



Study of Environment and pH Effects on the Spectral Behavior of Arylazobenzylidene Dyes

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ABSTRACT

In this study, four arylazobenzylidene dyes (a-d) were synthesized with excellent yields in two steps using Knoevenagel condensation and azo-coupling reactions. Absorption spectra of these dyes with different substituents were investigated in organic solvents as a function of solvent polarity. The spectral features of synthesized arylazobenzylidene dyes were described according to azo-hydrazone tautomerism as well as the solvatochromic characteristic of the approved tautomer. The nature and degree of solute-solvent interactions were illustrated using Kamlet-Taft multi-parameter polarity scales. The effects of acid-base on the UV-visible absorption spectra of the dyes were reported. In addition, different absorption characteristics were observed for these dyes in pure diethanolamine. Prog. Color Colorants Coat. 11 (2018), 69-78 © Institute for Color Science and Technology.

1. Introduction

Azo compounds are the most commonly used colorants in various areas, such as dyeing of fabrics [1, 2], chemo-sensors [3, 4], ink jet printing [5], thermal transfer printing [6], photography [7], color additives [8], biomedical area [9], molecular recognition [10], light controlled polymers [11], liquid crystal industry [12]. Furthermore, bis-azo dyes as disperse colorants have been fully studied to produce bright and strong color shades on synthetic textile fibers. These results led to the development of commercial products as a substitution for conventional azo-benzene disperse dyes. Several synthetic ways have been developed to synthesize bis-azo derivatives. However, the common methods for synthesis of these classes of dyes are the using of diazotization-coupling reactions by aromatic diamines or phenols [13-15]. On the other hand, it is

well known that azo dyes with hydroxyl group at ortho to azo band display tautomerism depending on the proton transfer. The physico-chemical features of these types of dyes are thoroughly connected to their tautomerism. Determination and characterization of azo-hydrazone tautomers in both solid and solution phases is really interesting from both the theoretical and experimental point of views, since the tautomers have different technical properties [16-20]. In other words, the type of solvent used can affect the absorption maxima of the azo dye. When absorption spectra are measured in solvents of different polarity, the positions, intensities, and shapes of the absorption bands are usually modified by these solvents. These changes are a result of physical intermolecular solute-solvent interaction forces (such as ion-dipole, dipole-dipole, hydrogen bonding, etc.), which tend to change

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the energy difference between the ground state and excited state of the absorbing species containing chromophore [21-24]. In previous publications, the absorption spectra of various arylazobenzylidene dyes in polar and non-polar solvents were studied and the results revealed that these dyes are influenced by the substituents and the solvent used. It was also concluded that the arylazobenzylidene dyes exist in azo-hydrazone tautomeric forms [15, 25].

In this paper, the synthesis route of four arylazobenzylidene dyes (Scheme 1) and their absorption spectra in organic solvents as a function of solvent polarity are reported. The spectral properties of synthesized dyes were described in accordance with azo-hydrazone tautomerism as well as solvatochromic characteristic of the approved tautomer. The nature of solute-solvent interactions was described using Kamlet-Taft multi-parameter polarity scales. The effects of acid-base on the UV-Vis absorption spectra of the prepared compounds were also reported.

