

Kinetic and equilibrium properties of foam films stabilized by mixtures of *n*-dodecyl- β -D-maltoside with nonionic or ionic surfactants

D. S. Ivanova^{1*}, J. K. Angarska¹, E.D. Manev²

¹Faculty of Natural Sciences, Konstantin Preslavsky University of Shumen, Universitetska str. 115, 9712 Shumen, Bulgaria

²Faculty of Chemistry and Pharmacy, Sofia University, James Bourchier Blvd. 1, 1164 Sofia, Bulgaria

Submitted on July 9, 2015; Revised on October 5, 2015

Experimental data for kinetic and equilibrium parameters for foam films, stabilized by mixtures of *n*-dodecyl- β -D-maltoside (C₁₂G₂) with hexaethyleneglycol monododecyl ether (C₁₂E₆), dodecanol (C₁₂E₀), and dodecyl trimethylammonium bromide (C₁₂TAB) are obtained interferometrically. The influence of mixture composition and total surfactant concentration on the film thinning, film type and its stability is investigated at low (0.001M) and high (0.1 M) ionic strength. The results follows that: (i) the film stability strongly depends on the surfactant nature at low ionic strength; (ii) the thinning rate for films from mixtures of 50:1 correlates with that for films stabilized by pure C₁₂G₂; (iii) for films from mixtures of 1:1 thinning rate is controlled by the component dominating in the adsorption layer - for films from mixtures of C₁₂G₂ + C₁₂E₆ it is determined primarily by C₁₂E₆, while for the C₁₂G₂ + C₁₂TAB mixtures – by both components.

Keywords: foam films; *n*-dodecyl- β -D-maltoside, hexaethylene glycol dodecyl ether, dodecyl trimethylammonium bromide, film thickness; velocity of film thinning.

1. INTRODUCTION

Since in the practice colloids are usually stabilized by mixtures of surfactants, investigations of films in the presence of such surfactant mixtures are of primary importance. The present study comprises the non-ionic surfactant *n*-dodecyl- β -D-maltoside (C₁₂G₂ or G₂) and its mixtures with the nonionic hexaethyleneglycol monododecyl ether (C₁₂E₆ or E₆), or dodecanol (C₁₂E₀ or E₀), and the ionic surfactant dodecyl trimethylammonium bromide (C₁₂TAB or TAB). The choice of these surfactants, possessing the same hydrophobic tail, but with different head groups, is not accidental - they have the following special properties: (i) sugar-based surfactants are non-toxic, biodegradable, insensitive to temperature, pH, and are not affected by the concentration of Ca and/or Mg ions in the water. Information about the advantages of these surfactants as an alternative to some widely used nonionic surfactants is given in Refs. [1-3]; (ii) ethylene oxides (C_iE_j) surfactants are widely used in the practice as stabilizers of colloidal systems, because of their low surface tension, CMC and foamability, as well as in mixture with other surfactants [3]; (iii) small traces of dodecanol in many commercial surfactants as impurity, its strong adsorption on interface at low

adsorption of some ionic surfactants at interface [4] as well as to increase the surface elasticity [5] and viscosity [6] make it an interesting additive; (iv) C₁₂TAB is often used as a stabilizer of foams and foam films [4, 7, 8] as well as an additive to nonionic surfactants [9-11]. Varying its concentration in the individual solution or its content in the mixture allows the surface charge on the film surfaces to be controlled. Furthermore, the investigation of mixtures with C₁₂TAB is of importance because of their antibacterial, antifungal and antiseptic action.

From the literature review, it is clear that there is a significant interest to the use of C₁₂G₂ and its mixtures as stabilizers of thin films. Their equilibrium [1,2,8,9] and kinetic [11-14] properties are studied intensively. Moreover, in these papers there is no convincing conclusion which surfactant in the mixture has the dominant role in the film behavior. Such a conclusion could be reached if the properties of the films from mixtures are compared to those of films with individual surfactants.

The above considerations determine the aims of the presented study:

- to test and discuss the influence of the surfactant type and concentration, ionic strength and molar ratio of the components in the mixture on the equilibrium state and velocity of thinning for films from mixed surfactant solutions: of C₁₂G₂ with the nonionic C₁₂E₆, or C₁₂E₀ and the cationic C₁₂TAB;

*To whom all correspondence should be sent:
E-mail: D.Ivanova@shu-bg.net

volume concentration and ability to compete the

- to compare the drainage rate of films from the mixtures to that of films from solutions of the individual surfactants, in order to differentiate their characteristics.

