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Spectrophotometric Studies on Aggregation of Some Acid Dyes in Different Media

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Abstract. The electronic absorption spectra of different acid dyes, Crocein Orange G, Acid Alizarin N and Acid Orange 10 have been investigated in aqueous and non-aqueous solutions. Also spectrophotometric results on the aggregation of the studied dyes in presence of electrolytes are presented.

As the concentration of the dye increases the absorption spectra shifts to shorter wavelengths, this behaviour has been attributed to the formation of high aggregates. The larger wavelength, almost worked in dilute solutions, is typical of the monomeric dye. The aggregation of these dyes was studied quantitatively using the Maximum Slope Method. The investigated three dyes are aggregated at room temperature with an average aggregation number.

Introduction

Many studies have been carried out on the aggregation of dyes molecules or ions in aqueous and non aqueous solutions[1-6]. The interactions between dye molecules and other molecular entities are of paramount importance in both medicine and many industrial processes, *e.g.* textile fibers, leather and paper in the present-day life[7]. The forces of attraction responsible for such interactions are also capable of causing physical interaction between dye molecules to produce molecular species ranging in size from dimmers to aggregates containing hundreds of molecules[8]. The aggregation of dyes in solution depends on several factors such as the structure, concentration of the dye, pH, nature of the media and foreign ions[9, 10]. The effect of dye concentration and nature and amount of solvents on the aggregation property of Acid Orange 8 and Acid Red 26 dyes have been investigated spectrophotometrically[11]. The average aggregation numbers and aggregation constants of the different solutions of the mentioned dyes were calculated. The behaviour of three Mordant dyes namely C.I. Mordant Yellow 5, C.I. Acid Red 183 and C.I. Acid Yellow 23 in presence of different concentrations of electrolytes was studied. The values of the aggregation numbers and aggregation constants are recorded from deviation of Beer's law[12]. The effect of added salts on the aggregation of Neutral Red and Acridine Orange showed that the tendency of the dye aggregation was increased in presence of salts[13].

Previous studies revealed that many techniques were employed to study the aggregation of dyes such as diffusion[14], conductivity[15], polarography[16], and proton magnetic resonance spectra[9]. Among these physico-chemical methods, the spectrophotometric method is the most convenient[17, 18]. The aim of the present work is investigation of the effect of nature and composition of media, dye structure and concentration, types and amount of added electrolytes on the aggregation number and aggregation constant of the reported acidic dyes.

Experimental

Materials and Reagents

Three acidic dyes namely, Crocein Orange G, Alizarin Violet N and Orange 10, were purchased from Aldrich Chemical Company. Purification of these dyes was achieved by recrystallization from 50% aqueous ethanol.

The solvents and salts used (K_2SO_4 and Na_2SO_4) are of analytical grade, methanol (BDH), ethanol (BDH) and propanol (GLC). The salts used were (WINLAB) grade. Solutions of dye in presence of different percentages of organic solvents (20, 40, 60% v/v) were prepared. Different amounts of salts were added to the dye solutions (0.25, 0.5, 1.0 M). Solutions were allowed to stand at room temperature for 24 hours before measuring the absorbance of these solutions.

Apparatus

The spectrophotometric measurements were carried out on a double beam digital reading recording instrument model Pharmacia Biotech, Ultra Spectra 2000. All measurements were taken at room temperature using a 1.0 cm quartz cell.

Results and Discussion

1. Effect of Solvents

The aggregation property of studied dyes was examined by measuring the electronic absorption spectra of several of various concentration of dyes in water and methanolic-water solutions of 20, 40, 60 (v/v) and the results are given in Tables 1, 2, and 3. At low dye concentration there is a broad band whose λ_{max} appears at 484 nm in pure water. This band may be assigned to the monomer form of Crocein G. As the dye concentration increases one notice a blue shift to shorter wavelength. This shift may be interpreted as due to the formation of one or more dye aggregates[16, 17]. For any dye concentration the intensity of the band decreases as one goes from pure water to methanol-water solutions. Another interesting phenomenon is the shift of the characteristic λ_{max} towards shorter wavelength as the percentage of methanol in water is raised. These spectral changes can be attributed to aggregation of the dye molecules under the influence of the strong dispersion forces associated with the length polarizability of the chromophoric chain[11].