2. Experimental

2.1. Materials and apparatus

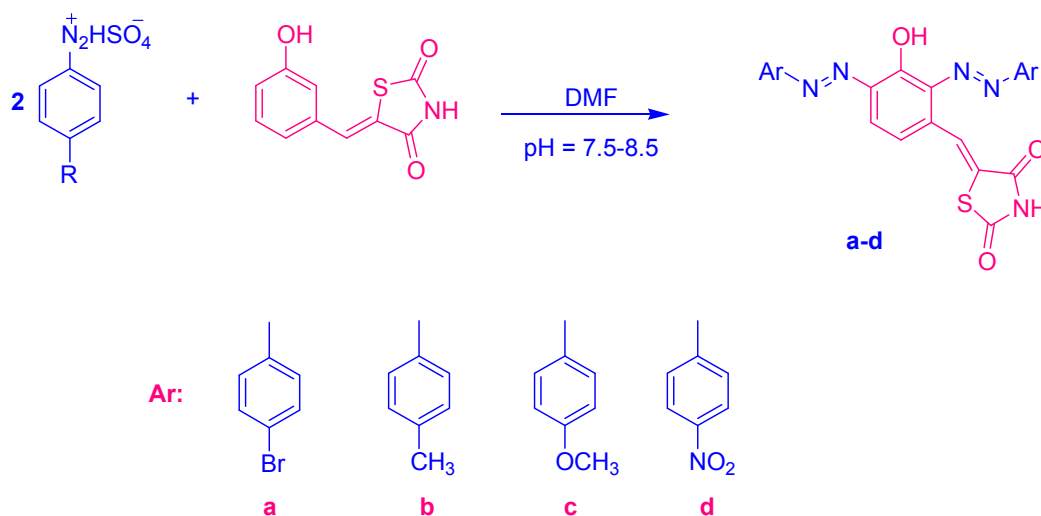
All chemicals used in this study were obtained from Merck Chemical Companies and were used without further purification. All melting points were determined on an Electrothermal melting point apparatus and are uncorrected. Infrared spectra were recorded on a Shimadzu 8400 FT-IR spectro-

photometer. The Proton nuclear magnetic resonance (^1H NMR) spectra were obtained on a FT-NMR (400 MHz) Bruker apparatus spectrometer, and the chemical shifts are expressed in δ ppm using TMS as an internal standard. The absorption spectra of the dyes were recorded on a double beam Shimadzu UV-Visible spectrophotometer (Model UV-2450 Scan). The purity determination of the substrates and reaction monitoring were accompanied by TLC using silica gel SIL G/UV 254 plates.

2.2 Synthesis of arylazobenzylidene dyes

In the present work, four arylazobenzylidene dyes containing donor and acceptor substituents were prepared using previously described methods [15, 26]. The structures of the prepared compounds were confirmed by analyzing their spectral characteristics.

Dye a: Red solid; yield 86%; M.p. 233-234 °C; FT-IR (KBr) ν cm^{-1} : 3114 (NH), 3045 (=C-H), 1744 (C=O), 1704 (C=O), 1608 (C=C), 1506 (N=N); ^1H NMR (400 MHz, DMSO- d_6) δ : 6.72 (dd, 1H, $J = 4.4$ Hz, Ar-H), 7.52 (d, 4H, $J = 8.4$ Hz, Ar-H), 7.59 (d, 2H, $J = 8.4$ Hz, Ar-H), 7.84 (d, 2H, $J = 8.4$ Hz, Ar-H), 7.90 (d, 1H, $J = 10$ Hz, Ar-H), 9.86 (s, 0.37H, OH), 12.29 (s, 1H, NH), 15.66 (br s, 0.63H, N-NH). ^{13}C NMR (DMSO- d_6): δ 115.9, 117.4, 118.6, 129.2, 129.3, 129.8, 130.9, 132.8, 135.1, 141.6, 143.6, 157.0, 157.6, 159.4, 161.1, 166.2, 170.9, 174.9, 178.5 ppm.



Scheme 1: Synthesis of arylazobenzylidene dyes (a-d).

Dye b: Dark Red solid; yield 83%; m.p. 244–245 °C; FT-IR (KBr) ν cm^{-1} : 3300 (OH), 3112 (NH), 3040 (=C-H), 1744 (C=O), 1700 (C=O), 1680 (C=O), 1610 (C=C), 1504 (N=N); ^1H NMR (400 MHz, CDCl_3) δ : 2.39 (s, 3H, CH_3), 2.48 (s, 3H, CH_3), 6.75 (d, 1H, $J = 9.6$ Hz, Ar-H), 7.25 (d, 2H, $J = 8.4$ Hz, Ar-H), 7.36 (m, 4H, Ar-H), 7.45 (d, 2H, $J = 8.0$ Hz, Ar-H), 7.73 (d, 1H, $J = 9.6$ Hz, Ar-H). ^{13}C NMR (DMSO-d_6): δ 26.7, 26.9, 119.8, 129.4, 130.0, 131.9, 133.5, 136.0, 141.4, 145.6, 161.4, 162.6, 164.3, 170.5, 171.9, 178.2, 178.7 ppm.