2. EXPERIMENTAL PART

2.1. Materials

The nonionic surfactants: *n*-dodecyl- β -*D*-maltoside (C₁₂G₂, purity>99.5%), purchased from Glycon; dodecanol (C₁₂E₀), purchased from Merck; hexaethyleneglycol monododecyl ether (C₁₂E₆), purchased from Fluka, were used without further purification. The cationic dodecyl trimethylammonium bromide (C₁₂TAB, purity >98%) was purified by a procedure given in [10]. Sodium chloride (NaCl) and sodium bromide (NaBr) were obtained from Merck and Sigma, respectively. They were roasted for 4h at 400°C to remove the organic contaminants.

All glassware was cleaned with concentrated chromic sulphuric acid and then was rinsed thoroughly with double distilled water before use. All solutions were prepared with double distilled water immediately before each experiment at 25°C \pm 1°C.

2.2. Experimental conditions

In accord with the aim of the presented study, experiments were performed with foam films of different radii, obtained from mixed solutions of varied total surfactant concentration, molar ratios of the surfactants and ionic strength. In order to compare the results for different mixtures, all concentrations are given in terms of their CMC, chosen as a reference. The used CMC values are given in [15]. The equilibrium surface tension (see Figs. 1-2) of mixed solutions, from which the foam films were formed, was measured by the Wilhelmy plate method.

2.3. Interferometric method for foam films investigation

From each solution of defined composition at least 50 foam films of different radii (from 0.05 to 0.15 mm) were obtained in a glass cell (with radius of the holder 2.15 mm) designed by Scheludko and Exerowa [16]. Each film was visually observed in reflected monochromatic light (wavelength, $\lambda = 551$ nm, 6.3 \times objective lens and 5 \times eye-piece) immediately after filling the holder of cell with solution, but its evolution was recorded (by Sony-SSC-M388CE photo camera) when the equilibrium state in the system was reached (after 1 hour). The film thicknesses at every one moment as well as equilibrium thickness (h_{weq}) were determined by a procedure described in details in [12]. The real equilibrium thickness (h_{eq}) was obtained by

correcting of h_{weq} value based on five (for C₁₂G₂ + C₁₂E₆), or three (C₁₂G₂ + C₁₂TAB) layers model of film structure [13,11]. The values of equilibrium thicknesses are presented in Table.1. They were used for calculating of the surface potential by the equilibrium method [17].

2.4. Determination of the film thinning velocity

For velocity of thinning in [18] is used the empirical equation (Eq.1) and it is shown that its form (Eq.2) yields a linear dependence whose slope gives the value of the drainage coefficient α

$$-dh/dt = \alpha h \quad (1),$$

$$\ln h = \ln h_0 - \alpha t \quad (2),$$

where: h is the current thickness; h_0 is the initial thickness (after the maximum in the kinetic curve), from which the time of thinning t from h_0 to h is measured.

Base on the linear dependence $\ln h$ vs. t the drainage coefficients for all investigated films were received (for details see Refs.12,13,15). It is clear that the thinning velocity expressed through α is obtained from the experimental $h(t)$ data, it is not derived from any of the models. We have employed the empirical parameter α for several worthy reasons; namely, it: (i) gives the real experimental thinning rate; (ii) accounts for all known and unknown factors, which affect film thinning; (iii) yields reliable information about the film thinning rates in the relevant thickness range below 100 nm.

3. RESULTS AND DISCUSSION

In this study we present experimental data for the equilibrium parameters and kinetic behavior of films from mixed solutions of C₁₂G₂ with non ionic surfactants C₁₂E₆ and C₁₂E₀ or the ionic C₁₂TAB. The results are systematized in two groups depending on the ionic strength of the solutions.

3.1 Equilibrium properties of foam films

Films, obtained from solutions with low ionic strength (0.001M). The experiments under these conditions allow checking the role of surfactant adsorption on the surface charge density, as well as the influence of the electrostatic repulsion on the equilibrium state and velocity of film thinning. From data presented in Table 1 the following conclusions can be extracted:

✓ Below 0.5 CMC all films are stable except the films stabilized by mixtures C₁₂G₂: C₁₂E₀ = 50:1 and C₁₂G₂: C₁₂TAB = 50:1.

