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Temperature Dependence on the Molecular Interaction of Amaranth Dye with Cetyltrimethylammonium bromide in the Pre-micelle Region: A Spectroscopy Study

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Abstract: Interactions of amaranth dye [Amr] with cetyltrimethylammonium ammonium bromide [CTABr] have been investigated using UV-spectrophotometric technique. Finding from the variation absorption spectral shows that absorbance values at the highest peak changes on addition of CTABr indicating that interaction exist between Amr and CTABr. The binding constant [Kb] between the micellar/water phases has been obtained using appropriate model from which the thermodynamic parameters have been calculated at different temperatures. The binding decreases with increasing temperatures. The free energy [ΔG0], entropy [ΔS0] and enthalpy [ΔH0] changes regarding binding process are spontaneous, exothermic and enthalpy driven.

Keywords: Amaranth; Pre-micelle; Reso-Drago; Binding constant; Spectroscopy

1. Introduction

Dye-surfactant interaction has recorded so many applications in recent years in the following area of industrial applications (i) pharmaceutical[1] (ii) photography printing,[2] (iii) photosensitization[3] etc. On this note, more attention has drawn to the study of dye-surfactant interaction.[4-11] Investigation on dye-surfactant interactions has shown that it help in understanding chemical equilibria mechanisms and kinetics of surfactants-sensitized color and fluorescence reaction.[12] Several factors influenced dye-surfactant interaction among which chemical structures played a prominent role. Irrespective of either of these factors, dye-surfactant complexes, ion-pairs, self-aggregate of dye; pre-micellar aggregate and pure micelles of surfactant with solubilized dye are formed in course of this interaction.

Amaranth (Amr) commonly referred to as Red No. 2, Food Red 9, Acid Red 27, and Azorubin S, belong the group of Azo dyes which are broadly used in the textile, color solvent, ink, paint, varnish, plastic, food, drug and cosmetic industries.[13] It is a water-soluble dye, with sharp absorption peak in the visible region ($\lambda \max$ = 520 nm) with no shift due to pH change.[14]

Cetyltrimethyl ammonium bromide (CTABr) is widely used in industrial application most especially as auxiliary in textile finishing process.[15] Undertaken this study is very important and interesting

for improving dyeing process from theoretically point of view, which account for its valuable exploring the interaction between dye and surfactant.[16]

When surfactant is found in solution it changes from pre-micelle to micelle. At low concentration, it arranges itself on the surface of the aqueous solution in such a way that the hydrophobic part is facing outward and hydrophilic part is towards an aqueous solution. Micelle formation is a cooperative process that occurs over a narrow range of concentration, where the transition from monomeric solution to a solution containing both monomers and micelle take place. The single concentration within this narrow range is called critical micelle concentration (CMC) with hydrophobic part within the core of the cluster and hydrophilic part facing the aqueous solution. Each kind of formation (assembly) has its own peculiar characteristic and performance, while its effect on reaction has always been a matter of study.

Interaction of dye- surfactant in pre-micellar region results in the formation of ion association complex while in post – micelle region dye molecule are localized at the micelle surface. Considering the above, Sidract et al.,[17] monitored the change in electrical conductivity and spectra behavior before and after micelle formation in the dye-surfactant system. Likewise, catalytic effect of CTABr reverse micellar systems on the alkaline fading of malachite green has been reported by Somnath et al.,[18] to taking place at a much faster rate than in normal micelles or in a continuous aqueous

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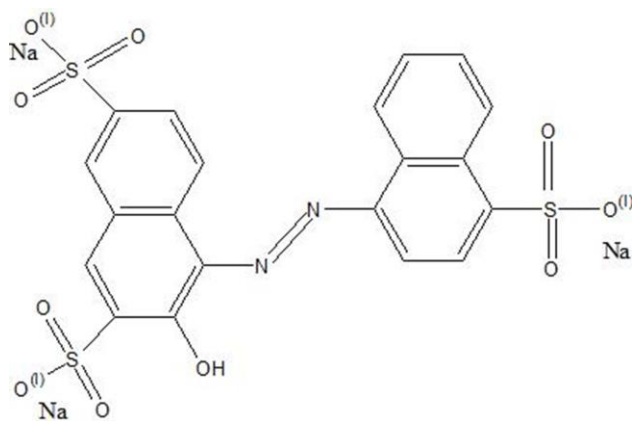
Scheme 1 Molecular structure of Amaranth (Amr)
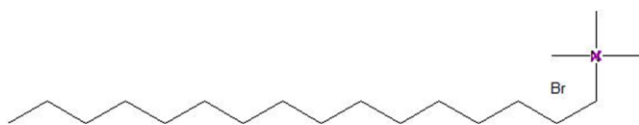
Scheme 2. Molecular structure of Cetyltrimethylammonium Bromide (CTABr)

