e.g. λ_{max} is 542 in CH₃COOH, 538 nm in DMSO, 530 nm in DMF, 520 nm in ethanol, 518 nm in CH₃CN, and 506 nm in chloroform). The same trends of absorption shifts in various solvents were observed for the entire series of dyes **1–8**.

As it is apparent in Table 3, the electronic absorption spectra of these benzothiazole aminoazo dyes don't show a regular variation with the polarity of solvents. These dyes, apparently, didn't exhibit a strong solvent dependence.

The substituent effects on the visible absorption spectra of the heterocyclic azo dyes **1–8** were also evaluated. For example, we found that dyes **3** and **7** that contain an electron acceptor chloro group on the 6 positions of benzothiazolyl group showed a bathochromic shift of +10 and +12 nm relative to dyes **1** and **5** in DMSO, respectively. It was also observed that the aminoazo dyes which have naphtothiazolyl group (**4** and **8**) show bathochromic shifts in comparison with benzothiazolyl amines. As a



Fig. 1. Absorption spectra of dye 6 in different solvents.



Scheme 3. Tautomeric changes on protonation of aminoazo dyes.

result, the λ_{max} of dye **4** showed bathochromic shift of +30 nm relative to **1** and λ_{max} of dye **8** is +28 nm longer than that of dye **5**, due to the extended resonance system in comparison with other dyes. Table 1 also illustrates the effect of two different coupling components. The dyes **1–4** which are based on the *N*, *N*-diethyl aniline as coupling component showing λ_{max} at the highest wavelength in the series.

As is well known [26–28] and shown in Scheme 3, in strong proton donating solvents such as acetic acid, azo dyes with amino groups in the para position undergo mono-protonation to give the ammonium (**B**) and/or the azonium (**C**) tautomers followed by further protonation to give (**D**). The color change for the ammonium isomer is hypsochromic and weaker (even colorless), while the azonium isomer is strongly bathochromic and more intense. The di-protonated product (**D**) is nearly the same color as the base dye.

The absorption maximum of all dyes except for dyes **4** and **8** showed two bands and bathochromic shift (positive solvatochromism) in acidic solvent (glacial acetic acid) relative to other investigated solvents in this study. This shift in absorption maximum can be attributed to the solvent-dye interaction and the presence of these dyes in azonium form



Absorption maxima of the prepared dyes 1-8 were also measured in their 80% water-ethanol solutions with different pH values (Table 4). The dyes showed relatively good pH stability. The nature of these changes in solutions with different acidity is the same, as shown for dye 1 in Fig. 3. Changes in the spectra are observed at acidity higher than ~ 10^{-2} mol·L⁻¹ (pH \leq 1.5) and the absorption curves are resembled those in high proton donating solvents such as acetic acid. It can be suggested that a bathochromically shifted band appears as a result of proton addition and this is attributed to the azonium (**C**) tautomer in the possible azonium-ammonium equilibrium. The appearance of this band only in acetic acid as a solvent can be ascribed to its high acidity as well as its pronounced character as a strong proton donating solvent. Further support is achieved by studying the spectroscopic behavior of these compounds in DMF containing increasing amounts of acetic acid. The spectra of the dyes were recorded in the DMF-acetic acid mixed solvents. All of the dyes except dyes 4 and 8 exhibited an isosbestic point which indicates the establishment of equilibrium between the free (azo form A) and the H-bond solvated species (azonium form C) of the dyes. This behavior indicates that acetic acid molecules have a greater tendency to form solvated complexes with the dye molecule than DMF. It is evident that with increasing acetic acid concentration the absorbance of first band decreases, whereas a new band appears at a longer wavelength. Fig. 4 shows the absorption spectra of



Fig. 2. Absorption spectra of dyes 1–8 in acetic acid (numbers represent the number of the dyes).

Table 4				
Absorption maxima (λ_{max} in nm) of	prepared azo	dyes in different	pH values.

	pH								
	1.50	2.97	3.39	5.45	7.05	7.30	10.00	11.05	
Dye	λ_{max}								
1	528, 608	522	524	522	524	524	522	522	
2	532, 606	532	532	532	530	534	530	532	
3	532, 610	530	532	528	528	530	530	528	
4	548	544	552	546	550	552	554	552	
5	518, 614	516	516	516	516	516	516	516	
6	526, 612	526	526	526	526	526	526	526	
7	526, 602	528	526	528	526	526	526	528	
8	544	542	544	542	542	544	542	542	



Fig. 3. Absorption spectra of dye 1 in different pH values.

dye 6 in mixed DMF-acetic acid. The spectrum does not show a band at about 320 nm due to the ammonium tautomer, and is influenced substantially by the acid concentration. The tautomeric equilibrium in these dyes is thus shifted almost completely to the azonium form [25].

This is due to the low ionization potential of acetic acid as well as to its high H-bond donating character. The visible spectra of compounds 4 and 8 in acetic acid are different from those discussed above, the absorption band being broad and exhibiting only one maximum. However, the wavelength bands are located within the range of the spectra of the other discussed dyes (1-3 and 5-7) in the same solvent. It can be concluded that dyes **4** and **8** are liable to form a solvated complex with acetic acid through an intermolecular H-bonding.

4. Conclusions

Four 2-aminobenzothiazole derivatives I-IV have been synthesized and characterized by elemental analysis, IR, UV-Vis and ¹HNMR spectroscopy. These compounds were diazotized and successfully coupled with N,N-diethyl aniline and N-phenyl-2, 2'-iminodiethanol to prepare corresponding azo dyes 1-8 in moderate to good yields. The solvatochromic behavior of these compounds was investigated by studying their visible spectra in several pure and mixed organic solvents.

The azo compounds displayed one band in their electronic absorption spectra in all used organic solvents except acetic acid. This band was attributed to electronic transition in aromatic and heteroaromatic systems involving the entire electronic system of the compounds. Whereas in acetic acid a second band at longer wavelengths for some azo compounds (i.e., 1-3 and 5-7) was observed.

The observation of a special behavior for compounds 1-3 and 5-7 in the presence of CH₃COOH in DMF-CH₃COOH mixture is examined and indicated that CH₃COOH has a greater tendency to form a solvated



Fig. 4. Visible electronic absorption spectra of 5.0×10^{-5} mol·L⁻¹ solutions of compound 6 in DMF-CH₃COOH mixtures. 1. 1.1 mol·L⁻¹ CH₃COOH, 2. 2.18 mol·L⁻¹ CH₃COOH, 3. 2.87 mol·L⁻¹ CH₃COOH, 4. 3.45 mol·L⁻¹ CH₃COOH, 5. 5.24 mol·L⁻¹ CH₃COOH, 6. 6.01 mol·L⁻¹ CH₃COOH, 7. 8.13 mol·L⁻¹ CH₃COOH, 8. 9.25 mol·L⁻¹ CH₃COOH, 9. 10.12 mol·L⁻¹ CH₃COOH, 10. 11.00 mol·L⁻¹ CH₃COOH, and 11. 12.11 mol·L⁻¹ CH₃COOH.

complex with the solute molecules. This is due to the low ionization potential and high hydrogen bond donating character of CH₃COOH.

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