Similar behaviour is observed for the spectra of different concentrations of acid dye Crocein Orange G in ethanol-water and propanol-water solution as illustrated in Table 1.

The spectra of Alizarin N and Acid Orange 10 in aqueous and non-aqueous solutions were done. The behaviour of the two dyes is similar to that of acid dye Crocein Orange G (Tables 2, 3). Bands of aggregate dyes, when observed, usually lie on the shorter wavelength. This phenomenon has been interpreted on the bases of Förster's model[11]. Thus, when the dimmer is formed from two monomers (free dye ions) lying close together, with their molecular planes opposite each other, the excited level is spitted into two, of which only the higher level has an appreciable transition moment, causing the blue shift of the dimmer-band.

Tables 1, 2 and 3 summarize the results obtained from the absorption spectra of all three dyes. It is clear from these tables that the molar absorptivities of the three dyes decrease as the concentration of the dyes increase.

So far the changes in absorption spectra have been described qualitatively, yet the aggregation of dyes in aqueous and non-aqueous solutions may be studied quantitatively. It is rather difficult to make a reliable estimate of the aggregation parameters, even for a simple equilibrium system between a monomer and a polymer of one definite degree of aggregation. Yet it is more difficult when variables aggregation numbers are present in dye solutions. The Maximum slope[19] has proved to be the simplest and the most convenient method to apply

Table 1. Data of concentration, absorbance and λ_{max} for Crocein Orange G acid dye in different media.

Dye conc. M/L	H_2O		Methanol						Ethanol					
	T = 25ºC		20%		40%		60%		20%		40%		60%	
	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.
4×10^{-5}	484	0.683	485	0.685	486	0.634	486	0.601	487	0.687	486	0.679	485	0.637
6×10^{-5}	484	1.020	485	1.047	486	0.968	485	0.903	486	1.034	487	0.964	485	0.876
8×10^{-5}	484	1.331	485	1.365	486	1.268	485	1.191	486	1.318	487	1.258	485	1.162
1.0×10^{-4}	483	1.661	484	1.695	486	1.594	484	1.489	486	1.668	486	1.544	485	1.481
1.2×10^{-4}	483	1.979	484	1.986	485	1.928	485	1.787	486	1.955	487	1.874	485	1.758
1.4×10^{-4}	483	2.247	484	2.295	486	2.225	485	2.093	486	2.281	487	2.185	485	2.038
1.6×10^{-4}	482	2.584	484	2.591	485	2.583	485	2.436	486	2.628	486	2.484	485	2.325
1.8×10^{-4}	482	2.848	484	2.880	485	2.906	485	2.625	485	2.873	485	2.820	485	2.619
2.0×10^{-4}	482	3.149	484	3.177	486	3.207	485	2.951	485	3.178	486	3.083	485	2.897
2.2×10^{-4}	483	3.406	483	3.494	486	3.471	485	3.220	485	3.451	486	3.329	485	3.235

Dye conc. M/L	Propanol						Na_2SO_4				K_2SO_4			
	20%		40%		60%		0.25 M		0.5 M		0.25 M		0.5 M	
	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.
4×10^{-5}	487	0.682	486	0.625	486	0.578	484	0.663	483	0.663	484	0.693	483	0.704
6×10^{-5}	487	1.986	485	0.885	485	0.880	483	0.993	482	0.958	483	0.983	482	0.989
8×10^{-5}	487	1.313	485	1.162	485	1.158	483	1.271	482	1.265	483	1.316	482	1.253
1.0×10^{-4}	487	1.641	485	1.458	485	1.410	482	1.572	482	1.551	482	1.598	482	1.234
1.2×10^{-4}	487	1.920	485	1.740	485	1.685	482	1.861	482	1.812	482	1.892	483	0.886
1.4×10^{-4}	487	2.246	485	2.067	485	1.985	481	2.143	481	2.099	482	2.188	483	0.583
1.6×10^{-4}	487	2.556	485	2.317	485	2.244	482	2.420	481	2.371	481	2.475	484	0.566
1.8×10^{-4}	487	2.839	485	2.573	485	2.567	481	2.698	481	2.624	481	2.773	483	0.575
2.0×10^{-4}	487	3.133	485	2.888	485	2.795	182	2.962	480	2.855	482	2.992	484	0.577
2.2×10^{-4}	487	3.430	485	3.172	485	3.041	481	3.216	481	3.185	481	3.239	483	0.558

Table 2. Data of concentration, absorbance and λ_{max} for Alizarin Violet N dye in different media.