medium. Observation from there study shows that both the electrostatic interaction between dye and surfactant and the dissolution of dye-surfactant aggregate depends on the concentration of surfactant.

There are different methods for the study of the dye-surfactant system which has been reported by various researchers such as spectrophotometric,[19,20,21,22] conductometric,[23,24,25] tensiometric,-[26,27] and potentiometric[28] etc. Result has shown that these methods reveal that the oppositely charge dye (+/-) and surfactant (-/+) have strongest interaction and same charge dye (+/-) and surfactant (+/-) have almost zero interaction.[29] Summarily, it was also believed that shifting in λ_{max} and increase or decrease in intensity also attributed to complexation or dye-surfactant interaction.

Surfactant in any of its formation either at pre or post micellar stage influence absorption spectral of many dyes.[19] Hence, spectrophotometric method is widely adopted in monitoring dye-surfactant interaction.[29] In literature, study on the interaction of food colourant dye with either cationic, anionic and non-ionic surfactant using spectrophotometric has not been comprehensively monitor. Herein, study was made on the interaction of Amaranth dye (Amr) with cetyltrimethylammonium bromide (CTABr) in order to explore the nature and the stability of the interaction as a function of temperature within the premicellar range.

The aim of the presence study is to investigate the interaction between Amr and CTABr at the temperatures 298.15K, 303.15K, 308.15K, 313.15K, and 318.15K using spectrophotometric method and to determine the values of binding constant viz a viz thermodynamic parameter.

2. Materials and method

2.1. Materials

Amaranth dye (Amr) Cetyltrimethyl ammonium bromides (CTABr) were purchased from Sigma Aldrich Chemical Company. The reagents were of analytical grade (AR) and used without further purification. The chemical structures of Amr dye and CTABr surfactant are shown in scheme 1 and 2 respectively.

2.2. Solution preparation

Aqueous stock solution of dye Amaranth (Amr) (1.0 x 10⁻⁴ M) and Cetyltrimethyl ammonium bromide (CTABr) (2.0 x 10⁻² M), were prepared by dissolving their known amounts in doubly distilled deionized water and expressed in molar concentration. The subsequent dilutions were made by using doubly distilled deionized water. The dye concentration was fixed at 2.7 x 10⁻⁵ M during the measurements so as to have absorbance of the solutions in the reasonable range.

2.3. Experimental techniques

A series of dye solution was prepared from stock solution of Amr (1.0 x 10⁻⁴ M) to determine the molar extinction coefficient (ε_{Amr}). For the spectrophotometric measurement, an exact volume of dye was added to a known volume of doubly distilled deionized water and the absorption spectrum of dye was recorded. The reaction mixture was prepared by taking an exact volume of dye and surfactant in a cell containing a known volume of deionized water. The variation of absorbance of the dye solution was recorded as a function of increase in surfactant concentration at the wavelength of maximum absorbance at varying temperature.

2.4. Calculations Binding Constant

The binding constant (K_b) between Amr and CTABr were determined in the pre-micellar region of CTABr and it can be expressed as:

$$K_b = \frac{x}{(C_{CTABr}^0 - C_x)(C_{Amr}^0 - C_x)} \tag{1}$$

Where C_x is the concentration of reaction product, and C_{CTABr}^0, C_{Amr}^0 are the initial concentration of surfactant and dye respectively.

