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Solvent and substituent effects on spectroscopical changes of some diazoaminobenzene derivatives

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Abstract

A series of diazoaminobenzene derivatives (seven) in which the substituents have a wide range of electronic characters are set out to understand the involvement of the substituent identity in controlling the changes in their electronic absorption spectra. The interactions between the diazoamino group and the different groups account for some spectral shifts. The UV–vis spectrum of each compound is measured in several solvents with wide variations of solvent polarity parameters to examine the role of the chemistry of the solvent in these spectroscopical changes. The electronic transitions are assigned and the solvent induced spectral shifts are analyzed in relation to the different solute–solvent interaction mechanisms using computational chemistry. The regression analysis is applied for correlating the different parameters. The results help to assign the solute–solvent interactions and the solvatochromic potential of the investigated compounds. It is concluded that the electronic character of the substituent and the chemical nature of the solvent are the major factors for the observed solvatochromism.
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1. Introduction

Compounds containing diazoamino group have attracted increasing attention due to their many emerging applications. The derivatives of diazoaminobenzene (DAAB) (1,3-diphenyl-1-triazene, DPT), Fig. 1, are applied in three major use areas. They can be used as a versatile metal complexing agent [1], as an intermediate in several industry sectors including organic synthesis, dye manufacture, and agrochemical manufacture such as insecticides [2–4], and as a polymer additive where DAAB is used as a coupler to promote adhesion of natural rubber to steel [5], and as a chemical blowing agent in a variety of polymer systems [6]. In addition, it has been reported to show semi-conducting properties [7,8]. Oral doses of DAAB were well absorbed, readily metabolized, and excreted predominantly in the urine [9]. The production of metabolites of aniline [10] and benzene [11,12] by human liver slices, as well as by rats and mice, indicated a toxic-carcinogenic potential for DAAB in humans. In this work, derivatives of diazoaminobenzene having both strong electron-donating and -withdrawing substituting moieties are used as probes to examine the effect of molecular structure on their UV–vis spectra, as well as, the solvatochromic behavior of these compounds. A careful analysis of the obtained data using statistical techniques showed that the solvatochromism correlate to the identity of the substituent and the solvent parameters as well.

2. Experimental

The DAAB derivatives were synthesized by diazotizing substituted anilines reactions [4,13,14]. The UV–vis spectra were measured with a Shimadzu UV 160A spectrophotometer. The 11 solvents used were of spectrophotometeric grade.

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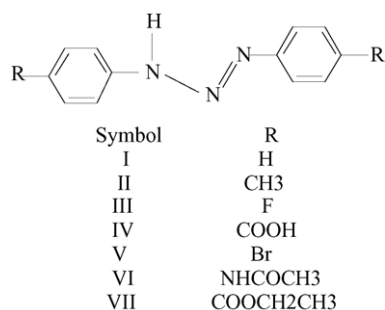 E-mail address: drmsmasoud@yahoo.com (M.S. Masoud).

Fig. 1. Structures and symbols of the investigated compounds.

Table 1
Solvent parameters used in the spectral correlation equations

Solvent	n	d	E	K	M	N
n-Hexane	1.375	1.9	30.9	0.188	0.188	0.00191
Toluene	1.497	2.4	33.9	0.241	0.226	0.026
Diethyl ether	1.353	4.2	34.6	0.340	0.178	0.300
Ethyl acetate	1.372	6.0	38.1	0.385	0.185	0.398
Acetone	1.359	20.7	42.2	0.465	0.180	0.648
DMSO	1.478	48.9	45.0	0.485	0.221	0.658
2-Propanol	1.377	18.3	48.6	0.460	0.187	0.622
Ethanol	1.361	24.3	51.9	0.470	0.181	0.665
Methanol	1.329	32.6	55.5	0.477	0.169	0.710
Water	1.333	78.5	63.1	0.491	0.171	0.757

They were purchased from Sigma and Aldrich and were used as received.