Dye c: Dark Red solid; yield 86%; m.p. 264–265 °C; FT-IR (KBr) ν cm^{-1} : 3112 (NH), 3048 (=C-H), 1744 (C=O), 1700 (C=O), 1680 (C=O), 1605 (C=C), 1508 (N=N); ^1H NMR (400 MHz, DMSO-d_6) δ : 2.33 (s, 3H, OCH_3), 2.43 (s, 3H, OCH_3), 6.72 (dd, 1H, $J = 5.2$ Hz, Ar-H), 7.25 (d, 2H, $J = 7.6$ Hz, Ar-H), 7.42 (br s, 6H, Ar-H), 7.86 (d, 1H, $J = 9.6$ Hz, Ar-H), 12.23 (s, 1H, NH), 15.72 (br s, 0.44H, N-NH), 16.25 (br s, 0.56H, N-NH). ^{13}C NMR (DMSO-d_6): δ 59.1, 59.8, 119.1, 127.7, 128.2, 130.4, 132.2, 132.9, 136.9, 140.3, 145.4, 161.2, 161.6, 161.8, 164.8, 170.1, 178.0, 178.7 ppm.

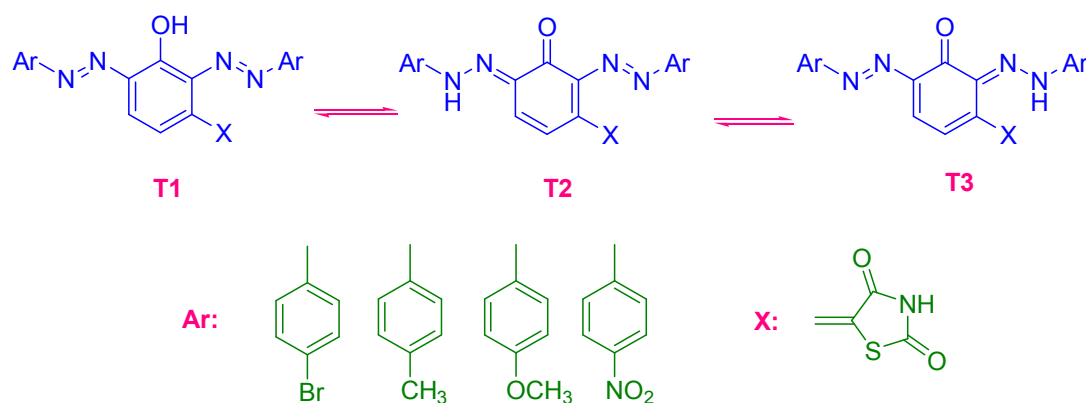
Dye d: Light Red solid; yield 79%; m.p. 299–300 °C; FT-IR (KBr) ν cm^{-1} : 3200–3440 (OH, NH), 3066 (=C-H), 1740 (C=O), 1700 (C=O), 1680 (C=O), 1598 (C=C), 1510 (N=N); ^1H NMR (400 MHz, DMSO-d_6) δ : 6.70 (d, 1H, $J = 9.6$ Hz, Ar-H), 7.70 (d, 2H, $J = 8.0$ Hz, Ar-H), 7.88 (d, 2H, $J = 8.8$ Hz, Ar-H), 7.93 (d, 1H, $J = 9.6$ Hz, Ar-H), 7.96 (s, 1H, $\text{CH}=\text{C}$), 8.24 (d, 2H, $J = 8.8$ Hz, Ar-H), 8.49 (d, 2H, $J = 8.8$ Hz, Ar-H), 9.85 (s, 0.12H, OH), 12.39 (br s, 1H, NH), 15.23 (br s,

0.74H, N-NH). ^{13}C NMR (DMSO-d_6): δ 118.1, 125.6, 127.4, 128.3, 131.0, 131.9, 134.2, 138.6, 145.1, 149.5, 148.0, 150.4, 150.8, 179.8, 182.1 ppm.

