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Spectrochimica Acta Part A 68 (2007) 757–762

SPECTROCHIMICA ACTA PART A

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Reversal in solvatochromism in some novel styrylpyridinium dyes having a hydrophobic cleft

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Received 30 September 2006; received in revised form 22 December 2006; accepted 22 December 2006

Abstract

The influence of solvent polarity on the electronic transition of four different *N*-hexadecyl styrylpyridinium dyes has been investigated in 15 solvents. The $E_T(30)$ scale has been used to propose a quantitative approach towards the relative stability of the electronic ground and excited state species. The extents of contribution of dipolar aprotic solvents towards the solvation of the excited species have been determined to be 42–48% for some of the dyes. Instead of a steady solvatochromism, all the dyes suffer a reversal in solvatochromism. The transitions of the solvatochromism, referred to as solvatochromic switches, are found to be at $E_T(30)$ values of ∼50 for methyl and *N*,*N*-dimethylamino substituted dyes while at 37.6 for hydroxyl substituted dye and ∼45 for 4-(1-methyl-2-phenylethenyl) pyridinium dye. A reversal in the trend of solvent effect in the later dye corresponding to 4-(4-methyl styryl)pyridinium dye has been attributed to an analogy of series and parallel electron flow.

Keywords: Styrylpyridinium dyes; Solvatochromism; $E_T(30)$ switch; Dipolar aprotic solvents; Nonpolar solvents; Hydroxylic solvents

1. Introduction

Solvatochromism represents the solvent-induced spectral band shift, which is due to changes in the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition energy because of differential solvation of ground and excited states of the probe. With increasing polarity of the solvent the spectral band can undergo a hypsochromic or a bathochromic shift depending on the electronic structure of the probe and its interaction with the environment given by the solvent shell. Accordingly, a negative or positive solvatochromism can be found, which gives qualitative and quantitative conclusions about the energetics of the dye solvation. Solvatochromic dyes generally exhibit steady bathochromic (positive solvatochromism) or hypsochromic (negative solvatochromism) band shifts in solvents with a change in polarity. Hydroxy styrylpyridinium dyes having solvatochromic characteristics exhibit a large change in dipole moment upon excitation due to the relative contribution of both the dipolar zwitterionic benzenoid and the neutral quinoid form [1–3]. The solvatochromism is explained through differential solvation of the ground and Franck–Condon excited state [4]. If the ground state is more stabilized than the excited state due to solvation by solvents of increasing polarity, a negative solvatochromism is exhibited and vice versa. The influence of solvent polarity on the preferential solvation of the solute due to differential solute–solvent interaction has also been proposed for diazoaminobenzene [5] and substituted naphthalimide [6].

Brooker et al. [7] carried out pioneering work on solvatochromic merocyanine dyes. Interestingly, some of the merocyanine dyes such as **1**, show an inverted solvatochromism [8], i.e., their long-wavelength absorption band first shows a bathochromic shift followed by a hypsochromic shift with increasing solvent polarity. This reversal in solvatochromism has been rationalized by semiempirical MO calculations [8,9] and the results were confirmed by Jacques [10]. From the transition energy calculations of **1**, Luzhkov and Warshel [11] have reported negative solvatochromism, whereas Morley [12] has shown a positive solvatochromism for it by the CNDOVS method.

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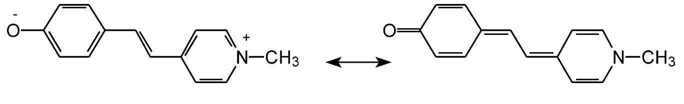
doi:10.1016/j.saa.2006.12.057



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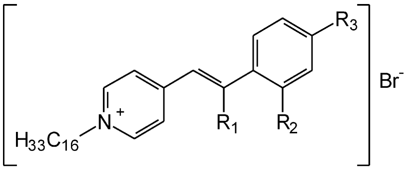
Recently, Masternak et al. [13] have reported about the behaviour of a betaine dye derived from purine and predicted a lower-energy absorption band of charge-transfer character that is highly solvatochromic, and a higher-energy absorption band with π–π^* character which is not solvatochromic. Cavalli et al. [14] studied the fluorosolvatochromism of Brooker's merocyanine in pure and mixed solvents and proposed a model based on solvent-exchange equilibria, which allowed a separation of the different contributions of the solvent species in the solvation shell of the dye.