2.1. Regression analysis calculations

Different one-, two- and three-parameter equations are applied using suitable combinations between the solvent polarity parameters E, K, M, J, H and N as reported before [15]. The parameter E is sensitive to both solute–solvent hydrogen bonding and dipolar interactions [16]. The dielectric function, K of Kirkwood adequately represents the dipolar interactions [17]. The functions H and J have been introduced to account for the non-specific solute–solvent interactions, such as dispersion and dipolar effects [18]. The functions M and N account for the solute permanent dipole–solvent induced dipole and solute permanent dipole–solvent permanent dipole interactions, respectively [19]. In the present work, the observed peak position of an absorption band Y in a given solvent has been expressed as a linear function of different solvent polarity parameters X_n, as follows:

$$Y = a + b_1 X_1 + b_2 X_2 + \cdots + b_n X_n \tag{1}$$

Eq. (1) is amenable to solution for the intercept a and the coefficients $b_1, b_2, \ldots, b_n$ by multiple regression technique. The regression intercept a has been considered as the peak position in the gas phase spectra [20]. A program of statistical package of social sciences (SPSS) has been used on a Pentium IV computer at the Faculty of Education in Damanhour, Egypt. The residual error S in a least-squares that fits the multi-linear Eq. (1) is:

$$S = \sum_{i=1}^{m} (Y_i - a - b_1 X_{1i} - b_2 X_{2i} - \cdots - b_n X_{ni})^2 \tag{2}$$

The least-squares criterion require that S is minimized. This result in sets of equations can be solved by use of standard matrix methods.

3. Results and discussion

The observed shifts of the electronic absorption bands of organic compounds induced by solvents are commonly understood as an indication of the extent of charge reorganization of solute molecules upon electronic excitation [21]. A more detailed consideration of solute–solvent interactions within the reaction potential concept should describe solvent shifts in the electronic absorption spectra, as an indication of the extent of influence of inertial solvent reorganization on the instantaneous charge reorganization of solute molecules upon electronic excitation. A variety of approaches to the theoretical treatment of solvent effects have been developed and applied to studies of UV–vis spectroscopic phenomena [22–24]. Diazoaminobenzene derivatives contain intramolecular charge-transfer chromophores and, therefore, the presence of electron-donating and -withdrawing moieties will have appreciable effect on their UV–vis absorption bands. The substituted diazoaminobenzenes (triazenes) studied are 1,3-diphenyl-1-triazene (I), 1,3-bis(*p*-tolyl)-1-triazene (II), 1,3-bis(4-flurophenyl)-1-triazene (III), 1,3-bis(4-carboxylicphenyl)-1-triazene (IV), 1,3-bis(4-bromophenyl)-1-triazene (V), 1,3-bis(4-acetamidophenyl)-1-triazene (VI), 1,3-bis(4-ethylbenzoato)-1-triazene (VII), Fig. 1. The solvatochromic behavior of these triazenes is examined using various solvents. The solvents are selected to have a wide variety of solvent parameters such as dielectric constant, d, refractive index, n, and hydrogen bonding capacity to make comparison easier, and permit a good understanding of solvatochromic behavior. Table 1 shows the values of these solvent parameters [25].

The $\lambda_{\max}$ values of the prepared triazenes in the UV–vis region are given in Table 2. Representative electronic spectra of some of these compounds measured in different solvents are presented in Fig. 2. Triazenes exhibit absorption maxima at the wavelengths 330–400 nm that make them efficient chromophores. Although their color strength does not reach that of azo chromophores [26–31].