When $C_{CTABr}^0 >>> C_{Amr}^0$, the CTABr concentration may be assume constant. On this note, the experimental data can be analyzed using Reso-Drago equation.[30,31] The equation can be expressed as;

$$C_X = \frac{KC_{CTABr}C_{Amr}}{1 + KC_{CTABr}} \tag{2}$$

According to Beer-Lambert law, the absorbance of a unit thickness absorbing layer can be expressed as:
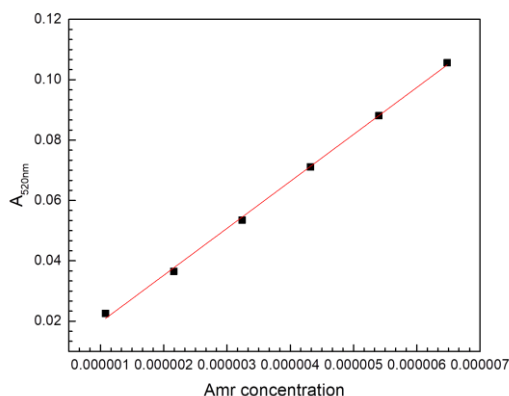
$$A = \varepsilon_{Amr}C_{Amr}^0 \tag{3}$$

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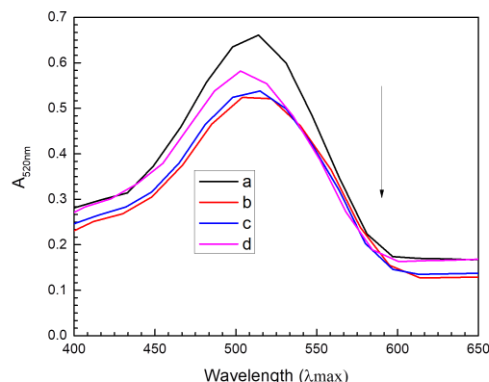**Fig. 1.** The absorbance of Amr in aqueous solutions of various concentrations at 520nm.

Fig. 2. Absorption spectrum of Amr (2.7 x 10^{-5} moldm^{-3}) in aqueous solution containing various concentration of CTABr: (a) 0.00, (b) 0.2, (c) 0.3 and (d) 0.5 mMoldm^{-3}

Table 1: K_b and ε_x values for the interaction of Amr with Cetyltrimethylammonium bromide within the pre-micelle region at different temperature

T/K	K_b M^{-1}	ε_x/M^{-1}cm^{-1}
298.15	28571	18873
303.15	11125	18814
308.15	10011	18465
313.15	7500	18214
318.15	6000	18005

Where A is the absorbance of Amr and ε_{Amr} is the molar absorptivity of Amr. At equilibrium, absorbance value can be expressed as:

$$A = \varepsilon_{CTABr}(C^0_{CTABr} - C_x) + \varepsilon_{Amr}(C^0_{Amr} - C_x) + \varepsilon_x C_x \quad (4)$$

Addition of equation (3) and (4) yields

$$\frac{C^0_{CTABr}.C^0_{Amr}}{A - A^1} = \frac{C^0_{CTABr}}{\varepsilon_x - \varepsilon_{CTABr} - \varepsilon_{Amr}} + \frac{1}{K(\varepsilon_x - \varepsilon_{CTABr} - \varepsilon_{Amr})} \quad (5)$$

Assuming the molar absorption coefficient for CTABr is ignored at wavelength of 520nm, equation (5) can be re-written as:

$$\frac{C^0_{CTABr}.C^0_{Amr}}{A - A^1} = \frac{C^0_{CTABr}}{\varepsilon_x - \varepsilon_{Amr}} + \frac{1}{K(\varepsilon_x - \varepsilon_{Amr})} \quad (6)$$

Where $A^1 = \varepsilon_{Amr}C^0_{Amr}$, K and ε_x are obtained from the slope and intercept of the plot of $\frac{C^0_{CTABr}C^0_{Amr}}{A - A^1}$ vs C^0_{CTABr}.