Earlier, we have reported a reversal in the sign of solvatochromism for a series of bischromophoric styrylpyridinium dyes with hydroxy group in the styryl unit. The reversal has been ascribed to a change in the hydrogen-bonding property of the solvents. Hydrogen-bond donor (HBD) solvents exhibit a negative solvatochromism, while the hydrogen-bond acceptor (HBA) solvents show a positive solvatochromism with increasing solvent polarity [15]. The reversion point has been considered as a switch in the $E_T(30)$ scale to identify the HBD and HBA characteristics of the solvents. To generalize the phenomenon, herein, we have made an attempt to synthesize and to analyze the influence of solvent of different polarity on the electronic transition of some *N*-hexadecyl (substituted styryl)pyridinium bromides (**2**–**5**). The absorption behaviour of **3** in aqueous and micellar media has been studied extensively and aggregations of the dye have been observed in aqueous and surfactant systems [16–22]. In aqueous medium **3** absorbs around 390–400 nm due to H-type of aggregation [17]. In cetyltrimenthylammonium bromide (CTAB) [18], sodium dodecyl sulphate (SDS) [19] and triton-X-100 (TX100) [20] micellar system it exists as monomer. However, in linear alkylbenzene sulfonate (LABS) the compound stacks with a slippage of one chromophoric unit over other leading towards J-aggregate and so exhibits bathochromic shift [21,22]. Similar phenomena has also been observed in CTAB-chloroform reversed micelle, wherein **3** exists as J-aggregates at the interface of water core and mesophase. In nonaqueous solvents the compound exists as monomer [17].



	R_1	R_2	R_3
2:	H	H	CH_3
3:	H	H	$N(CH_3)_2$
4:	H	OH	H
5:	CH_3	H	H

2. Experimental

2.1. Materials

The melting points of the compounds were recorded in open capillary in an acid bath and are uncorrected. All the solvents were analytical grade obtained from SRL and Merck, Mumbai. The solvents were distilled before use.

2.2. Synthesis of 1-n-hexadecyl-4-(4-methylstyryl) pyridinium bromide (2)

An ethanolic solution (20 mL) of *N*-hexadecyl-4-methylpyridinium bromide (0.01 mol, 0.4 g), 4-tolualdehyde (0.01 mol, 0.09 mL), and two drops of piperidine was refluxed for 5 h. The volume of the solution was reduced under vacuum to a pasty mass, which was then washed several times with anhydrous diethyl ether, and the solid thus obtained was recrystallized from ethanol to give a yellow crystalline solid. Yield 78%, mp 141 °C. ^{1}H NMR ($CDCl_3$): $\delta = 0.87$ (t, 3H of hexadecyl) 1.24 (distorted s, 26H of hexadecyl) 1.98 (q, β–CH_2– of hexadecyl) 4.77 (t, α–CH_2– of hexadecyl) 7.26 (d, Bz H), 7.53 (d, Bz H), 7.1 (d, –CH=), 7.68 (d, =CH–, $J = 15.9$ Hz), 8.02 (d, Py H), 9.12 (d, Py H), 2.401 (s, 3H on Bz).

Using this procedure the following compounds were synthesized.

2.2.1. 1-n-Hexadecyl-4-[4-N,N-(dimethylamino)styryl] pyridinium bromide (3)

Yield 78%, mp 220 °C. ^{1}H NMR ($CDCl_3$): $\delta = 0.87$ (t, 3H of hexadecyl), 1.24 (distorted s, 26H of hexadecyl), 4.63 (q, β–CH_2– of hexadecyl), 3.06 (s, 6H), 4.63 (t, α–CH_2– of hexadecyl, $J = 7.2$ Hz), 6.85 (d, –CH= of methane, $J = 15.9$ Hz), 7.52 (d, =CH– of methane, $J = 15.9$ Hz), 6.68 (d, Bz H), 7.52 (d, Bz H, $J = 8.8$ Hz), 7.88 (d, Py H), 8.94 (d, Py H, $J = 6.6$ Hz).

