Thermodynamic Analysis of Dye-Surfactant Interactions in Aqueous Solution Using Conductometric and Surface Tension Techniques

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ABSTRACT

The interaction between anionic dye C.I. Acid Orange 5 (AO5) and anionic surfactant sodium dodecyl sulfate (SDS), along with the effect of increasing the proportion of the dye in the surfactant solutions as well as the impact of increased temperature on these interactions were examined using both conductometric and surface tension techniques. The results revealed that the Critical Micelle Concentrations (CMC) have reflected a tendency towards increasing in the values with increasing in the proportion of the dye in the surfactant solutions. Correspondingly, the CMC values of the dye-surfactant mixture solutions increased with the increase of temperature. A theoretical model of calculations has been implemented to measure the standard Gibbs free energy changes of micellization, ΔG^{0}_{mic}, the standard enthalpy changes of micellization, ΔH^{0}_{mic} , and the standard entropy changes of micellization, ΔS^{0}_{mic}. Moreover, the interactions of dye-surfactant are spontaneous based on the negative values of $\Delta G^{0}_{mic}$for all systems. Significantly, the negative values of ΔH^{0}_{mic} revealed that the interactions of dye-surfactant system are exothermic. The entropy contribution of micelle formation ΔS^{0}_{mic} shows positive values.

Key Words: Conductivity; Dye; Micellization; Surface Tension; Surfactant.

INTRODUCTION

Surfactants have been widely used as auxiliaries in many areas of textile finishing. Study of the interactions between dyes and surfactants in aqueous solution is necessary to fully understand the mechanism of finishing procedures and to select the correct auxiliary for dyeing and finishing. Fundamental to this is the understanding of the interactions that could occur between the dye and the surfactant as well as the influence of the dye and the surfactant structure (Datyner, 1993).

Dyes can be classified as arylmethane, anthraquinone, azo, acridine, cyanine, phthalocyanine, nitro, nitroso, quinoneimine, thiazole or xanthene dyes according to the type of chromophore group on the dye molecule (Duman *et al*., 2011), dyes may also be sorted according to their solubility to soluble dyes like acid, basic, metal complex, direct, mordant and reactive dyes; or insoluble dyes including sulfur, azoic, vat and disperse dyes (Vesna *et al*., 2013). Almost 10^9 kg of dyes are produced annually in the world, of which azo dyes represent about 70% by weight. These large quantities of azo dyes are being produced to be daily used for leather tanning, textile, paper production, food industry, etc. (Elwakeel, 2009).

Surfactant is an abbreviation for Surface Active Agent which literally means active at surface. In other words, a surfactant is characterized by its tendency to absorb at surface and interfaces (Holmberg *et al*., 2002). Surfactants are composed of a hydrophilic surface and hydrophobic core in water media. This specific structure enables them to establish chemical interactions with either hydrophilic or lipophilic molecules (Hosseinzadeh *et al*., 2008). They are characterized by a wide variety of aggregates morphology formed by the self-assembly of surfactant molecules in aqueous solution to form micelles where their properties are different from those of the non-aggregated monomer molecules. The concentration at which micelles appear in solution is termed Critical Micelle Concentration (CMC). The formation of micelles was often characterized by discontinuity in system properties such as conductivity, surface tension, light scattering, self-diffusion and the molality of dissolved compounds. Experimentally, CMC can be determined from the inflection plots of some physical property of the solution as a function of concentration. CMC can serve as a measure of micelle stability in a given state and the thermodynamics of micellization can be determined from a study of the CMC-temperature dependence (Rosen, 1989 and Tadros, 2005).

Amphiphilic properties of surfactants have attracted growing attention for their use in biological and chemical research applications especially in the dyeing process where the role of surfactants is very important (Ghoreishi, 2009). Many studies on the interactions between dyes and surfactants in aqueous solutions have been performed, especially in dyeing systems, where level dyeing is controlled by surfactants (Novicka and Nowicki, 1994, Simoncic and Span, 1998 and Yamamoto, 1992). Ionic surfactants, which are used as leveling agents, operate in two main mechanisms depending on the ionic type of the dye (Datyner, 1983 and Shore, 1990). When the charge of the surfactant is opposite of that of a dye, the attractive forces between the dye and surfactant lead to dye-surfactant complex formation in the solution, resulting in level dyeing. In cases where the surfactant is of the same ionic type as the dye used, competition between the dye and the surfactant for the sorption sites in the fiber causes level dyeing (Simoncic and Kovac, 1999).