This behavior is accounted as that molecules in the ground state and in the excitation state indicate different polarities. This involves the highly simplifying assumptions that triazenes with non-polarized ground state are more strongly polarized in protic solvents, because the high-energy, polar structure of the excitation state is stabilized. The excited state is lowered, the ground state is hardly affected. The energy difference between ground and excited states is decreased and the excitation energy is decreased. The approximation of the

Table 2
The observed λ_{max} values of the investigated diazoaminobenzenes

R	n-C$_6$H$_{14}$	n-C$_5$H$_{12}$	Et$_2$O	(CH$_3$)$_2$CO	EtOAc	C$_6$H$_5$CH$_3$	(CH$_3$)$_2$COH	EtOH	MeOH	DMSO	H$_2$O
H		236	231		249		241	241	236		241
		270	292		313	288				297	
	359	358	367	364	339	370	385	387	382	364	375
CH$_3$		233	239		246		236		237		238
	290	269	297	323	259	293	292	295	295	305	292
	372	352	355	354	359	360	359	362	363	380	364
F		222	244		251		231	236	231		233
	272	264	297		292	288	290	288	292	300	287
	354	349	359	349	349	357	356	360	351	379	349
COOH	236	233	224		246		236	234	221		228
		291		326	300	297	300	301		313	
	351	348	349	349	344	351	359	355	348	367	354
Br		233			308	299	244	241	241	265	251
	298	294			339	334	303	298	297	302	323
	369	358		359	359	361	364	365	357	379	374
			221	218				226	227	223		217
NHCOOH$_3$	236	249			249						
	277	282	282		287	294	288	294	287	300	283
	377	361	365	354	367	366	371	373	369	380	
COOCH$_2$CH$_3$	233	235	249		251			253	251	267	245
		278	270		297	282	310			310	
			361	359	369	360	368	370	370	385	368

The UV–vis absorption spectrum of diazoaminobenzene in n-hexane exhibits one absorption band with λ_{max} at 359 nm, Table 2. This absorption band is assigned to the partly forbidden (n → π^*) transition. This band shows positive solvatochromism (bathochromic shift) upon increasing the solvent polarity, where a pronounced change in position of an electronic absorption band is accompanying a change in the polarity of the medium.

energy levels expresses itself in a bathochromic shift of the spectrum with increasing polarity of the solvent. A negative solvatochromism (hypsochromic shift) is noticed in solvents like ethyl acetate. This non-protic solvent destabilizes the polarized electronic state. This leads to a hypsochromic shifting of the spectrum with decreasing solvents polarity. For diazoaminobenzene derivatives with an electron-withdrawing or donating group on the benzene nucleus, a solvatochromism occurred in the (n → π^*) absorption upon increasing the

push–pull character of the substituents [32]. A solution in n-hexane solvent of triazene, where the substituent is the strong electron donating methyl group can be characterized by an absorption spectrum with an intense transition centered at 372 nm and a weak broad band at 290 nm. The lowest transition absorption band at 372 nm is assigned to (n → π^*) transition while the other absorption band corresponding to the highest energy centered at 290 nm is assigned as (π → σ^*) transition. This high-energy band shows a pos-

Table 3
The results of regression analysis for the high-energy transition of triazene (V)

Parameter	a_0	a_1	a_2	a_3	a_4	P	MCC
K	12.335	495.311				0.288	0.356
M	174.661	396.872				0.088	0.676
N	249.705	−1.913				0.987	0.000
E	275.141	−0.506				0.568	0.120
K, M	−31.126	440.302	375.017			0.044	0.956
K, N	−207.566	1301.956	−241.129			0.051	0.949
K, E	−75.333	−1.177	809.665			0.138	0.862
M, N	58.558	566.527	123.947			0.041	0.959
M, E	34.071	764.100	1.370			0.023	0.977
N, E	147.636	308.958	−2.595			0.400	0.600
K, M, N	1758.076	−8281.418	4149.987	2441.648		0.140	0.988
K, M, E	11.767	1.030	122.448	666.795		0.181	0.980
M, N, E	37.315	714.047	38.572	0.986		0.178	0.980
K, N, E	−299.025	−421.898	0.985	1643.504		0.220	0.970
K, M, N, E	1477.012	−6991.503	3705.347	2014.944	0.759	0.000	1.000