2.2.2. 1-n-Hexadecyl-4-(2-hydroxystyryl)pyridinium bromide (4)

Yield 70%, mp 151 °C. ^{1}H NMR ($CDCl_3$): $\delta = 0.87$ (t, 3H of hexadecyl), 1.24 (distorted s, 26H of hexadecyl), 4.63 (q, β–CH_2– of hexadecyl), 3.06 (s, 6H), 4.63 (t, α–CH2– of hexadecyl, $J = 7.2$ Hz), 6.85 (d, –CH= of methane), 7.52 (d, =CH– of methane, $J = 15.9$ Hz), 6.68 (d, Bz H), 7.52 (d, Bz H, $J = 8.8$ Hz), 7.88 (d, Py H), 8.94 (d, Py H, $J = 6.6$ Hz).

2.2.3. 1-n-Hexadecyl-4-[(2′-methyl)styryl]pyridinium bromide (5)

Yield 70%, mp 153 °C. ^{1}H NMR ($CDCl_3$): $\delta = 0.87$ (t, 3H of hexadecyl), 1.24 (distorted s, 26H of hexadecyl), 2.00 (q, β–CH_2– of hexadecyl), 3.14 (s, 6H), 4.89 (t, α–CH_2– of hexadecyl), 7.28 (s, 1H, –CH=), 2.68 (s, 3H on =C–), 9.17 (d, 4H, $J = 6.3$) 7.91 (d, 4H $J = 5.7$) 7.40 (s, 1H).

2.3. Methods

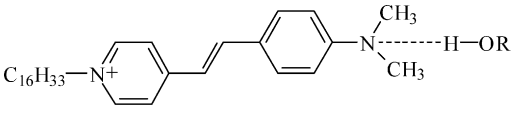
The UV–vis absorption spectra were recorded on a Lambda-25 Perkin-Elmer spectrophotometer in a matched pair of quartz cells of 1 cm thickness with thermostatic facilities to maintain the solution temperature at 300 ± 0.1 K by using a Fourtech cryostat.

3. Results and discussion

3.1. *Substituent-effect on the electronic transition of the dyes*

The styrylpyridinium dyes (**2–5**) have three different substituents, which can influence the electronic transition of the dyes. Each dye has a quaternary nitrogen acceptor at one terminus of the chromophoric group and thus assumes a vectorial flow of electron from the substituent present in the styryl unit. The absorption maxima have been obtained in 15 different solvents having HBD, HBA abilities and rest with non-hydrogen bonding characteristics (Table 1). In these solvents dye **2** with a methyl substituent absorbs within a solvent dependent range of $\lambda_{max} = 354–365$ nm, where as for dye **3** with a *N*,*N*-dimethyl amino substituent the variation in absorption maxima between the highest and lowest value of absorption maxima due to change in solvent ($\Delta\lambda_{max}$) is within 33 nm. However, dye **4** with a hydroxy group shows a variation of $\Delta\lambda_{max} = 55$ nm. The trend in the variation of the absorption maxima is in accordance with the electron-releasing ability of the substituent, R^2 of the linear plot of $\Delta\lambda_{max}$ versus σ_I (Taft's inductive parameter [23]) being 0.96 and those of plots of λ_{max} versus σ_I in each solvent being 0.92 ± 0.2.

For (**3**) with a NMe_2 substituent the λ_{max} is higher for HBD solvents when compared to HBA solvents, whereas for (**4**) with a hydroxy group, the reverse is the case. This may be attributed to the differential stabilization of the ground state and Frank Condon excited state of the hydrogen bonded structures (**6**) and (**7**) of dyes **3** and **4**, respectively.



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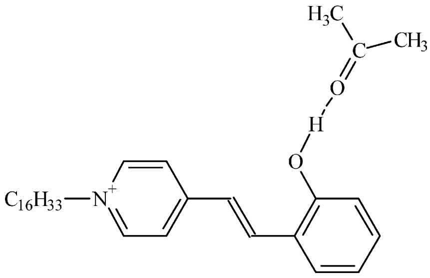

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Table 1
Absorption maxima (λ_{max}) of dyes **2–5** in different solvents

Solvents	λ_{max} (nm)			
	2	**3**	**4**	**5**
Hexane	355.4	454.6	530.1	398.5
Carbon tetrachloride	359.1	467.2	548.4	399.5
Toluene	363.2	474.8	554.2	393.5
Benzene	360.2	476.0	553.7	386.8
Dioxane	354.0	463.0	558.2	387.4
Chloroform	370.0	493.0	554.2	384.0
Dichloromethane	376.0	515.2	553.8	386.4
Acetone	360.0	477.0	547.0	383.2
DMF	360.6	475.3	549.1	384.4
DMSO	361.1	479.0	523.4	384.6
2-Propanol	363.8	481.2	520.4	385.4
1-Butanol	364.8	487.1	523.4	390.5
1-Propanol	364.7	485.6	518.9	398.5
Ethanol	364.0	484.9	518.9	399.5
Methanol	360.0	480.2	503.8	400.6

