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The synthesis, characterization and spectral properties of crown ether based disazo dyes

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Abstract

A series of crown ether based azo dyes (**7a–10**) were obtained by the coupling of crown ether based diazonium chloride with various substituted naphthols (**3a–3f**), pyrazolones (**4a–4d**), acetoacetanilides (**5a–5d**) and barbituric acid (**6**). The synthesized dyes were purified and characterized by elemental analysis, FT-IR, ^{1}H NMR and FAB mass spectra. The absorption properties of the synthesized dyes were studied and their application in solvent based inks was investigated; the fastness properties of formulated inks were also studied.

Keywords: Crown ether disazo dyes; Spectral properties; Solvent effect; Fastness studies

1. Introduction

Azo dyes and pigments constitute the largest and most varied group of synthetic organic colorants in use today. They have characteristically good tinctorial strength as well as stability and their preparation procedures, by the classic diazotization and coupling reactions, are very simple and of low cost. Hence, in addition to their conventional application as colorants for wools, fabrics, prints, inks, and toners, azo compounds have also found their way into high-technology areas, such as colorants for liquid-crystalline materials and as photoconductors for photoreceptors in copiers and laser printers [1,2].

With the increased usage of these azo colorants, there is an increasing demand to modulate their features and obtain colorants having enhanced permanence, solubility and stability. This can be achieved by specific modifications of the dye structure, in particular by increasing the molecular weight of the dyes with bulky organic groups known as ‘‘ballast group’’. Such modifications should, however, not influence the color properties of the dyes. There are various examples of ballast groups like the triazinyl group, carboxylic acid group, sulfone group etc. that are known to increase the solubility and long-term stability of the dyes [3–5].

There have been very few reports wherein macrocycles such as crown ethers and calixarenes have been used as components for the preparation of azo dyes. Calix[*n*]arenes have been mostly reported as the coupling component for various diazotized aromatic amines [6–8] while amino crown ether has been used as the diazo component [9]. More recently, there are examples wherein crown ethers have been covalently linked to azo dyes for improved fastness in water-soluble dyes for ink jet inks [10].

In the present investigation, we explored the possibility of using crown ethers as ballast for azo dyes. Hence a series of crown ether based azo dyes were synthesized by the coupling of crown ether diazonium chloride with various substituted naphthols, pyrazolones, acetoacetanilides and barbituric acid. The various substituted naphthols, pyrazolones, acetoacetanilides and barbituric acid chosen for the present work were on the basis of the fact that these coupling components are easily available and are known to give highly stable and brightly colored dyes [11].

The synthesized dyes were purified, characterized and studied for their application as active coloring agents in solvent based inks. Their spectral and fastness properties were also thoroughly investigated.

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2. Experimental

2.1. General

Compounds 3-hydroxy-*N*-phenyl-2-naphthamide **3a**, 3-methyl-1-phenyl-pyrazole-5-one **4a**, acetoacetanilide **5a** and corresponding derivatives were obtained as gift samples from Trident Colors, Ahmedabad and were purified before use. Barbituric acid **6** was purchased from Loba Chemie and used without purification. All other chemicals and solvents used were of analytical grade and were used without further purification.

Melting points were determined on a Toshniwal (India) melting point apparatus and are uncorrected. Elemental analysis was done on a Carlo Erba 1108 analyzer. FT-IR spectra were recorded on a JASCO 410 FT-IR spectrophotometer as KBr pellets. Visible spectra were recorded on a Hitachi U-3210 UV–visible spectrophotometer, and the FAB mass spectra were recorded on a JEOL SX 102/DA-6000 mass spectrometer/data system using argon/xenon (6 kV, 10 mA) as the FAB gas. The accelerating voltage was 10 kV and the spectra were recorded at room temperature. *m*-Nitro benzyl alcohol (NBA) was used as the matrix and the matrix peaks appeared at *m/z* 136, 137, 154, 289, 307. Proton NMR spectra were recorded on a DRX 300 spectrophotometer operating at 300 MHz in DMSO-d_6 with TMS as the internal standard.