✓ At 1 CMC are obtained several types of films: (i) stable CF from C₁₂E₆, C₁₂G₂: C₁₂E₆ = 1:1 and C₁₂G₂: C₁₂TAB = 50:1 solutions; (ii)

metastable CF, or NBF from $C_{12}G_2$ and $C_{12}G_2:C_{12}E_6 = 50:1$ solutions; (iii) NBF from $C_{12}G_2:C_{12}E_0=50:1$ solution.

Decreasing of the equilibrium thickness with an increase of the surfactant concentration, is due to the increasing of surfactant adsorption, which leads to a decrease of the surface charge. It was found that some films obtained from solutions of $C_{12}G_2$ or $C_{12}G_2:C_{12}E_6 = 50:1$ at concentration 1CMC, spontaneously jump into NBF, while some films become equilibrium CF. It means that these films are in a metastable state. The stability of the obtained CF can be connected with an electrostatic force, while the stability of NBF - with steric repulsive force.

The films, obtained from the mixed $C_{12}G_2:C_{12}E_0= 50:1$ solution, with concentration up to 0.5 CMC are unstable, which can be explained with the influence of $C_{12}E_0$ as an antifoaming agent. According to [5] the foams produced from dodecanol solution are unstable. Because of the

dodecanol high surface activity, even at low concentration, it predomines in the adsorption layer, which probably leads to a decrease in the film stability. When the total surfactant concentration of the $C_{12}G_2:C_{12}E_0= 50:1$ solution is above 0.5 CMC, the obtained films are stable NBF. At this condition their surfaces are covered predominantly of $C_{12}G_2$ molecules.

The films formed from a mixed solution $C_{12}G_2 : C_{12}TAB = 50:1$ up to 1CMC are unstable, too. It follows that a small addition of cationic surfactant leads to a full reset of the surface charge. In the films, from solutions at 0.5 CMC, mobile black spots appear which very quickly merger and rupture the film. It means that the adsorption of $C_{12}G_2$ under these conditions is high enough, but it is not sufficient to stabilize the film. The films formed of this mixture but at 1 CMC are equilibrium CF and they jump into NBF but only after some contraction.

Table 1.Type, stability, equilibrium thickness (nm) and surface potential (φ_0 , mV) for films obtained from single or mixed surfactant solutions with low and high ionic strength.

Ionic strength 0.001M			
	below 0.5 CMC	0.5 CMC	1.0 CMC
$C_{12}G_2$	CF (59.7 nm) $\varphi_0 = 44.6$ mV	CF (51.8 nm) $\varphi_0 = 23.9$ mV	CF (42.5 nm), $\varphi_0 = 14.1$ mV NBF (6 nm)
$C_{12}E_6$	CF (50.1 nm) $\varphi_0 = 22.7$ mV	-	CF (44.0 nm) $\varphi_0 = 14.4$ mV
$C_{12}TAB$	-	-	CF (28 nm) at $I = 0.15M$
$C_{12}G_2:C_{12}E_6=50:1$	CF (56.25 nm) $\varphi_0 = 31.9$ mV	-	CF (31.5), $\varphi_0 = 9.0$ mV NBF (6 nm)
$C_{12}G_2:C_{12}E_6=1:1$	CF (57.1 nm) $\varphi_0 = 33.1$ mV	CF (59.0 nm) $\varphi_0 = 31.8$ mV	CF (45 nm) $\varphi_0 = 15.0$ mV
$C_{12}G_2:C_{12}TAB=50:1$	unstable	unstable	CF (50.0 nm), $\varphi_0 = 19.5$ mV NBF (6 nm)
$C_{12}G_2:C_{12}E_0 = 50:1$	unstable	unstable or NBF	NBF (6 nm)
Ionic strength 0.1M			
$C_{12}G_2$	unstable	NBF (6.5 nm)	NBF (6.5 nm)
$C_{12}E_6$	unstable	CBF (12.0 nm)	CBF (12.0 nm)
$C_{12}TAB$	unstable	-	CBF (15.0 nm)
$C_{12}G_2:C_{12}E_6=50:1$	unstable	NBF (6.5 nm)	NBF (6.5 nm)
$C_{12}G_2:C_{12}E_6=1:1$	unstable	NBF (6.5 nm) CBF (13.0 nm)	CBF (12.0 nm)
$C_{12}G_2:C_{12}TAB=50:1$	-	-	NBF (6.7 nm)
$C_{12}G_2:C_{12}TAB=1:1$	unstable	-	CBF (16.0 nm) NBF (6.7 nm)
$C_{12}G_2:C_{12}TAB=1:50$	-	-	CBF (16.0 nm)