	H_2O		Methanol						Ethanol					
Dye conc. M/L	**T = 25ºC**		**20%**		**40%**		**60%**		**20%**		**40%**		**60%**	
	Abs.	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**	**Abs.**	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**
4×10^{-5}	533	0.709	508	0.352	508	0.374	508	0.357	528	0.437	528	0.447	524	0.475
6×10^{-5}	533	0.905	508	0.523	508	0.546	508	0.539	528	0.641	528	0.691	528	0.729
8×10^{-5}	533	1.158	508	0.702	504	0.735	524	0.679	528	0.826	528	0.883	528	0.985
1.0×10^{-4}	533	1.291	508	0.856	508	0.889	524	0.854	486	1.052	528	1.114	528	1.214
1.2×10^{-4}	533	1.438	508	1.021	508	1.060	524	1.005	486	1.257	528	1.313	528	1.427
1.4×10^{-4}	533	1.627	520	1.151	508	1.225	508	1.221	486	1.430	528	1.561	528	1.609
1.6×10^{-4}	533	1.844	516	1.315	508	1.402	508	1.388	486	1.609	528	1.714	528	1.849
1.8×10^{-4}	533	1.930	516	1.490	508	1.582	524	1.501	485	1.961	528	1.877	528	2.073
2.0×10^{-4}	534	2.113	520	1.641	508	1.734	524	1.343	485	2.081	528	2.149	528	2.319
2.2×10^{-4}	534	2.266	520	1.778	512	1.870	520	1.865	485	2.181	528	2.330	528	2.521

	Propanol						Na_2SO_4				K_2SO_4			
Dye conc. M/L	**20%**		**40%**		**60%**		**0.25 M**		**0.5 M**		**0.25 M**		**0.5 M**	
	Abs.	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**	**Abs.**	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**	**λ_{max}**	**Abs.**
4×10^{-5}	508	0.360	528	0.449	508	0.375	516	0.323	483	0.403	524	0.380	516	0.316
6×10^{-5}	524	0.514	528	0.534	508	0.529	532	0.481	482	0.597	524	0.567	524	0.478
8×10^{-5}	532	0.642	532	0.704	508	0.705	504	0.638	482	0.773	528	0.736	544	0.677
1.0×10^{-4}	504	0.879	528	0.851	508	0.883	520	0.782	482	0.938	528	0.918	520	0.763
1.2×10^{-4}	520	1.030	532	0.973	508	1.023	504	0.922	482	1.138	528	1.100	520	0.904
1.4×10^{-4}	524	1.166	532	1.123	508	1.171	504	1.102	481	1.306	528	1.277	528	1.066
1.6×10^{-4}	524	1.354	532	1.272	508	1.334	504	1.242	481	1.458	528	1.457	524	1.187
1.8×10^{-4}	524	1.500	528	1.386	508	1.541	524	1.379	481	1.628	528	1.619	524	1.311
2.0×10^{-4}	528	1.636	532	1.486	524	1.653	504	1.514	480	1.803	528	1.778	524	1.458
2.2×10^{-4}	524	1.818	528	1.602	508	1.864	508	1.653	481	1.959	528	1.951	524	1.586

Table 3. Data of concentration, absorbance and λ_{max} for Orange 10 dye in different media.