The ink formulations were prepared using a Marshal MV614 vibroshaker. The resultant formulations were applied to polyester films, aluminium foils, coated as well as uncoated paper manually using a barcoater no. 2.

2.2. Synthesis

The synthesis of dibenzo-18-crown-6 [12], nitration to 4,4′-dinitrodibenzo-18-crown-6 [13] and reduction to 4,4′-diaminodibenzo-18-crown-6 [9] was carried out by reported procedures. 4,4′-Diaminodibenzo-18-crown-6 was diazotized by the conventional procedure to obtain the crown ether diazonium chloride **2** [14] which was then coupled with **3a**, **4a**, **5a**, **6** and the corresponding derivatives using reported methods [11].

2.2.1. Synthesis of crown ether diazonium chloride (2)

4,4′-Diaminodibenzo-18-crown-6 (5.0 g, 0.013 mol) was dissolved in concentrated hydrochloric acid (15 ml) and the solution was then cooled to 0–5 °C. Sodium nitrite (2.0 g, 0.029 mol) in water (10 ml) was then added to this solution dropwise with vigorous stirring, for 1 h at 0–5 °C. The excess of nitrous acid was destroyed using sulphamic acid until the solution was negative to starch iodide paper (Fig. 1).

2.2.2. Synthesis of 3-hydroxy-N-phenyl-2-naphthamide based crown dyes (*7a–7f*)

The compound (**3a–3f**) (0.034 mol) was dissolved in 200 ml of 0.2 M sodium hydroxide solution and heated slowly to 90 °C. The resulting clear solution was then cooled to room temperature and a dilute solution of acetic acid was introduced dropwise into the above solution until a fine dispersion of the coupler was obtained. This suspension was then cooled to 0 °C and the diazotized solution was slowly added with stirring. A pH of 6–7 was maintained during the reaction using sodium acetate. Stirring was continued for about 3–4 h at 0 °C and the solution was gently heated to 90 °C. The precipitated dye was filtered and recrystallized in methanol (Fig. 2).

2.2.3. Synthesis of 5-methyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one and barbituric acid based crown dyes (*8a–8d* and *10*)

The coupler (**4a–4d** and **6**) (0.034 mol) was dissolved in 100 ml water containing sodium hydroxide (3 g, 0.075 mol) and 5 g of sodium acetate. The solution was cooled to 0 °C and the diazonium solution was added slowly. The pH during the reaction was maintained between 9 and 12. The stirring was continued for 4–6 h and then heated to boiling. The dye was collected by filtration and recrystallized in methanol (Fig. 2).

2.2.4. Synthesis of 3-oxo-N-phenylbutanamide based crown dyes (*9a–9c*)

The diazo solution was neutralized with sodium acetate to the point where Congo red paper just turned violet.

The compound (**5a–5d**) (0.034 mol) was dissolved in 50 ml water containing sodium hydroxide (1.36 g, 0.034 mol) to form a clear solution. This solution was cooled and 7 g of sodium acetate was added. Dilute acetic acid was then slowly added until a faintly acid reaction to litmus was produced but no precipitation occurs. The diazo solution was then added and the reaction continued for 10 h. The dye was collected by filtration (Fig. 2).

2.3. General procedure for spectral studies and ionization constants (pK)

For the spectral studies $\sim 10^{-4}$–10^{-5} M dye solutions in various solvents were used and the effect of acid and base was studied by addition of 0.1 M sodium hydroxide and 50% acetic acid, respectively.

For pK_a measurements $\sim 10^{-4}$–10^{-5} M (methanol) dye solutions in the pH range of 2–13 were made and the spectra were recorded at 25 °C in the range of 300–650 nm. The ionization constants were then calculated using the following equation:

$$\mathrm{p}K_\mathrm{a} = \mathrm{pH} + \log_{10}\frac{A - A_\mathrm{b}}{A_\mathrm{a} - A}$$

where A is the absorbance at the specific pH, A_a and A_b are the absorbances for the most acidic and most basic pH, respectively [15].