Tanaka *et al*. (1994) used two monoazo acid dyes, C. I. Acid Orange 7 (Orange II) and C. I. Acid Yellow 36 (Metanil Yellow) with sodium dodecyl sulfate (SDS and dye are uni-univalent strong electrolytes and have a common cation). Adsorbed film and micelle compositions were determined by applying thermodynamic equations to the experimental results. Orange II molecules were forced more strongly than Metanil Yellow molecules to move away from the adsorbed film and micelle due to the presence of SDS molecules. In the dyeing process of nylon, it has been known that the anionic surfactant acts more efficiently as a leveling agent in the Orange II system than in the Metanil Yellow system.

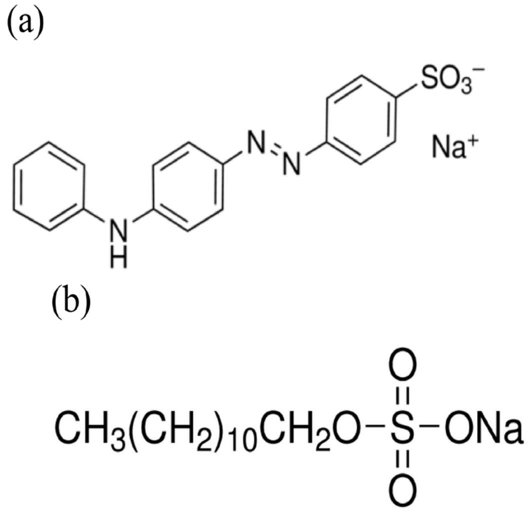
The aim of this study is to examine the interaction between anionic dye C.I. Acid Orange 5 (AO5) and anionic surfactant sodium dodecyl sulfate (SDS), as well as to investigate the effect of increasing the proportion of the dye in the surfactant solutions and the impact of increasing temperature on these interactions. Both conductometric and surface tension techniques were implemented to confirm the dye-surfactant interaction. In order to calculate CMC values, a series of conductance and surface tension measurements were performed at three different temperatures, at pure surfactant, and at different percentage of % dye-surfactant. The critical micelle concentrations are calculated to identify the interaction between dye and surfactant, furthermore, to determine the thermodynamic functions.

MATERIALS AND METHODS

Materials

The anionic dye C.I.Acid Orange 5 (AO5), sodium 4-[[4-(phenylamino) phenyl] azo] benzenesulfonate, $Na(C_6H_5NHC_6H_4N{=}NC_6H_4SO_3)$, common name, “Tropaeolin OO”, C.I.number (13080) was obtained from Sigma chemicals® and used without further purification. The anionic surfactant sodium dodecyl sulfate (SDS), $CH_3(CH_2)_{11}OSO_3Na$, was obtained from Panreac®. The chemical structure of dye and surfactant used in this study are given in Fig (1)

Figure (1) Molecular structure of (a) Acid



$$CH_3(CH_2)_{10}CH_2O-\overset{\overset{\displaystyle O}{\|}}{\underset{\underset{\displaystyle O}{\|}}{S}}-ONa$$

Orange 5; (b) Sodium Dodecyl Sulfate.

Preparation of solutions

All stock solutions of dye and surfactant were prepared in doubly distilled water. Stock solution of dye was $1x10^{-3}$ M (375.4g/mol, 0.3754g). Stock solution of surfactant was $1x10^{-2}$ M (288.372 g/mol, 2.8837 g). Surfactant solutions above and below the CMC of surfactant are necessary to correct determination of CMC. 5, 10, 15% (v/v) of dye–surfactant solutions were prepared. These solutions are placed on a constant temperature water bath before measurements at 298.15, 308. 15, and 318.15K.

Instruments

The electrical conductivity was measured with a conductometer; model B36664 from Thermo Scientific®, and the conductivity cell was calibrated with KCl solution in the appropriate concentration range. The cell constant was $0.10cm^{-1}$.

Surface tension measurements were done by applying du Nouy ring method using a Sigma 700 from Attention® model tensiometer and platinum ring.

Water bath Lauda CS-C20 Circulating Bath. Control accuracy is $\pm 0.01C^0$.