Further, the polarity change in each class of solvent has also an adverse effect. With increasing polarity the λ_{max} decreases, which envisages the stability of the ground state species in polar medium. Dye **2** with a methyl group does not experience such type of solvent effect. The influence of substituent on the λ_{max} values of **2–5** maintains a trend with that of Hammett substituent constant. The presence of a methyl group in between the electron delocalisation path of one terminus to the other in dye **5** has a significant effect on the λ_{max} value. The compound suffers a bathochromic shift to an extent of $\Delta\lambda_{max} = 20–40$ nm, which may be due to the proximity of methyl group (with +I effect) to the acceptor group.

To have an overall picture of the effect of substituent on the solvent effect, the absorption maxima of all the four compounds were compared through scattergrams (Figs. 1–3). The linearity of the plot in Fig. 1 reveals that with the change of solvents neither the solvation pattern nor the solvated structure changes

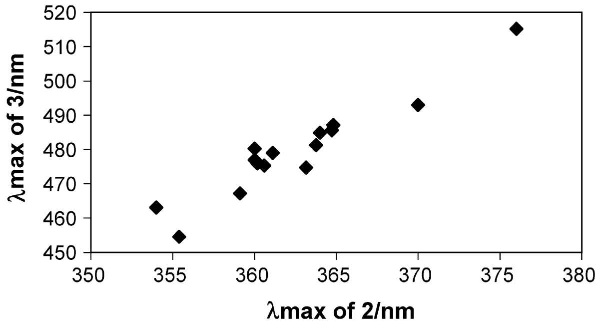


Fig. 1. Plot of UV–vis absorption maxima of **2** vs. those of **3**.

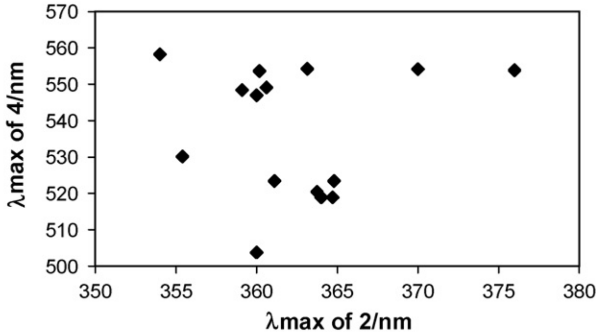


Fig. 2. Plot of UV–vis absorption maxima of **2** vs. those of **4**.

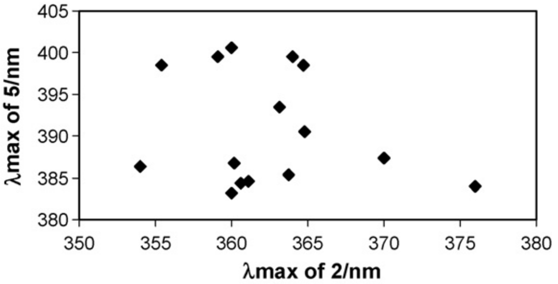
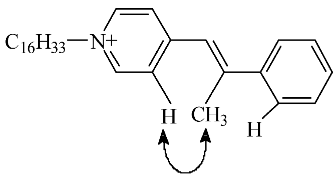


Fig. 3. Plot of UV–vis absorption maxima of **2** vs. those of **5**.

for dyes **2** and **3**. The colonies of points in Fig. 2 may propose a difference in structure of **4** in presence of solvents. Dye **4** having a hydroxyl substituent, assumes a structural change due to the presence of hydrogen bond donor–acceptor solvents. In the presence of these solvents the dye can have some extent of merocyanine characteristics and thus the trend in the absorption maxima changes with respect to dye **2**. However, Fig. 3 produces a scattergram of the relationship of absorption maxima of **2** and **5** in different solvents. This result may be attributed to a differential solvation of the dye **5**. The dye **5** with two electron-releasing groups (phenyl and methyl) connected in parallel does not have similar characteristics, when those are connected serially (**2**). Thus the vectorial electron flow from both the electron-releasing groups may have different type of solvation effect on the absorption maxima. Further, the chromophoric group may deviate from planarity due to the steric interaction between the methyl group and 3-H of pyridinium nucleus (**8**).