2.4. Preparation of inks

The synthesized dyes were used for formulating solvent based inks [16]. To a mixture of 6 g dye, 25 g resin (maleic resin for paper, acrylic resin for polyester film and nitrocellulose resin for aluminium foil) and 60 g of appropriate solvent (ethanol for paper, ethyl acetate for polyester films, glycol ether and ethyl acetate mixture for aluminium foils) in a vibroshaker

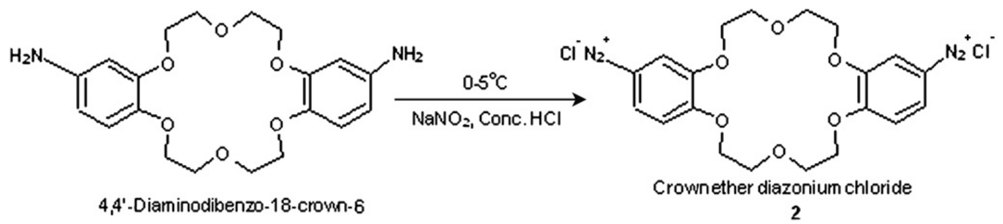

Fig. 1. Synthesis of crown ether diazonium chloride.

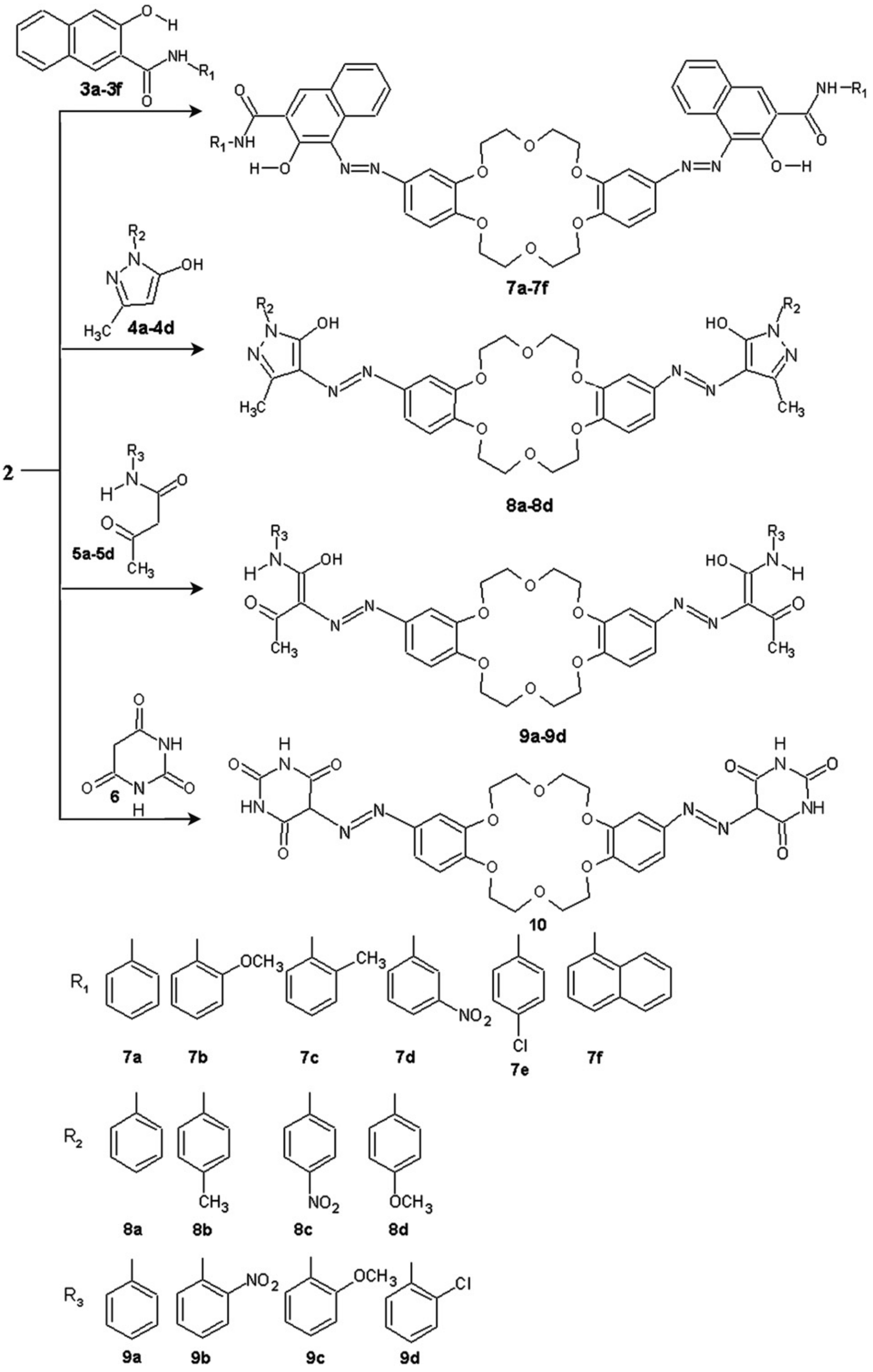

Fig. 2. Synthetic scheme for the dyes **7a–10**.

container was added 80 g glass beads and the mixture was shaken for 20 min.

The inks were then transferred to their corresponding substrates manually using a barcoater rod no. 2, and then allowed to dry for 3–5 min at 35 °C. The light and wet fastness studies were done according to standard method [17].

3. Results and discussion

3.1. Characterizations

In the present work 15 new crown azo compounds were synthesized and characterized using elemental analysis, FT-IR, ^{1}H NMR and FAB mass spectra (Tables 1 and 2). The appearance of the azo band at 1514–1610 cm^{-1} confirms that all the dyes contain the azo group in the solid state while the appearance of the –OH bands at a much lower value 3450–3472 cm^{-1} for dyes **7a–7f**, **8a–8d** and **9a–9d** indicates the presence of strong intramolecular hydrogen bonding. The presence of three carbonyl bands and the absence of the –OH band in the FT-IR spectra of dye **10** suggest that it is in the azo–triketo form in the solid state.