Films, obtained from solutions with high ionic strength (0.1M). At high ionic strength the stabilizing role of electrostatical disjoining pressure can be excluded and the film behavior could be connected with some appropriate non-DLVO forces. From data presented in Table 1 it follows:

✓ up to 0.5 CMC all films are unstable;

✓ at 1 CMC the obtained films are CBF (from $C_{12}E_6$; $C_{12}G_2:C_{12}E_6 = 1:1$; $C_{12}TAB$; $C_{12}G_2 : C_{12}TAB = 1:1$ and $1:50$ solutions), or NBF (from $C_{12}G_2$; $C_{12}G_2:C_{12}E_6 = 50:1$; $C_{12}G_2 : C_{12}TAB = 50:1$ solutions). The latter ones form by spontaneous transition from CF.

These results show that the film type depends on the surfactant type and the composition of the mixture. The films from the mixed solutions of $C_{12}G_2$ with $C_{12}E_6$ and $C_{12}TAB$ (at molar ratio 50:1) jump spontaneously into NBF, which is an evidence of the significant role of $C_{12}G_2$ on the film equilibrium state. Data for films from $C_{12}G_2 : C_{12}E_6 = 1:1$ solution at 0.5 CMC, show that the film reaches to different equilibrium states: NBF and CBF, which could be associated with a small difference in the predominant adsorption of the surfactants from the mixture on the film interfaces. At total concentration 1 CMC the obtained films are only CBF, which is a proof for the dominating role of $C_{12}E_6$ in the film surfaces. The impact of the competitive adsorption in the films from mixed solution of $C_{12}G_2 : C_{12}TAB = 1:1$ is strongly expressed. In the films produced from it, before

reaching of the equilibrium state of adsorption (in the first few films), Newtonian spots, arise which is an evidence of the dominant adsorption of $C_{12}G_2$ molecules. When the equilibrium state in the system is reached, the films have a thickness similar to that of films stabilized by single $C_{12}TAB$. It is a proof that in the films from a mixed solution 1:1 the surfaces are firstly covered by $C_{12}G_2$ molecules, partially replaced afterwards by $C_{12}TAB$ species through a competitive adsorption.

3.2. Influence of the surfactant type and the molar ratio of the components in the mixture on the velocity of film thinning

Comparison of the velocity of thinning for films from solutions of low ionic strength. Fig.1ab presents the values of the drainage coefficient α