Thermodynamics Measurements

There is a growing interest in determining the thermodynamic parameters of micellization in aqueous solutions, namely, standard Gibbs free energy of micellization, ΔG^0_{mic}, enthalpy of micellization, ΔH^0_{mic} and entropy of micellization, ΔS^0_{mic}, in order to know more about the process of micellization. These parameters can be derived from the temperature dependence of the CMC.. The standard Gibbs free energy changes, of micelle formation per mole, ΔG^0_{mic}, is given by:

$$\Delta G^0_{mic} = RT \ln CMC \qquad (1)$$

Whereas R and T are the gas constant and absolute temperature, respectively. The standard enthalpy micelle formation, ΔH^0_{mic}, can be obtained from the temperature variation of CMC by applying the Gibbs–Helmholtz equation to Eq. (1):

$$\Delta H^0_{mic} = -RT^2 dlnCMC \ / \ dT \qquad (2)$$

Once the Gibbs free energy and the enthalpy of micelle formation are obtained, obviously the entropy of micelle formation can be determined by:

$$\Delta S^0_{mic} = (\Delta H^0_{mic} - \Delta G^0_{mic})/T \qquad (3)$$

RESULTS AND DISCUSSION

CMC determination using the Conductivity Measurements

The specific conductance (κ) ($mS\ cm^{-1}$) in doubly distilled water for pure surfactant SDS and mixture of (5, 10, 15% AO5–SDS) as a function of surfactant SDS concentration at 298.15, 308.15, and 318.15K is illustrated in Fig.2. The results revealed two straight lines with different slopes. The first one corresponds to the concentration range below the CMC, when micelles start to form; the change of slope appears due to the increases in the conductivity in a different manner. The intersection of these two straight lines was considered as the CMC value of the surfactant.

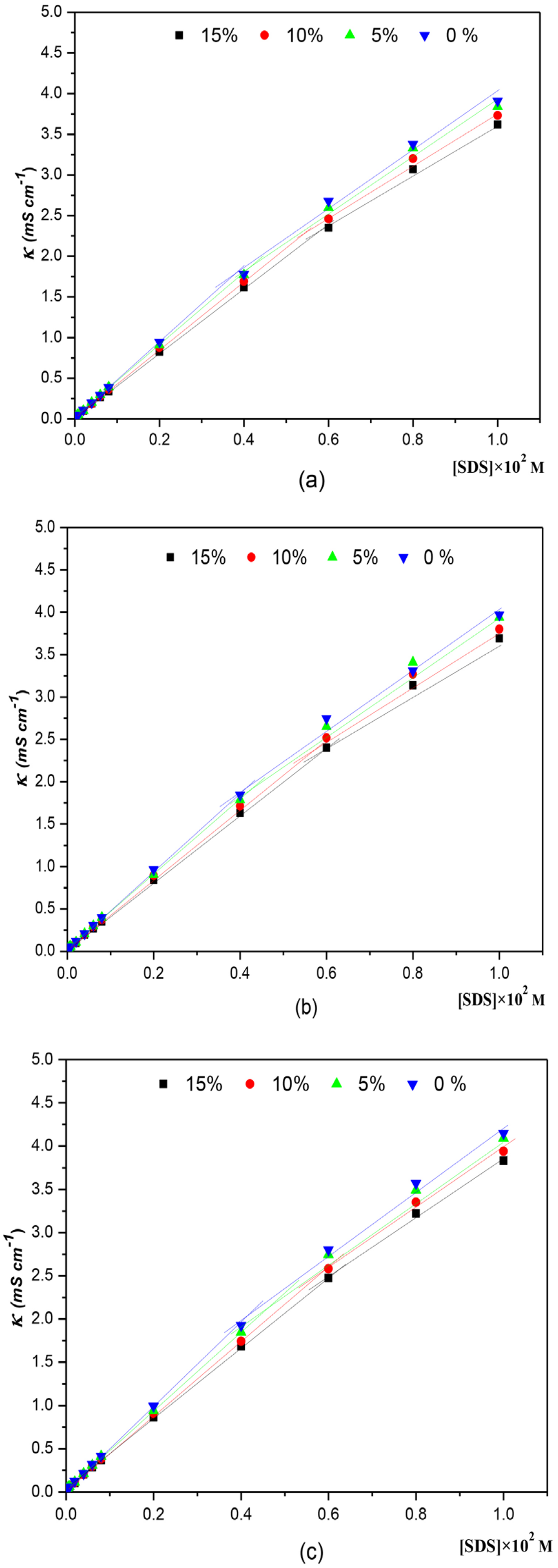


Figure (2) specific conductance (κ) (mS cm^{-1}) vis. concentration (M) for pure SDS and mixture of (5, 10, 15% AO5-SDS) at different temperatures (a) 298.15K, (b) 308.15K, (c) 318.15K.