The –OH and –NH peaks observed in the ^{1}H NMR were very broad and diminished in nature. In fact the enolic –OH proton peak for dyes **8a–8d** and –NH for dye **10** (observed in the FT-IR) were not at all observed in the NMR spectra.

3.2. Thermal properties

The melting points and decomposition temperatures of all the synthesized dyes are given in Table 2. It was observed that the dyes prepared from low melting components like **5a** (mp. 84–86 °C) showed low thermal stability and those prepared from high melting components like **6** (mp. 250–252 °C) showed high thermal stability.

3.3. Proton ionization constants

The ionization constants are a core property of a molecule which reflects its structural characteristics, reactivity, spectral properties and the isolation conditions. From the values of the ionization constants summarized in Table 3 it can be easily concluded that dye **10** is the most acidic in nature. This implies directly that of all the dyes synthesized, dye **10** has more tendency to be taken up in an aqueous solution than into organic solvents. Hence it will have comparatively less solvent solubility and water fastness than the other dyes.

3.4. Absorption spectra

The spectral details of the synthesized crown dyes are summarized in Table 3. The dyes showed colors ranging from deep red ($\lambda_{\max}$ 546 nm) to yellow ($\lambda_{\max}$ 384 nm). The different substituents are in positions slightly away from the chromophoric systems. Hence they may not directly influence the absorption maxima. But the general observation made is that most of the substituted dyes have higher absorption maxima when compared to their unsubstituted analogues.