3. Results and Discussion

3.1. Synthesis and spectral characteristics

The treatment of corresponding prepared diazonium salts with 5-(3-hydroxybenzylidene) thiazolidine-2,4-dione, as important intermediate in alkaline medium, provided novel bis-azo dyes **a-d** in good yields (Scheme 1). The structures of the prepared dyes were confirmed by the IR, ^1H and ^{13}C -NMR spectroscopy. The dyes synthesized in this study may exist in three main tautomeric forms (Scheme 2). It was achieved that the equilibrium between the tautomers is affected by the structure of the dye and the solvent used. The NMR spectra of the dyes exhibited significant signals at δ 15.23–16.36 and around 9.85 ppm. These signals correspond to the imine N–H proton resonance of the hydrazone form and the hydroxyl O–H proton resonance of the azo-enol form of compounds, respectively. The UV-Vis absorption spectra of all the prepared organic dyes showed two strong bands at about 280–360 and 450–550 nm, which can be attributed to $n \rightarrow \pi^*$ and/or $\pi \rightarrow \pi^*$ electronic transitions of azo (T1) and hydrazo (T2 and T3) tautomeric forms, respectively. The UV-Vis, IR, ^1H and ^{13}C -NMR spectra also showed important signals at their expected positions for all prepared bis-azo dyes.



Scheme 2: The equilibrium between the azo form (T1) and the hydrazone forms (T2 and T3) of arylazobenzylidene dyes.

3.2. The UV–Visible spectra and solvatochromic studies

In order to study the solvent effects on spectral features of the dyes, we recorded their absorption spectra in 14 solvents with different polarities at a concentration of 1×10^{-5} M at room temperature. The UV–Vis absorption spectra of dyes **a-d** in selected solvents are shown in Figure 1.

The UV-Vis absorption spectra of the synthesized dyes showed two absorption bands at about 322-338 nm and 478-512 nm, assigned to the azo and hydrazone tautomeric forms, respectively (Scheme 2). The UV-Vis absorption spectra of the dyes (a-d) are affected by the solvent-solute interactions. As seen in Figure 1, the absorption bands of all dyes are shifted bathochromically when the solvent polarity increases.