As can be observed from Figure 2, the specific conductance curve deviated from linearity at certain concentration of surfactant and at certain temperature. This indicates that the components interacted to form corresponding ion pairs. These ion pairs are probably less-conductive or nonconductive species since the specific conductivity of the solution deviated from the linearity and varied as another line with a smaller slope (Rashidi-Alavijeh *et al.*, 2011 and Shahir *et al.*, 2011).

At constant temperature, Fig. 2 illustrates that the specific conductance values of the dye-surfactant mixtures decreased when % dye-surfactant increased. The decrease was attributed to the formation of a non-conducting or a less-conducting species in the solution (Dezhampanah and Firouzi, 2012). This behavior is due to interionic attraction. At low %dye-surfactant, the ions were relatively far apart. So, they exerted little influence upon one another. As %dye-surfactant is increased, the ions come closer to each other. The attraction between ions resulted in a decrease of their speeds and consequently in the specific conductance of the solution.

The values of the CMCs in aqueous solutions at various temperatures are shown in Table 1. CMC values increase with increasing the temperature for a given system. The increase in the CMC with respect to temperature is usually analyzed in two ways. First, the degree of hydration of the hydrophilic head groups decreased (when temperature increased), which favors micellization (and thereby decreased the CMC). Second, as the temperature an increase, the water structure around the hydrophobic group is gradually destroyed, which is unfavorable for micellization, and hence an increase in the CMC happens (Koya *et al.*, 2012 and Kumaraguru *et al.*, 2006). Therefore, we can assume that the dominant effect is the dehydration of the hydrophobic group which results in increasing CMC values.

Table (1)
Thermodynamic parameters for pure SDS and mixture of %AO5-SDS, using conductance and surface tension techniques.

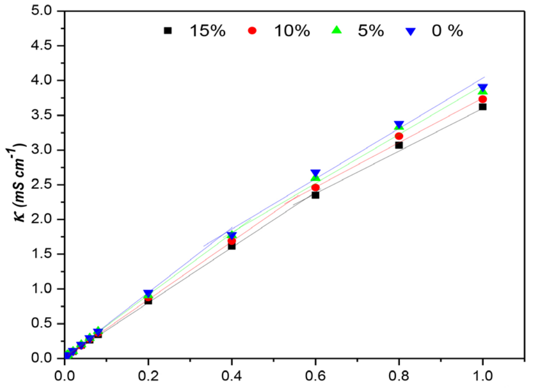
%AO5	T	Conductance technique					Surface tension technique				
		CMCx10^3	ΔG^{o}_{mic}	ΔH^{o}_{mic}	ΔS^{o}_{mic}	T. ΔS^{o}_{mic}	CMC x10^3	ΔG^{o}_{mic}	ΔH^{o}_{mic}	ΔS^{o}_{mic}	T. ΔS^{o}_{mic}
0	298.15	3.906	-13.746	-2.735	0.0369	11.011	4.066	-13.646	-1.256	0.042	12.390
	308.15	4.201	-14.020	-2.921	0.0360	11.099	4.178	-14.034	-1.342	0.041	12.692
	318.15	4.205	-14.473	-3.114	0.0357	11.359	4.205	-14.473	-1.431	0.041	13.042
5	298.15	4.513	-13.388	-5.026	0.0280	8.362	4.508	-13.390	-9.090	0.014	4.300
	308.15	4.882	-13.635	-5.368	0.0268	8.267	4.798	-13.680	-9.710	0.013	3.969
	318.15	5.170	-13.926	-5.722	0.0258	8.204	5.760	-13.640	-10.351	0.010	3.289
10	298.15	5.584	-12.860	-2.291	0.0354	10.569	5.621	-12.843	-2.291	0.035	10.552
	308.15	5.822	-13.184	-2.447	0.0348	10.737	5.881	-13.158	-2.447	0.035	10.711
	318.15	5.947	-13.556	-2.609	0.0344	10.947	6.283	-13.410	-2.609	0.034	10.802
15	298.15	5.921	-12.714	-1.922	0.0362	10.793	5.930	-12.711	-1.922	0.036	10.789
	308.15	6.046	-13.087	-2.053	0.0358	11.035	6.266	-12.996	-2.053	0.036	10.943
	318.15	6.239	-13.429	-2.188	0.0353	11.241	6.241	-13.428	-2.188	0.035	11.240