itive solvatochromism (bathochromic shift) in all solvents except ethyl acetate. The degree of the solvatochromic behavior is increased upon increasing the solvent polarity. The $(n \rightarrow \pi^*)$ band shows a negative solvatochromism in all solvents except dimethyl sulfoxide. The spectral shifts decrease with gradual introduction of the more polar solvent. In these solvents the NH group in the diazoaminobenzene moiety will be the proton acceptor center in the process of hydrogen bonds formation, whereas the solvents that have the ability to form hydrogen bonds are the proton donors. In presence of dipolar aprotic media such as ethyl acetate, diethyl ether and dimethyl sulfoxide, the acidic NH group of the diazoaminobenzene moiety act as proton donor. However, these solvents have relatively high dipole moments. Dimethyl sulfoxide differs from the other group of solvents used in that its dielectric constant and its polarizability are appreciably greater than the other solvents. The dipole–dipole interactions with the solute molecules especially in their excited states will encounter for the blue shifts observed in these solvents. The energy of a charge transfer will increase as the strength of such hydrogen bonds increases. The observed marginal shift in the absorption maximum for the azo dyes may arise from contributions other than solvatochromic effects. However, in addition to these shifts, a significant band broadening for the azo compounds with increasing solvent polarity was observed. It is concluded that solvatochromism is a useful indicator of the strength of hydrogen bonding and has even been a good tool to assess their strengths. A positive solvatochromism is observed for the $(n \rightarrow \pi^*)$ absorption in *n*-hexane in case of methyl, acetamido and bromine substitution, whereas a negative solvatochromism is observed for this band in case of fluorine and carboxylic substituted compounds. The bathochromic shifts for this band follows the order $NHCOCH_3 > CH_3 > Br$. The high polarizability of bromine retards its withdrawing character. The hypsochromic shift for this band follows the order $COOH > F$ in the opposite direction of their electronegativities. This is because the involvement of the fluorine atom in intramolecular hydrogen bonding or intermolecular hydrogen bonding with the solvent molecules is more than that of the carboxylic group. Only a negative solvatochromism is observed for all sub-
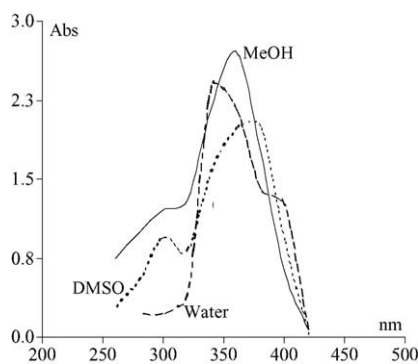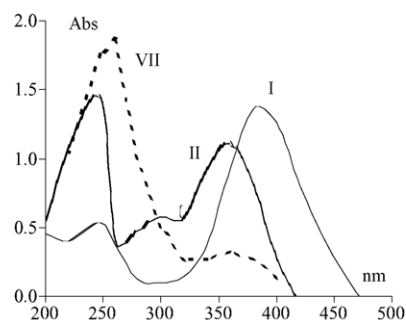
Fig. 3. Electronic spectra of compounds I, II and VII in ethanol.

stituents using solvents have the ability to form hydrogen bonding with the solute molecules like isopropyl alcohol, ethanol, methanol and water. It seems like when there is a strong hydrogen bonding and the push–pull effect is retarded. The opposite trend in the solvatochromic behavior of $(n \rightarrow \pi^*)$ absorption for these compounds is observed in solvents like ethyl acetate and dimethyl sulfoxide. Absorption spectra for the same triazenes with various electron donating or accepting abilities in ethanol are shown in Fig. 3. The UV–vis spectrum of the triazene I in *n*-pentane exhibits three absorption bands. The band at about 358 nm is assigned to a $(n \rightarrow \pi^*)$ transition that is red shifted in all other solvents except ethyl acetate. The same behavior was found for the second absorption band at 270 nm and assigned as $(\pi \rightarrow \sigma^*)$. A third band appears in the spectrum of the parent triazene at 236 nm in *n*-pentane due to localized aromatic rings of $(\pi \rightarrow \pi^*)$ transition and shows a negative solvatochromism in all other solvents and disappears in *n*-hexane, acetone, toluene and dimethyl sulfoxide.