	H_2O		Methanol						Ethanol					
Dye conc. M/L	T = 25ºC		20%		40%		60%		20%		40%		60%	
	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.
4×10^{-5}	478	0.739	479	0.723	480	0.676	480	0.762	480	0.760	480	0.745	479	0.719
6×10^{-5}	478	1.108	479	1.074	479	1.081	479	1.103	781	1.091	480	1.090	479	1.046
8×10^{-5}	478	1.448	479	1.449	480	1.380	495	1.446	480	1.467	480	1.426	496	1.403
1.0×10^{-4}	478	1.795	479	1.779	480	1.731	495	1.775	479	1.760	480	1.780	496	1.755
1.2×10^{-4}	478	2.154	478	2.187	479	2.043	495	2.123	479	2.133	480	2.145	496	2.093
1.4×10^{-4}	478	2.505	479	2.507	478	2.470	479	2.478	480	2.493	479	2.472	496	2.446
1.6×10^{-4}	478	2.850	478	2.827	478	2.773	479	2.807	479	2.820	480	2.812	495	2.819
1.8×10^{-4}	478	3.190	480	3.178	479	3.023	478	3.183	481	3.160	481	3.160	497	3.098
2.0×10^{-4}	478	3.480	480	3.459	480	3.433	482	3.467	479	3.473	480	3.458	496	3.380
2.2×10^{-4}	478	3.708	480	3.696	477	3.689	479	3.689	479	3.697	478	3.702	494	3.651
	Propanol						Na_2SO_4				K_2SO_4			
Dye conc. M/L	20%		40%		60%		0.25 M		0.5 M		0.25 M		0.5 M	
	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	Abs.	λ_{max}	Abs.	λ_{max}	Abs.	λ_{max}	Abs.
4×10^{-5}	480	0.708	480	0.739	497	0.720	497	0.751	477	0.724	479	0.737	479	0.703
6×10^{-5}	480	0.062	480	1.056	479	1.067	479	1.089	479	1.079	478	1.116	478	1.125
8×10^{-5}	480	1.436	479	1.387	496	1.374	478	1.448	478	1.427	478	1.458	478	1.474
1.0×10^{-4}	480	1.751	479	1.729	496	1.730	477	1.812	478	1.777	478	1.802	478	1.822
1.2×10^{-4}	480	2.128	480	2.059	496	2.036	478	2.167	478	2.167	478	2.150	478	2.171
1.4×10^{-4}	480	2.454	479	2.426	496	2.439	478	2.509	478	2.452	477	2.500	477	2.511
1.6×10^{-4}	480	2.820	480	2.771	494	2.737	478	2.823	478	2.849	478	2.845	478	2.844
1.8×10^{-4}	479	3.156	479	3.073	495	3.022	477	3.168	477	3.193	477	3.192	477	3.164
2.0×10^{-4}	478	3.448	479	3.383	496	3.333	477	3.483	478	3.492	479	3.477	479	3.462
2.2×10^{-4}	478	3.712	480	3.627	495	3.524	477	3.731	480	3.668	479	3.722	479	3.716

for such quantitative study. This method is based on the assumption that a simple equilibrium model between a monomer (m) and polymer $(m)_n$ is operative:

$$n(m) \rightleftharpoons (m)_n$$

First the following equation is used to estimate the values for degree of aggregation and aggregation constant[17]:

$$\log C\,(\varepsilon_1 - \varepsilon) = n \log C\left(\varepsilon - \varepsilon'_n\right) + \log n\,K_n\left(\varepsilon_1 - \varepsilon'_n\right)^{1-n}$$

Where $\varepsilon'_n = \dfrac{\varepsilon_n}{n}$

ε_1 = molar absorbativity of monomer
ε = experimentally measured molar absorptivities
ε_n = molar absorbativity of polymer
C = concentration of dye solution in mole/L
n = aggregation number
K_n = aggregation constant

By plotting $\log C\,(\varepsilon_1 - \varepsilon)$ against $\log C\left(\varepsilon - \varepsilon'_n\right)$, the points should lie on a straight line; the slope of which gives the degree of aggregation (n), and the aggregation constant (K_n) can be calculated from the intercept. Reliable results are very difficult to be obtained due to the assumption one has to make for ε_1 and ε_n. The above equation is then rearranged in the form:

$$\frac{1}{n-1}\log(\varepsilon_1 - \varepsilon) - \frac{n}{n-1}\log\left(\varepsilon - \varepsilon'_n\right) = \log \alpha C = X$$

Where $\alpha = (nk)^{\frac{1}{n-1}}\left(\varepsilon_1 - \varepsilon'_n\right)^{-1}$

The Maximum Slope Method consists of correlating the curves of ε *vs.* log C and ε *vs.* X for different values of n and ε_n until a value of n is reached which gives the best fit between the experimental results and the theoretical curve.

Figure 1 shows a typical graph of ε *vs.* log C and ε *vs.* X for acid dye Crocein Orange G in H_2O.

It is obvious from this graph that the best fit between the theoretical and experimental curves is obtained for n = 3. Similar curves are drawn for the other dyes in different solutions. Table 4 lists the average aggregation number (n) and the aggregation constant (k_n) calculated for the three acid dyes using the Maximum Slope Method.