The molar extinction coefficient ($\varepsilon_{\max}$) of the dyes showed that most of them are intensely absorbing. However, it is the oscillator strength (f) that gives a full measure of the tinctorial strength rather than molar extinction coefficient ($\varepsilon_{\max}$) [2,18].

Table 1
Spectral data for the synthesized dyes

Dye	IR (cm^{-1})					^{1}H NMR (δ, ppm)				
	$\nu_{-\mathrm{OH}}$	$\nu_{-\mathrm{NH}}$	$\nu_{-\mathrm{N=N}-}$	$\nu_{-\mathrm{C=O}}$	$\nu_{-\mathrm{NO_2}}$	–OCH$_2$	Ar–H	–OH	–NH	–OCH$_3$ and –CH$_3$
7a	3450	3242	1558	1634	–	3.5–4.2 (16H, m)	7.0–8.7 (26H, m)	10.7 (2H, b)	11.5 (2H, b)	–
7b	3454	3230	1578	1622	–	3.8–4.3 (16H, m)	6.8–8.9 (24H, m)	9.8 (2H, b)	11.2 (2H, b)	3.2 (6H, OCH$_3$, s)
7c	3458	3241	1608	1632	–	3.6–4.5 (16H, m)	7.3–8.2 (24H, m)	10.1 (2H, b)	11.7 (2H, b)	2.2 (6H, CH$_3$, s)
7d	3466	3253	1560	1641	1518, 1388	3.3–4.7 (16H, m)	7.2–8.8 (24H, m)	11.0 (2H, b)	11.8 (2H, b)	–
7e	3458	3250	1610	1627	–	3.1–4.0 (16H, m)	6.8–8.2 (24H, m)	10.3 (2H, b)	11.2 (2H, b)	–
7f	3471	3244	1562	1621	–	3.2–4.5 (16H, m)	7.2–8.9 (30H, m)	10.3 (2H, b)	11.4 (2H, b)	–
8a	3457	–	1552	–	–	3.4–4.1 (16H, m)	6.8–7.3 (16H, m)	–	–	2.4 (6H, CH$_3$, s)
8b	3456	–	1558	–	–	3.9–4.2 (16H, m)	7.1–7.9 (14H, m)	–	–	2.5, 2.1 (6H and 6H, CH$_3$, s)
8c	3457	–	1561	–	1525, 1367	3.5–4.1 (16H, m)	7.2–8.3 (14H, m)	–	–	2.4 (6H, CH$_3$, s)
8d	3461	–	1555	–	–	3.4–4.4 (16H, m)	6.6–7.9 (14H, m)	–	–	2.5, 3.3 (6H, CH$_3$, s; 6H, OCH$_3$, s)
9a	3470	3240	1520	1697	–	3.7–4.3 (16H, m)	7.3–8.5 (16H, m)	13.3 (2H, b)	14.8 (2H, b)	(6H, CH$_3$, s)
9b	3468	3239	1523	1669	1519, 1370	3.7–4.3 (16H, m)	7.6–8.7 (14H, m)	12.9 (2H, b)	14.6 (2H, b)	2.3 (6H, CH$_3$, s)
9c	3466	3238	1534	1692	–	3.7–4.3 (16H, m)	7.3–8.4 (14H, m)	13.2 (2H, b)	13.9 (2H, b)	2.3 (6H, CH$_3$, s)
9d	3472	3235	1525	1689	–	3.7–4.3 (16H, m)	7.1–8.2 (14H, m)	12.7 (2H, b)	13.8 (2H, b)	2.5, 3.7 (6H, CH$_3$, s; 6H, OCH$_3$, s)
10	–	3244	1514	1756, 1704, 1655	–	3.6–4.1 (16H, m)	6.6–7.4 (6H, m)	–	–	–