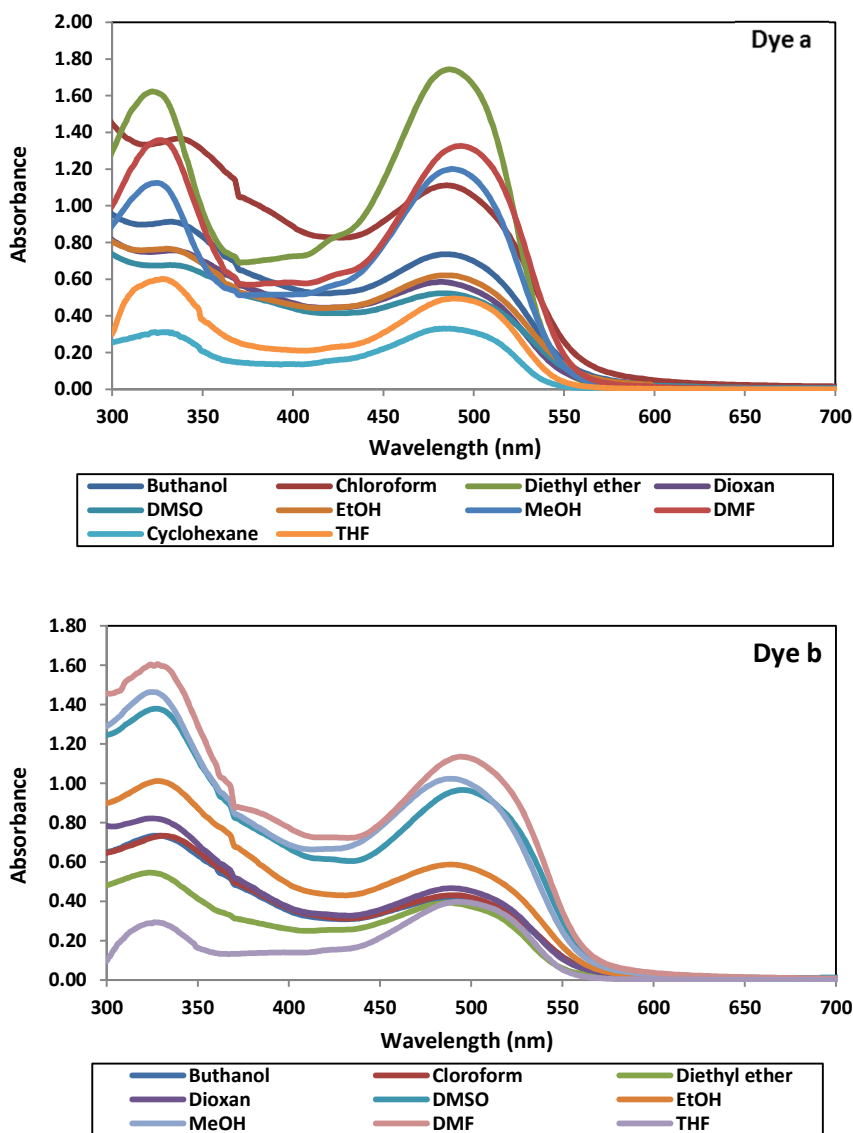


Figure 1: The UV–Vis absorption spectra of dyes **a-d** in selected solvents.

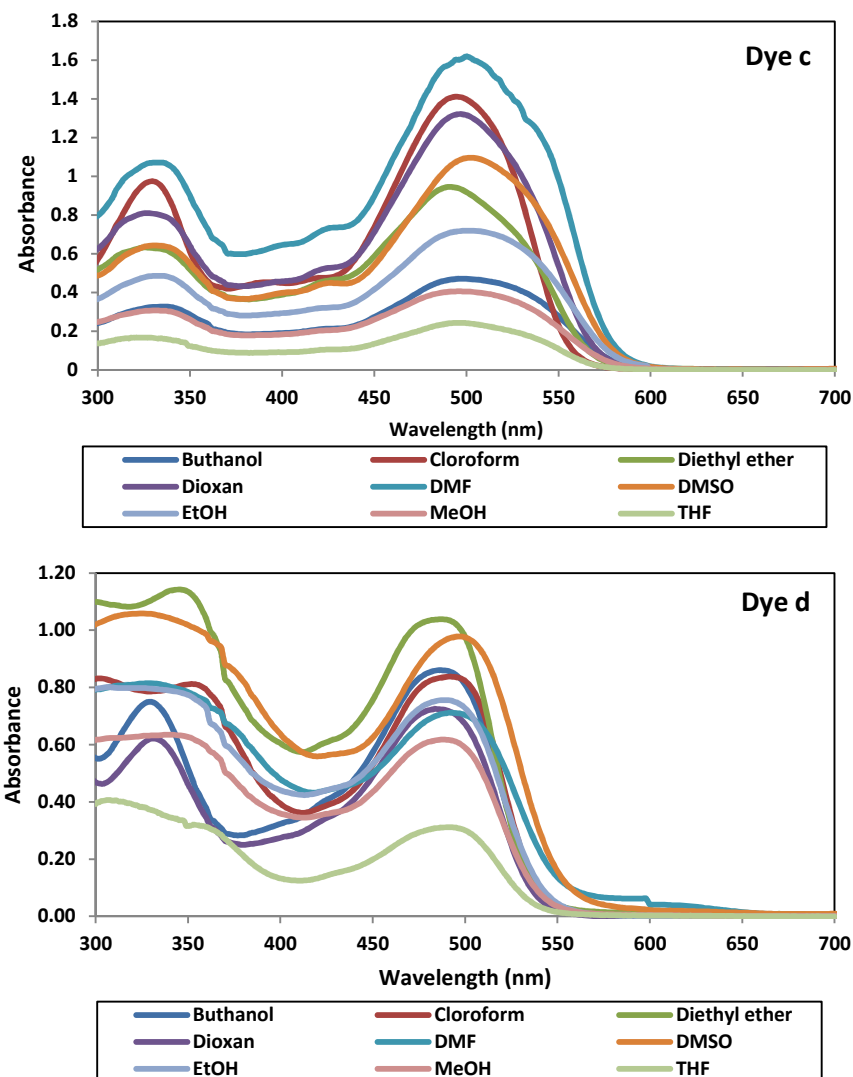


Figure 1: Continue.