It is clear from the above study that the tendency of the examined acidic dyes to aggregate increases as the concentration of organic solvents increases in water.

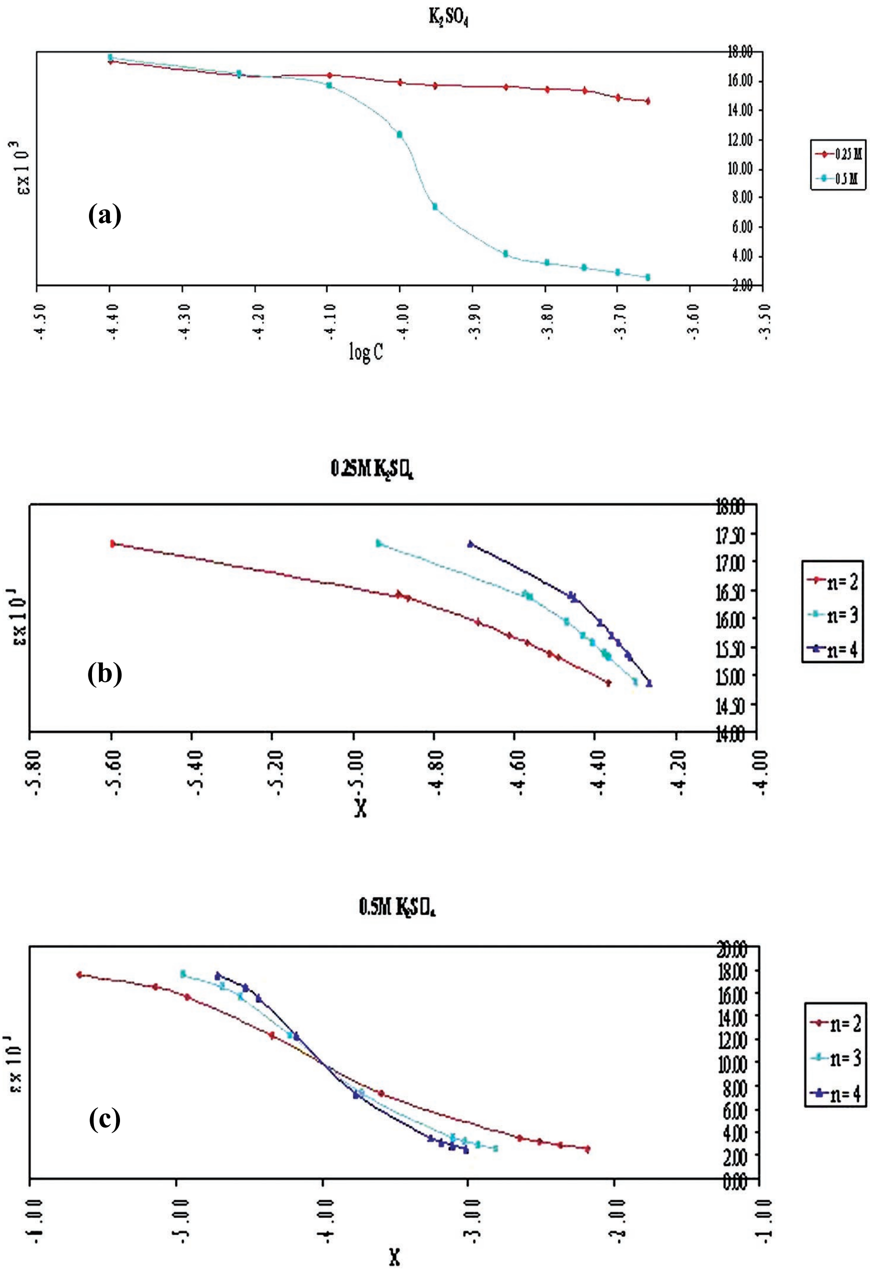

Fig. 1. The experimental (a) and theoretical (b,c) data for acid dye Crocein Orange G in presence of different concentrations of K_2SO_4.

Table 4. The aggregation constants (K_n) and aggregation numbers (n) for acid dyes using the maximum slope method in different media.