Several one-, two- and three-parameter equations have been used to correlate the spectral shifts with various empirical solvent polarity parameters using the multiple linear regression technique. Each of the solvent parameter used has a fixed relative sensitivity to each of the various interaction mechanisms. Table 3 is a model representative example showing the results of regression analysis for the high-energy transition of triazene (V). The multiple correlation coefficient (MCC), has been used in a one-tail test to obtain the level of significance for each test. The MCC value is considered as a measure of the goodness of the fit. The high value of MCC (near one) means that a certain solvent parameter has a good correlation to the spectral shifts. In other words, the spectral shifts for the peak are greatly sensitive to the solvent parameter that gives a value of MCC near to unity. Alternatively, the small value (near zero) of the significance parameter (*P*) means the correlation is good. The parameters that gave the best correlations among the others for the different electronic transitions of the investigated compounds are shown in Table 4. Also, the parameters that gave the best significance among the other parameters for the different electronic transitions of the investigated compounds are shown in Table 5.

Fig. 2. Representative solvent induced spectral effect for compound (II).

Table 4
The best correlating parameters for the different peaks of all compounds

| Compounds | Electronic transition | | | | | | | | |
| | High-energy transition | | | Moderate-energy transition | | | Low-energy transition | | |
	One-parameter	Two-parameter	Three-parameter	One-parameter	Two-parameter	Three-parameter	One-parameter	Two-parameter	Three-parameter
I	M (0.226)	K, N (0.539)	K, N, E (0.819)	–	–	–	E (0.468)	N, E (0.484)	K, N, E (0.505)
II	–	–	–	N (0.077)	N, E (0.139)	K, N, E (0.224)	M (0.194)	M, E (0.273)	K, M, N (0.607)
III	N (0.714)	K, N (0.728)	K, N, E (0.821)	K (0.351)	K, N (0.814)	K, N, E (0.829)	M (0.426)	M, N (0.557)	M, N, E (0.588)
IV	M (0.624)	K, M (0.681)	K, M, N (0.684)	N (0.268)	N, E (0.708)	K, N, E (0.825)	M (0.228)	M, N (0.580)	K, M, N (0.690)
V	M (0.676)	M, E (0.977)	K, M, N (0.988)	E (0.205)	N, E (0.258)	K, N, E (0.534)	M (0.095)	M, E (0.289)	K, M, N (0.601)
VI	–	–	–	M (0.422)	K, M (0.861)	K, M, N (0.872)	M (0.104)	M, E (0.158)	K, M, N (0.313)
VII	K (0.528)	N, E (0.967)	M, N, E (0.997)	E (0.821)	M, E (0.868)	K, N, E (0.978)	K (0.290)	K, M (0.654)	K, N, E (0.816)

The analysis of the spectral shifts of the high-energy transitions in all of the investigated compounds using one-parameter equation, Table 4, showed that the best MCC values in case of compounds I, IV and V are obtained for the parameter *M* which is sensitive to dipole–dipole interactions. The respective MCC values are 0.226, 0.624, 0.714 and indicate that the solvatochromism for these cases can be interpreted in terms of solute permanent dipole–solvent permanent dipole interactions. However, the correlation in case of compound I is not that good (0.226), and this means that the one solvent polarity parameter, *M* alone can not control the spectral changes and, therefore, a combination of parameters is required. The parameters *N* (0.714) in case of compound III and *K* (0.528) in case of compound VII gave the best correlations to the solvent induced spectral shifts for the high-energy electronic transitions among all of the other parameters. This means that the solute permanent dipole–solvent induced dipole interactions and the dipolar interactions are the major factors affecting the solvatochromism in these cases, respectively. The correlation of the two-parameter equations with the solvent spectral shifts was also studied and gave, as expected, better fit to these spectral shifts than the corresponding one-parameter fits. The solvent ability to form hydrogen bonds with the solute molecules, which is reflected by the parameter *E* when combined with the previously mentioned parameters *K*, *M* or *N* is the reason for improving the correlations. For instance, when the parameter *E* is combined with the parameters