2.5. Thermodynamic Parameters

The free energy changes, ΔG^0, the enthalpy changes, ΔH^0, and entropy changes, ΔS^0, belonging to the association equilibrium constant K_b for the interaction of Amr with CTABr at different temperature can be calculate from the equation below;[32]

$$\Delta G^0 = -RT \ln K_b \quad (7)$$

$$\Delta H^0 = \left[\frac{\partial(\Delta G^0/T)}{\partial(1/T)}\right]_P \quad (8)$$

$$\Delta S^0 = \frac{\Delta H^0 - \Delta G^0}{T} \quad (9)$$

3. Results and Discussions

3.1. Behavior of Amr in the absence and presence of CTABr at pre-micellar region

The visible absorption spectrum of Amr (2.7 x 10^{-5} mol dm^{-3}) in aqueous solution exhibit an absorption maximum at 520nm with a molar extinction coefficient (ℇAmr) of 1.554 x 104 L mol^{-1} cm^{-1} obtained by plotting series of absorbance against different concentrations of Amr as shown in Fig. 1.

Spectra of Amr in the absence and presence of CTABr were presented in fig. 2. On addition of CTABr, the absorbance decreases without any significant change in the λmax. The decrease in the absorbance with increasing CTABr concentration in the pre micelle region can be explained in terms of dye–surfactant ion pair complex formation due to long-range electrostatic interaction between the positive charges of CTABr molecules and anionic sulfonate group of the Amr and short range hydrophobic forces which are strong enough for the formation of a stable ion pair complex of Amr-CTABr.[33]

This is further established by the fact that there is also no perturbation in the spectrum of the Amr and CTABr which has also been observed for other different dye– surfactant systems.[34-38] The dye interacted with the polar and non-polar part of the surfactant depending on the substituents.[33]

3.2. Effect of temperature on the behavior of Amr in the presence of CTABr at pre-micellar region

In order to analyze the influence of temperature on 2.7 x 105 moldm^{-3} spectra of Amr in the presence of 0.2mmoldm^{-3} CTABr, UV-Vis spectra were recorded. Fig. 3 shows variation in the UV-Vis spectra of

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Table 2. Thermodynamic parameters for the binding process in pre-micelle region at different temperature

Process	T/K	ΔG^0/KJmol	ΔH^0/KJmol	ΔS^0/JmolK^{-1}
Binding	298.15	-25420.3	-62595	-124.75
	303.15	-23470.7		-129.12
	308.15	-23587.8		-126.64
	313.15	-23219.3		-125.80
	318.15	-23000.2		-124.51
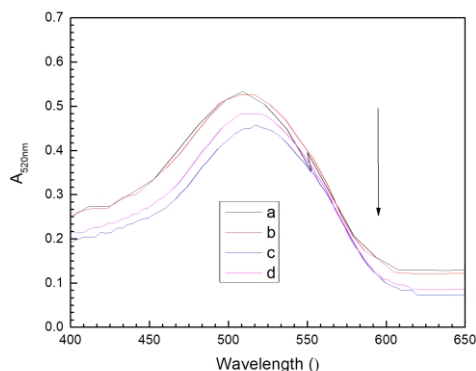
Fig. 3. UV-Vis absorption spectra of 2.7 x 10^{-5} mol.dm^{-3} Amr at different temperature (a = 298.15k, b = 303.15k, c = 313.15k and d = 318.15k) in the presence of 0.2 mmol.dm^{-3} CTABr concentrations
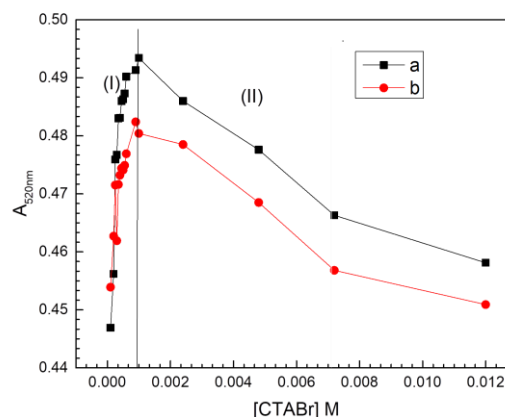
Fig. 4: Variation of the monomer absorbance of 2.7 x 10^{-5} moldm^{-3} Amr with CTABr at different temperature (a = 298.15k and b = 303.15k)

Amr at different temperatures. The characteristic peak at 520nm did not change with increasing temperature. Observation shows there is significant decrease in absorbance value of Amr as temperature increases. This may be attributed to the interaction between anionic dye (Amr) and cationic surfactant (CTABr).