Name of dye	Solvent concentration (40 v/v)	Aggregation constant $K_n \times 10^{-3}$	Aggregation number (n)
1 – Acid Crocein Orange G	0	2.12	3.33
	20% Methanol	5.7	3.05
	40% Methanol	–	–
	60% Methanol	6.85	3.57
	20% Ethanol	6.33	2.75
	40% Ethanol	0.25	3.85
	60% Ethanol	–	3.86
	40% Propanol	0.056	–
	60% Propanol	0.099	3.60
2 – Acid Alizarin Violet N	0	0.006	1.60
	20% Methanol	0.098	–
	40% Methanol	0.119	–
	60% Methanol	2.600	3.20
	60% Ethanol	0.061	2.19
	20% Propanol	1.025	2.45
	40% Propanol	–	3.48
	0.25 M Na_2SO_4	1.300	2.80
	0.50 M Na_2SO_4	–	3.33
	0.25 M K_2SO_4	1.300	2.80
	0.50 M K_2SO_4	–	3.33
3 – Acid Orange 10	0	0.920	2.04
	20% Methanol	3.315	2.76
	60% Methanol	–	2.90
	20% Ethanol	–	2.32
	40% Ethanol	1.717	2.89
	60% Ethanol	1.322	3.15
	20% Propanol	–	2.80
	40% Propanol	0.038	3.00
	60% Propanol	2.465	3.20
	0.50 M Na_2SO_4	0.050	1.17
	1.00 M Na_2SO_4	0.090	2.14
	0.50 M K_2SO_4	0.351	2.40

The increase of the association is generally ascribed to the lower polarity of the medium[20]. This effect is attributed to the standing capacity of solvents for breaking up hydrogen bonds in water and thus destroying the ice-bergs which presumably are associated with fairly scale co-operative structures stabilized by water-water hydrogen bonding.

2. *Effect of the Added Salts on the Aggregation of the Dyes*

The dependence of the absorbance spectra of the studied dyes on the concentration of the added salts, Na_2SO_4 and K_2SO_4 are summarized in Tables 1, 2 and 3 (for example). It is evident that the λ_{max} shifts towards shorter wavelength by increasing the concentration of the added salts. Also the absorbance of different concentrations of the dyes decreases with the addition of increasing amounts of salts. According to Moulik and Ghosh[21], the presence of positive ionic species helps the dye molecules to associate in water, which in turn brings about changes in the spectral behaviour. Such association may lead to dye-dye interactions, thus facilitating aggregation.

To estimate the effect of salts on aggregation, the evaluation of aggregation number and aggregation constant were evaluated by using the Maximum Slope Method and the results are given in Table 4. The results show that the aggregation number (n) and aggregation constant (K_n) increase as the salt concentration increases.

Aliphatic and aromatic molecules or groups present in water are surrounded by a law entropy structured region known as Frank's "ice-berg" region[10]. Solvation of the nitrogen centers of the dye molecules through hydrogen bonding via the centers of water dipoles may contribute to this region. The hydrophobic association-dissociation phenomenon of the dye molecules may energetically arise by the partial melting and forming of this ice-berg. Ions of an added salt can loosen the protective water sheath around the dye molecule, thus destroying the ice-berg. Once this protection is disturbed, the bare dye molecules associate as a result of the hyrophobic force. The positive environments of cations surrounding the anions dye can also have the power to compel the partly or fully naked dye monomer to unite[10].

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دراسة طيفية على تجمع بعض الصبغات الحمضية في أوساط مختلفة

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المستخلص. تمت دراسة تأثير إضافة نسب متزايدة من بعض المذيبات العضوية ، بالإضافة إلى تركيزات مختلفة من بعض الأملاح على السلوك الطيفي لبعض الصبغات الحمضية (Crocein Orange G, Alizarine N and Acid Orange10). وقد أوضحت نتائج الدراسة أن إضافة النسب المختلفة من المذيبات العضوية يؤدي إلى إزاحة طيف الامتصاص في اتجاه الطول الموجي الأكبر.

ومن ناحية أخرى تبين من نتائج البحث أن إضافة نسب الأملاح المختلفة تؤدي إلى إزاحة الطول الموجي باتجاه الطول الموجي الأصغر ، وزيادة ميل جزيئات الصبغة للتجمع .

وقد تم استخدام طريقة أقصى الميل لتعيين كل من عدد وثابت التجمع (Kn, n)، حيث أوضحت القيم التي تم الحصول عليها زيادة تجمع الصبغات المستخدمة بزيادة تركيز كل من المذيبات والأملاح المضافة .