Table 2
Physical characteristics of the dyes

Dye	Molecular formula	FAB-mass	Color	% Yield	%C		%H		%N		Melting point (decom. temp.) (°C)
					Calc.	Found	Calc.	Found	Calc.	Found	
7a	$C_{54}H_{46}N_6O_{10}$	939 (M + 1)	Red	86	69.07	69.01	4.94	4.72	8.95	8.85	178(>200)
7b	$C_{56}H_{50}N_6O_{12}$	983 (M + 1)	Red	86	68.43	68.01	5.04	5.32	8.41	8.75	175(>200)
7c	$C_{56}H_{50}N_6O_{10}$	967 (M + 1)	Red	87	69.55	69.77	5.21	5.33	8.69	8.58	180(>200)
7d	$C_{54}H_{44}N_8O_{14}$	1029 (M + 1)	Red	82	63.03	63.02	4.31	4.69	10.89	10.54	169(>200)
7e	$C_{54}H_{44}Cl_2N_6O_{10}$	1009 (M + 2)	Red	89	64.35	64.21	4.40	4.51	8.34	8.21	170(>200)
7f	$C_{62}H_{50}N_6O_{10}$	1039 (M + 1)	Red	87	71.66	71.51	4.85	4.72	8.09	7.99	173(>200)
8a	$C_{40}H_{40}N_8O_8$	784 (M + 1)	Orange	86	61.22	61.01	5.30	5.22	14.73	14.85	182(>200)
8b	$C_{42}H_{44}N_8O_8$	789 (M + 1)	Orange	88	63.95	63.81	5.62	5.55	14.20	14.11	181(>200)
8c	$C_{40}H_{38}N_{10}O_{12}$	851 (M + 1)	Orange	88	56.47	56.39	4.50	4.37	16.46	16.42	190(>200)
8d	$C_{42}H_{44}N_8O_{10}$	821 (M + 1)	Orange	86	61.45	61.28	5.40	5.33	13.65	13.51	187(>200)
9a	$C_{40}H_{42}N_6O_{10}$	767 (M + 1)	Bright yellow	84	62.65	62.10	5.52	5.02	10.96	10.85	140(>180)
9b	$C_{40}H_{40}N_8O_{14}$	857 (M + 1)	Bright yellow	89	56.07	55.89	4.71	4.56	13.08	12.88	150(>180)
9c	$C_{42}H_{46}N_6O_{12}$	827 (M + 1)	Bright yellow	87	61.01	60.95	5.61	5.45	10.16	10.20	147(>180)
9d	$C_{40}H_{40}Cl_2N_6O_{10}$	836 (M + 2)	Bright yellow	85	57.55	57.34	4.82	4.77	10.06	10.01	151(>180)
10	$C_{28}H_{28}N_8O_{12}$	691 (M + 23)	Yellow	90	50.29	50.01	4.22	4.70	16.76	16.85	264(>300)

Hence, the oscillator strength was calculated using the following equation:

$$f = 4.32 \times 10^{-9} \times \Delta\nu_{1/2} \times \varepsilon_{\max} \qquad (1)$$

The full width at half maximum ($\Delta\nu_{1/2}$) of the dyes were determined from the plot of molar extinction coefficient (ε) versus wave number (ν). It is evident from the values that the naphthol based crown ether dyes **7a–7f** having high half-band widths and molar extinction coefficients will give bright hues and high tinctorial strengths while the barbituric acid based dye **10** is tinctorially a very weak dye.

3.4.1. Solvatochromic effects

Generally, the ground state for almost all molecules is less polar than the excited state so that a polar solvent will tend to stabilize the excited state more than the ground state. Hence with increase in polarity of the solvent a bathochromic shift is observed in the absorption maximum, which is termed ‘positive solvatochromism’. The solvatochromic effects of synthesized dyes are shown in Table 4. It is clear that all synthesized dyes except **10** exhibit positive solvatochromism in accordance with most chromogens so that the absorption bands of dyes move towards longer wavelengths as the polarity of the solvent increases [19]. As observed in dyes **7a–7f**, going from the DMSO to DMF causes only a maximum of 4 nm change in the absorption maxima of the dyes. We may conclude that probably there is not much change in the stability of the excited state in going from DMSO to DMF. Hence in some cases the absorption maxima remains the same. In case of dyes **9a** and **8a** also we may say that the stabilization is less in going from ethyl acetate to methanol.