Adoption of Reso – Drago equation for the interaction of Amr and CTABr at different temperature accounts for the parameter obtained in Table 1. Indication from the Table 1 shows that as temperature increases, both the binding constant Kb and the molar absorption coefficient Ɛx for the interaction of Amr and CTABr decreases. This means that high temperature causes a reduction in the interaction between Amr and CTABr molecule. In other word, the strength of interaction between Amr and CTABr at 318.15K is weaker than at 298.15K.

Effect of temperature on the trend of monomers absorbance of Amr in the presence CTABr were calculated from the data obtained in fig. 3 and depicted in fig. 4. As seen in Fig. 4, monomers of CTABr are available rather than the micellar aggregate.

Hence the monomer is interacting with the Amr molecules. The profile can be divided into two regions. In region I, the absorbance of Amr increases with increasing CTABr concentration. In this region, ion pair between Amr and CTABr and aggregate of ion pair are formed with CTABr concentration. Upon this, Amr-CTABr complex dissociate and the concentration of free Amr increases which allow Amr molecule to be accommodated into the pre-micelle monomeric molecule. In region II, there is decrease in absorbance value of Amr. This is due to the fact that there is less dissociation of Amr- CTABr complex. Consequently, the number of free Amr molecule decreases hence decrease in absorbance

To evaluate the thermodynamic aspects of the binding of Amr to CTABr pre-micelle, the thermodynamic parameters were calculated and are summarized in Table 2. The values of the Gibbs free energy of interaction (ΔG^0), the standard enthalpy (ΔH^0) and the standard

entropy (ΔS^0) changes were calculated from the values obtained for Kb at different temperatures from the spectral studies using equations (7) – (9) respectively.[32] As seen in Table 2, all ΔG^0 values are negative indicating that the binding process of Amr molecule to the CTABr monomeric molecule occur spontaneously. The ΔG^0 values become less negative with the increase in temperature for binding of Amr to CTABr pre-micelle. This means that binding process is preferred at lower temperatures.

The negative values of ΔH^0 indicate that binding process is exothermic in nature. The net ΔH^0 is the sum of the change in enthalpies resulting from hydrophobic interactions, electrostatic interactions, hydration of polar head groups, and counter ion binding to micelles.[39-41] Negative values of ΔH^0 may occur when electrostatic interactions and hydration of polar head groups become more effective. In these cases, hydration of water molecules around the hydrophilic head group become more important than that of the decrease of hydration around the hydrophobic alkyl chains of surfactant monomers.[39-40,42] Positive values of ΔH^0 may arise when the hydrophobic interactions are stronger and due to the transfer of hydrophilic head groups from the aqueous environment into micelle core.[43] For Amr – CTABr system, significant negative value of ΔH^0 for binding process indicate that the electrostatic interactions play a major role. The binding process is also accompanied with negative entropy (ΔS^0) as the temperature increases. The values of ΔH^0 and ΔS^0 indicate that binding process of Amr to CTABr pre-micelles are enthalpy controlled over the range of studied temperatures.

4. Conclusions

The addition of CTABr does not change the characteristic absorption peaks of Amr but decrease in intensity, indicating interactions between Amr and CTABr molecule and formation of ion pair

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complex. By measuring the absorbance of Amr in different solutions as a function of temperature and using the appropriate association models to analyze the experimental data, the association binding constant K_b and thermodynamic parameters were obtained. The results show that the absorbance value of dye increases with the addition of CTABr within the pre-micelle region. The increase in the temperature of solution causes a reduction in the binding constant values of Amar – CTABr system due to the weakening of interactions between dye and surfactant molecules at high temperatures. The negative values for the thermodynamic parameters ΔG^0, ΔS^0 and ΔH^0 pointed out that the binding of Amar to CTABr are spontaneous in nature and enthalpy driven.

Conflicts of Interest

The authors declare no conflict of interest

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