Units: T (K); CMC (M); ΔG^{o}_{mic}, ΔH^{o}_{mic}, (kJ mol^{-1}); ΔS^{o}_{mic} (kJ mol-$^{1}K^{-1}$); T. ΔS (kJ mol^{-1}).

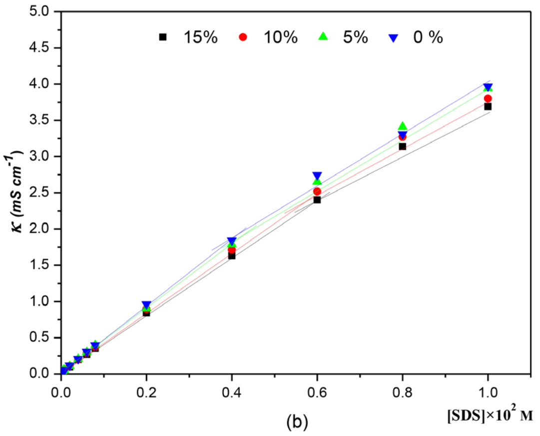
CMC determination using the surface tension Measurements

The surface tension (γ) (mN m^{-1}) of pure surfactant and mixture of (5, 10, 15% AO5-SDS), is plotted as a function of natural logarithm surfactant SDS concentration at 298.15, 308.15, and 318.15K (Fig.3). The surface tension (γ) of surfactant was measured for a range of concentrations above and below the critical micelle concentration, and the CMC values were determined at sharp break point (Table 1).

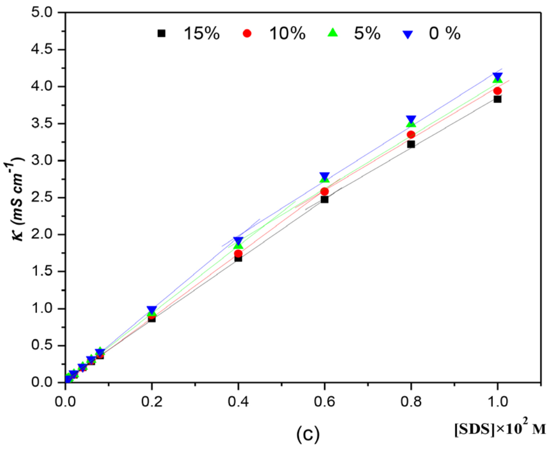
A linear decrease in surface tension was observed with the increase in surfactant concentration for the aqueous pure surfactant and mixture solutions as illustrated in Fig.3 .This is a common behavior by surfactants in solution and is used to determine their purity and CMCs.



(a)



(b)



(c)

Figure (3) Surface tension (γ) (mN m^{-1}) vis. concentration (M) for pure SDS and mixture of (5, 10, 15% AO5-SDS) at different temperatures (a) 298.15K, (b) 308.15K, (c) 318.15K.

Continued Figure (3) Surface tension (γ) (mN m^{-1}) vis. concentration (M) for pure SDS and mixture of (5, 10, 15% AO5-SDS) at different temperatures (a) 298.15K, (b) 308.15K, (c) 318.15K.

The data in Table (1) indicated that, in the temperature range studied, the CMC values increased as the temperature increased. This may be due to the fact that the surfactants molecules dissolved in water; the hydrophobic group distorted the water structure. The increase in temperature also caused increase breakdown of the structured water surrounding the hydrophobic group that disfavors micellization (Li-Jen *et al*., 1998).

The result obtained from the measurements of CMC corresponded with those previously obtained from specific conductance measurements. A valid evidence for such correspondence is that at 308.15K, the CMC values measured by conductivity for pure surfactant and 5, 10, 15 % dye-surfactant were 4.201, 4.882, 5.822, 6.046, respectively, which are almost comparable of those obtained by surface tension technique (4.178, 4.798, 5.881, 6.266) x10^{-3} M, respectively. Likewise, the CMC values (with given percentage at all temperatures) were found to be in agreement with measured specific conductivity as shown in Table1. Consequently, coincide results of conductometric and surface tension techniques confirmed the interaction between dye and surfactant, and the effect of increasing the proportion of dye and the temperature.