In case of dye **10** due to the presence of the nonbonding electron pairs of the carbonyl oxygen and nitrogen atoms in the barbituric acid ring, it is capable of strong n–π* transitions. In polar protic solvents (DMF) the lone pair of electron is engaged in hydrogen bonding and the promotion of these electrons to a π* orbital requires energy to weaken or break the hydrogen bond in addition to the normal transition energy. This results

Table 3
Oscillator strength and ionization constants for the dyes

Dye	$\lambda_{\max}$ (nm) (DMF)	$\Delta\nu_{1/2}$ (cm^{-1})	$\varepsilon_{\max}$, (l mol^{-1} cm^{-1})	f	Ionization constants
7a	528	6225	30,553	0.822	10.36
7b	536	6225	31,251	0.840	10.54
7c	530	6220	30,950	0.832	10.52
7d	527	6223	31,632	0.850	10.32
7e	534	6224	31,340	0.843	10.28
7f	546	6225	31,900	0.858	10.39
8a	407	4610	37,527	0.747	7.32
8b	395	4612	37,530	0.748	7.34
8c	415	4618	37,533	0.749	7.34
8d	410	4615	37,529	0.748	7.31
9a	384	4730	30275	0.619	8.64
9b	390	4734	30,272	0.619	8.59
9c	389	4729	30,277	0.618	8.65
9d	385	4729	30,271	0.618	8.62
10	395	3934	32,080	0.545	6.85

Table 4
Solvatochromic properties of the dyes

Dye	$\lambda_{\max}$ (nm)				
	DMSO	DMF	Ethyl acetate	Methanol	Chloroform
7a	529	528	518	514	510
7b	536	536	524	516	513
7c	533	530	519	512	509
7d	530	527	515	514	510
7e	537	534	523	520	514
7f	550	546	538	535	533
8a	415	407	405	405	403
8b	410	395	390	391	387
8c	421	415	410	408	392
8d	420	410	406	404	403
9a	391	384	383	383	380
9b	395	390	387	384	382
9c	392	389	387	383	379
9d	392	385	383	381	376
10	396	394	396	398	409

Table 5
Effect of acid and base on the dyes

Dye	λ_{max} (nm)			
	Methanol	Methanol + 0.1 M sodium hydroxide	Methanol + 50% acetic acid	Methanol + 1 M H_2SO_4
7a	514	426, 510 (s)	514	526
7b	516	430, 514 (s)	514	531
7c	512	424, 511 (s)	510	526
7d	514	425, 517 (s)	515	524
7e	520	431, 516 (s)	518	530
7f	535	442, 536 (s)	532	548
8a	405	370, 407 (s)	405	421
8b	391	376, 392 (s)	393	402
8c	408	377, 407 (s)	406	420
8d	404	373, 406 (s)	407	414
9a	383	360, 386 (s)	390	395
9b	384	368, 383 (s)	382	394
9c	383	365, 387 (s)	383	396
9d	381	364, 382 (s)	379	392
10	400	361,401 (s)	400	411

s = shoulder.

in absorption at shorter wavelength or rather a blue shift in going from polar to non-polar solvents [20].

But there is a slight increase (bathochromic shift) in going from DMF to DMSO; this may be due to the inability of DMSO to form hydrogen bonding with the dye. Thus the ease in electronic transition causes the absorption maxima to shift to longer wavelength.

3.4.2. *Effect of acid and base*

The effect of acid and base on the absorption of the dye solutions was investigated and the results are shown in Table 5. With the addition of base (0.1 M sodium hydroxide), the λ_{max} of the dyes showed a hypsochromic shift along with a shoulder at longer wavelength.