This effect in different solvents may be due to the change in the solvation behavior leading to the formation of azo and hydrazone tautomeric forms. On the other hand, the tautomeric equilibria strongly depend on the solvent-solute interactions. In addition, the data from Table 1 and Figure 1 indicate that the azo and hydrazone tautomeric equilibria of investigated dyes depend on the substituent effect [27]. Therefore, the introduction of electron-donating substituents into diazo components in all solvents causes the tautomeric equilibria to shift to the azo tautomeric form in the solution. Thus, it appears that the azo form is stabilized more with electron-donating substituents on the diazo components [28]. The results presented in Table 1 are in agreement with these conclusions.

In addition, multi-parameter solvent polarity scale can be used for quantitative assessment of the

solvent/solute interaction and the absorption shifts. The solvent effects on the absorption spectra of dyes are interpreted using Kamlet-Taft Solvatochromic Equation (1) of the following form [29]:

$$\nu = \nu_0 + s\pi^* + b\beta + a\alpha \quad (1)$$

where π^* is an index of the solvent dipolarity/polarizability, β is a measure of the solvent hydrogen-bonding acceptor (HBA) basicity, α is a measure of the solvent hydrogen-bonding donor (HBD) acidity and ν_0 is the regression value of the solute effect in cyclohexane as the reference solvent. The regression coefficients s , b and a in Eq. (1) measure the susceptibilities of the solvent-dependent solute property relative to the indicated solvent parameters. The solvent parameters [30] are given in Table 2.

Table 1: Absorption maxima of the azo and hydrazone tautomers of dyes (a-d) in the studied solvents.

Solvent	Dyes											
	a			b			c			d		
	λ_{azo}	$\lambda_{\text{hydrazone}}$	$A_{\text{h}}/A_{\text{a}}$	λ_{azo}	$\lambda_{\text{hydrazone}}$	$A_{\text{h}}/A_{\text{a}}$	λ_{azo}	$\lambda_{\text{hydrazone}}$	$A_{\text{h}}/A_{\text{a}}$	λ_{azo}	$\lambda_{\text{hydrazone}}$	$A_{\text{h}}/A_{\text{a}}$
Cyclohexane	322	484	1.25	-	487	~0	-	-	-	-	-	-
Diethyl ether	322	486	1.04	324	486	0.72	326	492	1.49	324	488	0.95
1-BuOH	330	493	1.00	328	496	1.42	338	498	1.63	332	486	0.84
EtOH	327	496	0.78	327	502	0.56	333	511	1.47	303	490	0.94
Ethylacetate	332	480	0.76	324	486	0.76	328	492	1.62	334	486	0.87
1,4-Dioxane	332	484	0.77	330	490	0.56	326	496	1.62	332	484	1.16
Acetic acid	332	488	0.84	330	494	0.58	336	510	1.72	334	488	0.92
THF	328	488	0.80	327	493	1.50	327	495	1.50	308	492	0.75
MeOH	324	488	1.06	326	490	0.82	332	498	1.33	332	488	0.96
Acetone	326	478	1.08	328	484	0.60	334	508	1.36	330	490	0.93
Chloroform	334	486	0.58	330	490	0.58	330	494	1.42	332	492	1.06
Dichloromethane	336	484	0.72	328	488	0.53	332	498	1.50	334	492	0.95
DMF	326	494	1.02	328	496	0.68	326	500	1.52	330	492	0.87
DMSO	332	332	0.78	328	496	0.70	332	504	1.70	326	498	0.92

Table 2: Solvent parameters.