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3.2. *Quantitative structure-solvent-spectra relationship*

To have a quantitative picture of the above observations the wave number ($\bar{\nu}_{max}$) values at λ_{max} of the dyes in various solvents have been correlated with solvent parameters. The $E_T(30)$ parameter developed by Reichardt [24] is reported to be the most appropriate to explain the electronic transition of various organic compounds. The parameter has been generated from a pyridinium *N*-phenolate betaine dye. Because of the analogy of functional group of the dyes (**2–5**) with the betaine, $E_T(30)$ parameter has been considered to explain the effect of solvents.

For all the compounds, $\bar{\nu}_{max}$ observed in nonpolar solvents exhibit a linear relationship with the polarity scale (Figs. 4–7). Similar is the trend in the relationship for hydrogen acceptor solvents. However, the increasing trend with polarity is less significant for the later class of solvents when compared to the former. This trend may be ascribed to the differential contribution of the two mesomeric structures (as example **3** and **9**) to the ground state and excited state. During excitation, differential intramolecular charge transfer steers the population of

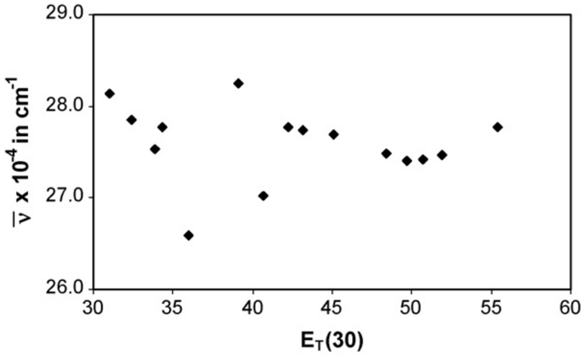


Fig. 4. Plot of $E_T(30)$ vs. $\bar{\nu}_{max}$ of dye **2**.

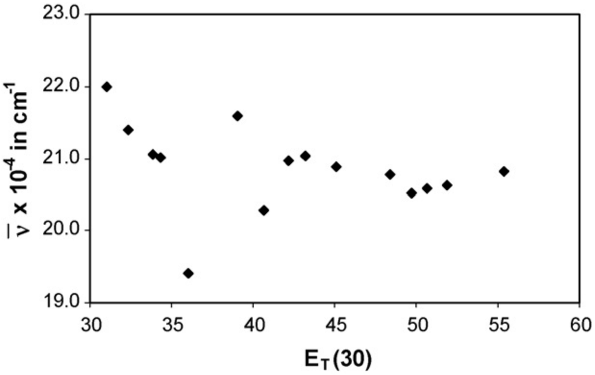


Fig. 5. Plot of $E_T(30)$ vs. $\bar{\nu}_{max}$ of dye **3**.

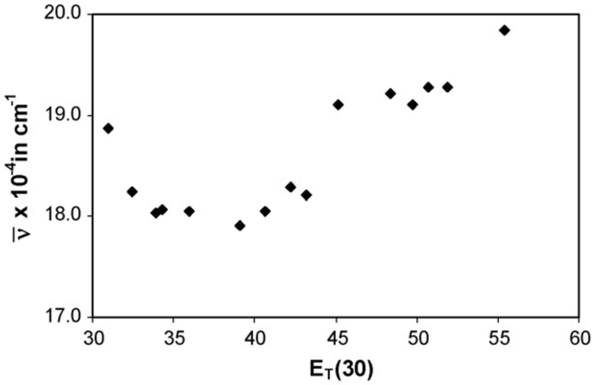


Fig. 6. Plot of $E_T(30)$ vs. $\bar{\nu}_{max}$ of dye **4**.