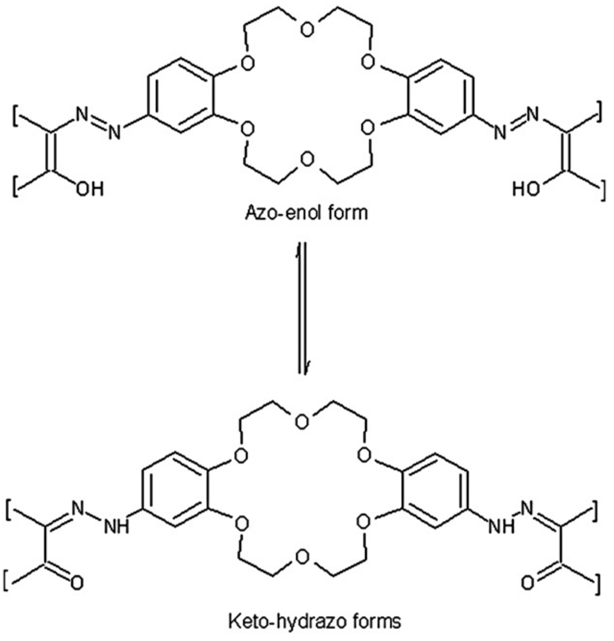

Fig. 3. Tautomeric forms of the synthesized dyes.

Table 6
Fastness studies of the dyes

Dye	Uncoated paper		Coated paper	
	Light fastness	Wet fastness	Light fastness	Wet fastness
7a	6	4–5	3–4	4–5
7b	5–6	4–5	3–4	4–5
7c	5–6	4–5	3–4	4–5
7d	6	4–5	3–4	4–5
7e	6	4–5	3–4	4–5
7f	6	4–5	3–4	4–5
8a	5–6	5	4	5
8b	5–6	5	4	5
8c	5–6	5	4	5
8d	5–6	5	4	5
9a	6	4–5	4–5	4–5
9b	6	4–5	4–5	4–5
9c	6	4–5	5	4–5
9d	6	4–5	4–5	4–5
10	3–4	3	2–3	3

The addition of acid (50% acetic acid) caused no significant shift in the absorption spectra of the dyes. But on addition of a stronger acid (1 M H_2SO_4) a bathochromic shift was observed. Hence, we may conclude that protonation causes a displacement of the absorption band to a higher wavelength and the dyes show a positive halochromic effect.

This clearly indicates that the dyes exist in a mixture of tautomeric forms (azo/hydrazone) and in basic media with the azo form being predominant while in the acidic media the hydrazo form being predominant (Fig. 3) [21].

3.5. *Fastness properties*

The light fastness of the dyes on all substrates except uncoated paper was in the range of 3–4 while the water fastnesses on all the substrates were in the range of 4–5. The light fastness on uncoated paper and coated paper is summarized in Table 6. The dyes show promising results for uncoated paper. This may be due to hydrogen bonding between the free hydroxyl groups in cellulose of paper with the oxygen on the crown ethers [10] as shown in Fig. 4.

4. Conclusions

A series of novel disazo dyes derived from dibenzo-18-crown-6 were synthesized using the crown ether diazonium

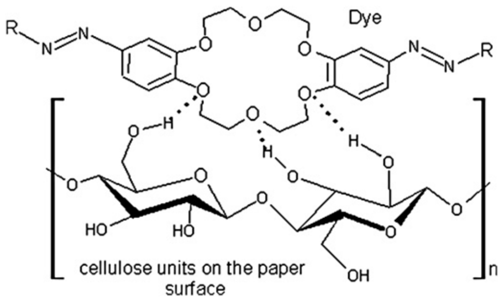

Fig. 4. Adhesion of the dyes to the paper surface.

chloride and various couplers. The dyes were all stable upto 200 °C and hence can be effectively used in almost all kinds of inks. The dyes also had good acid resistance and high tinctorial value. The fastness properties of the inks formulated showed that the synthesized dyes are suitable in inks for uncoated paper.

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