Solvents	α	β	π^*
Cyclohexane	0.00	0.00	0.00
1,4-Dioxane	0.00	0.37	0.49
Chloroform	0.20	0.10	0.69
Dichloromethane	0.13	0.10	0.73
Ethylacetate	0.00	0.45	0.55
Diethyl ether	0.00	0.47	0.27
Acetic acid	1.12	0.45	0.64
1-BuOH	0.69	0.80	0.40
Acetone	0.08	0.48	0.71
EtOH	0.86	0.75	0.54
MeOH	0.98	0.66	0.60
THF	0.00	0.55	0.58
DMF	0.00	0.69	0.88
DMSO	0.00	0.76	1.00

Table 3: Regression fits to the solvatochromic parameters (Eq.1).

Dye		ν_0	a	b	s	R ²	Prob.>F
a	$\bar{\nu}_{azo}(cm^{-1})$	29818.86	204.999-	1270.777	-108.56	0.94	0.001432
	$\bar{\nu}_{hydrazone}(cm^{-1})$	21209.27	97.1518-	-740.383	548.202-	0.912	0.001412
b	$\bar{\nu}_{azo}(cm^{-1})$	366009.5	-157239	81732.25	-40764	0.94	0.024417
	$\bar{\nu}_{hydrazone}(cm^{-1})$	21038	131.718-	-640.929	-729.99	0.951	0.001015
c	$\bar{\nu}_{azo}(cm^{-1})$	30554.46	-790.28	498.2836	-312.12	0.9490	0.004782
	$\bar{\nu}_{hydrazone}(cm^{-1})$	20711.95	-580.96	-409.426	-552.55	0.966	<0.001
d	$\bar{\nu}_{azo}(cm^{-1})$	30217.26	1255.848	1573.81	-792.48	0.938	0.091069
	$\bar{\nu}_{hydrazone}(cm^{-1})$	20742.29	104.3258	-216.51	-484.72	0.966	0.013207

The correlations of the spectroscopic data for azo and hydrazone tautomers of synthesized dyes were determined using multiple linear regression analysis. It was concluded that the absorption frequencies (ν_{max}) for investigated dyes in polar and non-polar solvents showed satisfactory correlation with π^* , β and α parameters (Table 3). The negative sign of coefficients **b** and **s** for all dyes indicates a bathochromic shift with both increasing the solvent hydrogen bond acceptor basicities and solvent dipolarity/polarizability. This suggests the stabilization of excited electronic state in respect to the ground state. The positive sign of coefficient **a** for all dyes indicates a hypsochromic shift with increasing the solvent hydrogen bond donor acidities. This suggests the stabilization of the ground state regarding the excited electronic state.

The percentage contributions of the solvatochromic parameters for the azo and hydrazone tautomers of synthesized dyes showed that most of the solvatochromism in the hydrazone form is due to the solvent dipolarity/polarizability and solvent basicity rather than solvent acidity (Table 4). However, for the dye **c** with electron-donating group in arylazo moiety, the major contribution comes from the solvent hydrogen-bonding donor acidity. Furthermore, in the azo form, all the solvent parameters contribute significantly. This shows that the absorption bands in the investigated dyes respond differently to the various solvent parameters which include nonspecific (π^*) and specific