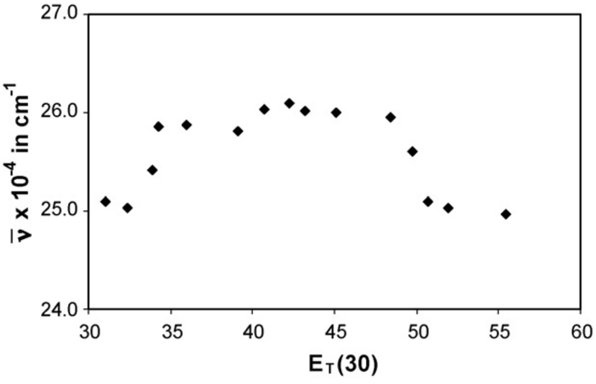
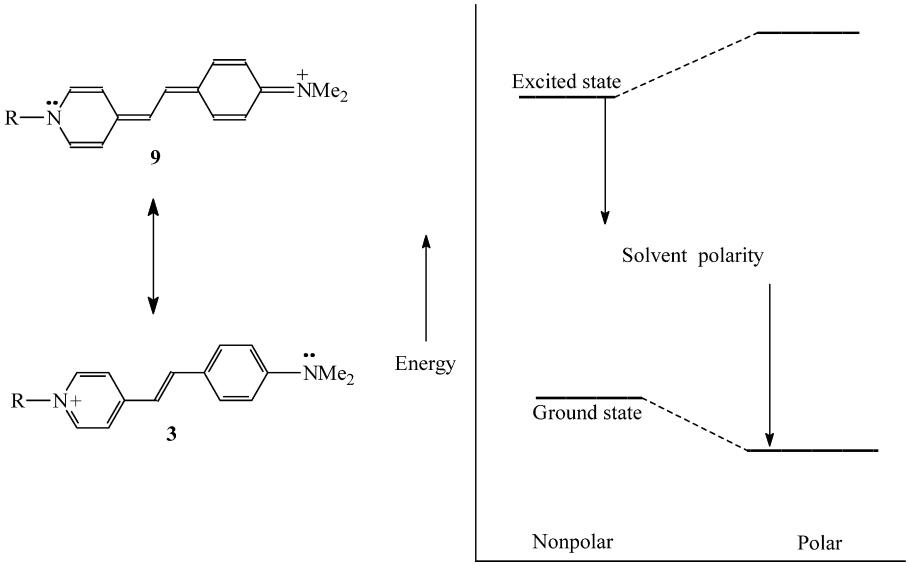


Fig. 7. Plot of $E_T(30)$ vs. $\bar{\nu}_{max}$ of dye **5**.



Scheme 1. Qualitative representation of the solvent effect on the stability of ground and excited states of the styrylpyridinium dyes (compound **3** is reported as a representative dye).

both the mesomers. Accordingly the differential solvation of the species results in shift of absorption maxima. In polar solvents the increasing trend suffers a reversal at an $E_T(30)$ value of 49.4. The decreasing trend is mostly due to hydroxylic solvents. The hypsochromic shift with increasing polarity may be ascribed to the stability of the ground state of the dye (Scheme 1).

Assuming (i) the contribution of nonpolar solvent to the stability of excited state and (ii) that of hydroxylic solvent to the stability of ground state are 100 percent each, the extents of contribution of dipolar aprotic solvents to both the states have been calculated from the sensitivity (α) of $E_T(30)$ towards $\bar{\nu}_{max}$ (Eq. (1)). In Eq. (1), β is the $\bar{\nu}_{max}$ in a hypothetical environment with '0' $E_T(30)$ value.

$$\bar{\nu}_{max} = \alpha E_T(30) + \beta \qquad (1)$$

For three nonpolar solvents (hexane, tetrachloromethane and toluene) the 'α' and 'β' are found to be $(-2.07 \pm 0.001) \times 10^3$ and $(345.5 \pm 0.003) \times 10^3$, respectively. Similarly for hydroxylic solvents the values are found to be $(0.68 \pm 0.1) \times 10^3$ and (239.8 ± 5.8) and for dipolar aprotic solvents the values being $(-0.96 \pm 0.26) \times 10^3$ and $(319.6 \pm 11.2) \times 10^3$, respectively. By considering the sensitivity of the $E_T(30)$ scale towards the $\bar{\nu}_{max}$, the extent of contribution of the dipolar aprotic solvent to the stability of the excited species can be calculated as $= 100 \times (\alpha_{dipolaraprotic} - \alpha_{hydroxylic})/(\alpha_{nonpolar} - \alpha_{hydroxylic}) = 59.8\%$.