Thermodynamics Parameters of Micellization

In all cases studied, the values of Gibbs free energy of micellization as shown in table 1 are negative, indicating spontaneous micelle formation. Furthermore, results revealed that the values of Gibbs free energy of micellization become slightly more negative as the temperature increased, reflecting that the dehydration of surfactant molecules is predominant factor in the formation of micelles at higher temperature. Whereas, a reverse trend is observed when increasing of ٪dye-surfactant demonstrating that the process of micellization became less spontaneous (He *et al*., 2014).

Obviously, the enthalpy of micellization ΔH^0_{mic} is negative and slightly dependent on temperature, and becomes more negative in 5%dye-surfactant; this is due to the repulsion between common ion of dye and surfactant which is less comparing with the high concentration of dye. The negative values signifying that the complex formation processes of dye-surfactant system are exothermic (Dezhampanah and Firouzi, 2012).

The entropy contribution of micelle formation ΔS^0_{mic} represented positive values in all temperature range and decreased with increase in temperature. This is due to the fact that the head group is more hydrated than the hydrophobic tail with increasing temperature, which leads to an overall ordering of the system hence, lowering of the entropy with the increase in temperature (Owoyomi *et al*., 2011). TΔS^0_{mic} is large and positive; validating that, in the micellization process, there is a net increase in entropy (Mohajeri and Noudeh, 2012).

CONCLUSION

Conductometric and surface tension methods were used to investigate the interaction between the dye and surfactant. The Critical Micelle Concentration (CMC) revealed a tendency for increasing with increasing in %dye-surfactant, as well as increasing in temperature, for all mixture compositions system. Additionally, the CMC values were in agreement with measured specific conductivity and surface tension techniques. The Gibbs free energy of micellization ΔG^0_{mic} was negative in all cases; which meant that the interactions of dye-surfactant were spontaneous and became less negative with increased ٪dye – surfactant and more negative with increased temperature. The negative values of ΔH^0_{mic} signified that the complex formation processes of dye-surfactant system were exothermic. The entropy contribution of micelle formation ΔS^0_{mic} showed positive values.

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تحليل ديناميكي حراري لتداخل صبغة – مادة نشطة سطحياً في وسط مائي باستخدام تقنيتي التوصيلية و التوتر السطحي

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الملخص

تهدف هذه الدراسة إلى فحص التداخلات المختلفة بين صبغة الآزو الأنيونية الحمضية البرتقالية 5 (AO5) والمادة الأنيونية النشطة سطحياً دوديسيل كبريتات الصوديوم (SDS)، ومدى تأثير زيادة نسبة الصبغة على محاليل المادة النشطة سطحياً وكذلك تأثير زيادة درجة الحرارة على هذه التداخلات؛ باستخدام تقنيتين إحداهما التوصيلية، والأخرى التوتر السطحي. بينت النتائج الميل لزيادة التراكيز الحرجة لمخفض التوتر (CMC) بزيادة نسبة الصبغة إلى المادة النشطة سطحياً، وكذلك بالمثل بزيادة درجة الحرارة عند جميع النسب المدروسة. تم حساب دوال الديناميكا الحرارية، التغير في طاقة جبس الحرة لمخفض التوتر (ΔG^0_{mic})، التغير في الجهد الديناميكي الحراري لمخفض التوتر (ΔH^0_{mic})، وكذلك التغير في انتروبي مخفض التوتر (ΔS^0_{mic}). ولقد بينت النتائج أنّ التداخل بين الصبغة والمادة النشطة سطحياً تلقائياً لجميع الأنظمة المدروسة، وهذا يتضح من القيم السالبة للتغير في طاقة جبس الحرة منخفض التوتر. كما أنّ هذه التداخلات طاردة للحرارة والتي تبرهنها القيم السالبة للتغير في الجهد الديناميكي الحراري لمخفض التوتر. ويتضح من الدراسة أنّ قيم الانتروبي الراجعة لنظم خفض التوتر كانت إيجابية.

الكلمات المفتاحية: التوتر السطحي، التوصيلية، الصبغة، مادة نشطة سطحياً، منخفض التوتر.