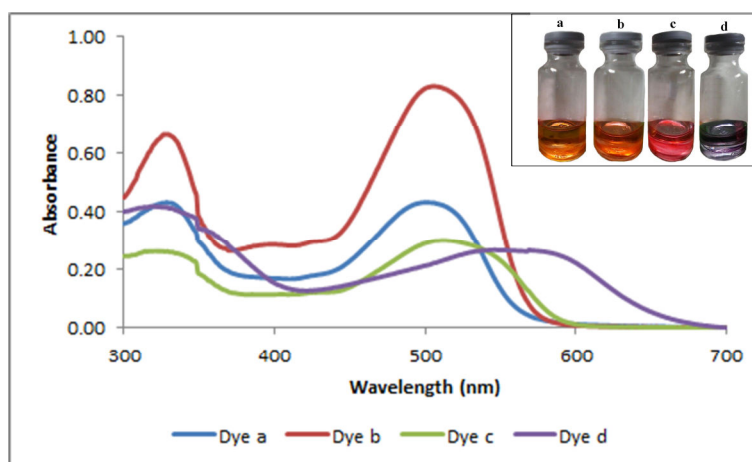
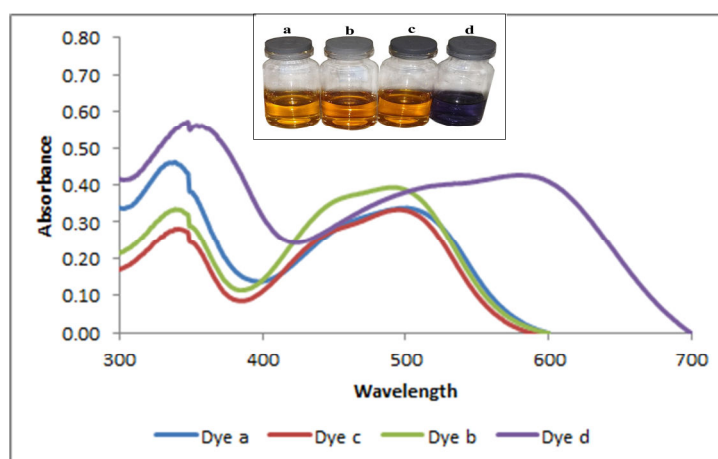
interaction (HBD and HBA). These consequences could be described by the effect of positive charge on the nitrogen (-NH) atom in the hydrazone form and stabilization of this form commonly due to the solvent dipolarity/polarizability using hydrogen bond donating and hydrogen bond accepting features.

3.3. Effect of diethanolamine on the spectral properties of investigated dyes

The absorption spectra of the dyes (a–d) at a concentration of 1×10^{-5} M were recorded in diethanolamine as a polar protic solvent. The absorption spectra of the investigated dyes in diethanolamine and diethanolamine +NaOH solutions are shown in Figures 2 and 3. The presence of one secondary amino group and two hydroxyl groups on the structure of diethanolamine causes several interactions between dye solute and solvent molecules. These interactions can be specific, such as hydrogen bonding, and non-specific, such as dipole-dipole interactions. It is obvious that the absorption spectra of compound **d** with electron-withdrawing group (NO_2) are affected by the diethanolamine and diethanolamine +NaOH solutions. As seen in Figures 2 and 3, the UV-Vis absorption spectra of the *p*-nitro derivative (compound **d**) were shifted bathochromically when compared with other dyes (dyes a-c). These changes in spectral position for compound **d** can be attributed to different types of interactions between the dye solute and diethanolamine solvent.

Table 4: Percentage contribution of the solvatochromic parameters.

Tautomers	Dye	Substituent	P_{α} (%)	P_{β} (%)	P_{π^*} (%)
Azo	a	Br	13	80	7
	b	CH ₃	24	13	63
	c	OCH ₃	49	31	19
	d	NO ₂	35	43	22
Hydrazone	a	Br	7	53	40
	b	CH ₃	9	43	49
	c	OCH ₃	38	27	36
	d	NO ₂	13	27	60

**Figure 2:** The UV-Vis absorption spectra of dyes a-d in diethanolamine.**Figure 3:** The UV-Vis absorption spectra of dyes a-d in diethanolamine+NaOH solution.

4. Conclusions

In this study, four arylazobenzylidene dyes with electro-donating and electron-withdrawing substituents on the arylazo group were synthesized and fully characterized. Electronic absorption pattern of investigated dyes displayed that the solvent effect on UV-Vis absorption spectra completely depends on the feature of the substituent on the diazo component. The spectral properties have been satisfactorily correlated with solvatochromic parameters, α , β and π^* using linear solvation energy relation. The spectral properties of the dyes were influenced by dipolarity/polarizability, hydrogen bond donocity and hydrogen bond acceptor ability. The percentage contributions of the

solvatochromic parameters for the synthesized dyes showed that most of the solvatochromism in the hydrazo form is due to the solvent dipolarity/polarizability and solvent basicity rather than solvent acidity. However, the presence of an electron releasing substituent, methoxy group, causes major contribution comes from the solvent hydrogen-bonding donor acidity.

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