Though the nonlinearity in the solvatochromic plots questions the generality of the $E_T(30)$ scale, the parameter can successfully classify the solvents into three aforesaid classes with respect to the solvation. The absolute sensitivity in nonpolar solvent is more than hydroxylic solvent, which has almost same sensitivity as that in dipolar aprotic solvents.

The absorption maxima of dye **3** in different solvents also behave similarly as those of dye **2**. The sensitivity of polarity in nonpolar solvent towards $\bar{\nu}_{max}$ increases significantly $(-2.97 \pm 0.39) \times 10^3$ while for the dipolar aprotic solvent and hydroxylic solvents the values come to $(-1.2 \pm 0.34) \times 10^3$ and $(0.5 \pm 0.04) \times 10^3$, respectively. Thus, the extent of contribution of the dipolar aprotic solvent being 49.2%. The reversal in solvatochromism is observed at an $E_T(30)$ value of 50.0 with a transition from dipolar aprotic solvents to hydroxylic solvents.

3.3. *Reversal of solvatochromism*

Earlier we have reported a reversal of solvatochromism for a series of bischromophoric styrylpyridinium dyes with hydroxyl substituents in the styryl unit at an $E_T(30)$ scale of 49.7 kcal/mol with a transition from hydrogen-bond acceptor to donor system [15]. The result was ascribed to the interaction of the hydroxyl group of the dyes with the hydrogen-bonding group of the solvent. Interestingly the trend of solvatochromism in the dye **4** suffers a reversal at $E_T(30)$ of 37.6 with a transition from nonpolar to polar solvents. Further the sensitivity of solvent polarity is less for hydroxylic solvents than for dipolar aprotic solvents.

All the above observation may lead to the following proposition for solvation.

- In a nonpolar solvent-cage the effect of change in polarity of the solvent is more when compared to that in a polar solvent cage. The polar solvents having p-orbitals may have secondary interactions with the p-orbitals of the dye, which probably masks the sensitivity of the polar effect of the solvent on the electronic transition of the dye.
- The dipolar aprotic and hydroxylic solvents have solvent cages with almost similar polarity. However the reversal may be attributed to the interaction of polar functional group of the solvent with the quaternary nitrogen of the pyridinium ring.
- For hydroxylic dye, the hydrogen bond acceptor characteristic of dipolar aprotic solvent on the hydroxy group makes the

ground state more stable due to a partial negative charge on the hydroxyl group (**7**). With increasing hydrogen-bonding ability of the solvent, the ground state stabilizes more leading to a hypsochromism. Thus the reversal in this case is observed at $E_T(30)$ value of 37.6.

For a similar dye Da Silva et al. have also observed a reversal at $E_T(30)$ value of 37.0 [25].

Surprisingly the plot of $E_T(30)$ against $\bar{\nu}_{max}$ of **5** (Fig. 7) shows a complete reversal of the corresponding plot for other dyes (Figs. 5 and 6). The plot exhibits that there is a sharp increase in $\bar{\nu}_{max}$ in nonpolar solvents with increase in polarity, the trend in increase reduces in dipolar aprotic solvents, and then there is a sharp decrease with increase in polarity of hydroxylic solvents. The reversal in solvatochromism is observed at 44.0 kcal/mol $E_T(30)$ value. Thus the reversal in solvatochromism may be considered as a switch to explain either structural transition of the dye due to solvent or a transition due to solvent cage with differential polarity.

The analysis of the plots in Figs. 4–7 reveals some interesting observations.

- The three distinctly grouped solvents due to their polarity stabilize the ground and excited species of the dyes **2** and **3**. Thus the groups of solvents provide specific solvent cages, wherein the changes in polarity of the medium have effect on the electronic transition to different extent.
- In dye **4**, the polar solvents cause a structural change of the dye (**7**) for which there is a difference in the polarity switch.
- The electron-releasing vector in dye **5** provides a parallel electron flow from methyl group and phenyl group to the pyridinium unit whereas dye **2** experiences an electron flow in series connection of the phenyl and methyl groups. In analogy to inverse relationship of electron flow in parallel and series connection, the $\bar{\nu}_{max}$ experience a reversal trend with respect to $E_T(30)$ scale in **2** and **5**.

Acknowledgements

The authors thank the Department of Science and Technology and the University Grants Commission, New Delhi, for financial support through FIST and DRS programs, respectively.

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