M (compound V) and *N* (compound VII) the MCC values jumped to 0.977 and 0.967, respectively. The correlation is improved as expected on analyzing the spectral shifts using three-parameter equations and the best MCC values are observed (Table 4). The solvatochromic behavior is analyzed using one-parameter equations for the moderate-energy electronic transition of all triazenes. The best MCC value is obtained for the parameter *E* (0.821) in case of compound VII. This means the solvatochromic behavior for this transition in compound VII is mainly controlled by the solvent ability to form hydrogen bonding with the solute molecules. Form the values of MCC for the rest of the compounds (Table 4), it is clear that one solvent parameter can not interpret the solvatochromism for this electronic transition alone. The correlation is improved as expected on analyzing these spectral shifts using two- and three-parameter equations. It concluded that the parameters *N*, *E* and *K* are the major parameters affecting the solvatochromism when coming in combinations. Thus, solute permanent dipole–solvent permanent dipole interactions combined with the solvent hydrogen bonding capacity and/or dipolar interactions are contributing mainly to the observed solvatochromism. On analyzing the solvatochromic behavior of the low-energy transitions in all of the investigated triazenes, the same trend is observed. Different combinations of the *K*, *M*, *N* parameters gave the best correlations. This means that in addition to *K* and *N*, the parameter *M*, which is related to the solvent refractive index and accounts for the solute permanent dipole–solvent

Table 5
The best values of the significance parameters for the different peaks of all compounds

| Compounds | Electronic transition | | | | | | | | |
| | High-energy transition | | | Moderate-energy transition | | | Low-energy transition | | |
	One-parameter	Two-parameter	Three-parameter	One-parameter	Two-parameter	Three-parameter	One-parameter	Two-parameter	Three-parameter
I	M (0.341)	K, N (0.313)	K, N, E (0.259)	–	–	–	E (0.029)	N, E (0.096)	K, N, E (0.210)
II	–	–	–	N (0.437)	N, E (0.592)	K, N, E (0.651)	M (0.203)	M, E (0.327)	K, M, N (0.112)
III	N (0.034)	N, E (0144)	K, N, E (0.256)	K (0.093)	K, N (0.006)	K, N, E (0.023)	M (0.041)	M, N (0.056)	M, N, E (0.127)
IV	M (0.035)	K, M (0.102)	K, M, N (0.271)	N (0.292)	N, E (0.158)	K, N, E (0.251)	M (0.163)	M, N (0.048)	K, M, N (0.057)
V	M (0.088)	M, E (0.023)	K, M, N (0.140)	E (0.307)	N, E (0.550)	K, N, E (0.456)	M (0.420)	M, E (0.359)	K, M, N (0.173)
VI	–	–	–	M (0.058)	K, M (0.003)	K, M, N (0.011)	M (0.397)	N, E (0.568)	K, M, N (0.564)
VII	K (0.064)	N, E (0.001)	K, M, E (0.002)	E (0.034)	M, E (0.132)	K, N, E (0.189)	K (0.135)	K, M (0.042)	K, N, E (0.027)

induced dipole interactions should be involved in the interpretation of the observed solvatochromism. Thus, the determination of the solvent spectral shifts is controlled by mainly the dipolar interactions, as well as, both the solvent dielectric constant and the solvent refractive index. Generally, it is concluded that the addition of a third solvent parameter to the two-parameter equations always gave rise to improvements in the correlation with the solvent induced spectral shifts. In most cases, the different three-parameter combinations have been selected on the basis of the results of the two-parameter combinations discussed before. In a test for the significance of a one-tail test, the level of significance for all these different three-parameter combinations were found to be above 90%. This indicated that specific solute–solvent interactions in particular hydrogen bonding and non-specific solute–solvent interactions such as dispersion and dipolar effects had provided a reasonable model for describing the solvent induced spectral shifts in a predictive manner. The values of the significance parameters in Table 5 reinforce the argument about the correlation analysis for the observed spectroscopical changes observed for the investigated diazoaminobenzene derivatives.

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