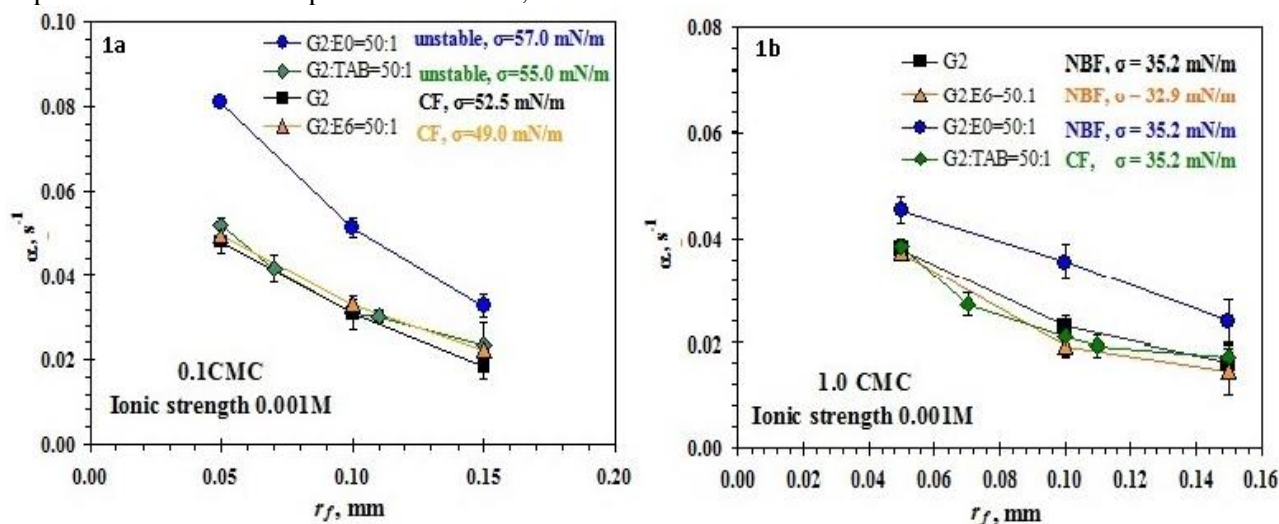


Fig.1. Drainage coefficient (α) of foam films stabilised by $C_{12}G_2$ or by its mixtures with three different additives - $C_{12}E_6$, $C_{12}E_0$ and $C_{12}TAB$ at ratio $C_{12}G_2$: additive = 50:1: (a) at 0.1 CMC; (b) at 1.0 CMC.

for films stabilized by $C_{12}G_2$ and its mixtures with $C_{12}E_6$, $C_{12}E_0$ and $C_{12}TAB$. The mixed solutions are at total surfactant concentration 0.1 CMC (a), or 1.0 CMC (b) and molar ratio $C_{12}G_2 : \text{additive} = 50:1$. From the dependences shown, it is clear that in both cases the rates of thinning of the films from the mixture with $C_{12}E_0$ are higher than those for $C_{12}G_2$ films. The dependences $\alpha(r_f)$ for films from the other mixtures have a similar trend. They are close to the values for individual $C_{12}G_2$ films. From data presented in Fig.1 it follows that:

✓ adding of $C_{12}E_0$ to the solution of $C_{12}G_2$ accelerates the film drainage which means that the films surfaces have greater capability of corrugation. We expected dodecanol to boost up the Gibbs elasticity and viscosity of the mixed surface layer with $C_{12}G_2$. Our measurements by the DSA method revealed that Gibbs elasticity increased

with the concentration, but only up to 0.5 CMC. Elasticity decreased above this level, to reach its minimum at CMC, which explains the accelerated thinning. Indeed, the measured Gibbs elasticity of mixed adsorption layers (at 0.1 or 1.0 CMC) is lower (46.6 and 26.3 mN/m) than that of $C_{12}G_2$ layers (50.1 and 67.0 mN/m);

✓ Adding $C_{12}E_6$ and $C_{12}TAB$ to the solution of $C_{12}G_2$ at molar ratio 50:1 does not substantially affect the film thinning, but it changes the film stability: the films from the mixture with $C_{12}TAB$ are unstable while those with $C_{12}E_6$ are stable.

Comparison of the velocity of thinning for films from solutions of high ionic strength. Fig. 2 presents juxtaposition of the coefficients of thinning for films from solutions of individual substances $C_{12}E_6$, $C_{12}E_0$ and $C_{12}TAB$ and their mixtures of different molar ratios. By comparing the data for individual surfactants with those for the

corresponding mixtures we aim to establish which of the substances in the mixture has the predominant effect on α . For mixtures with a molar ratio 50:1 the answer to this question is almost trivial, since $C_{12}G_2$ concentration is strongly prevailing. Of a particular interest is the mixture of molar ratio 1:1, for which the dominating component could not be predicted.

Fig. 2a presents data for α of films from mixed solutions from nonionic substances. It can be seen from that there are no large differences in the values of α , regardless of the composition. The

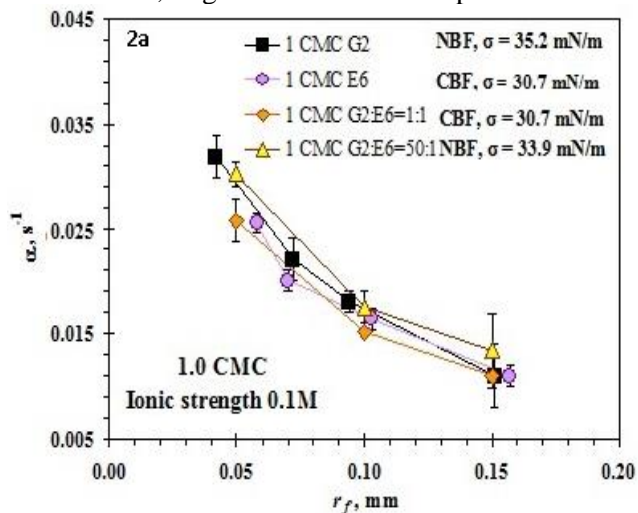
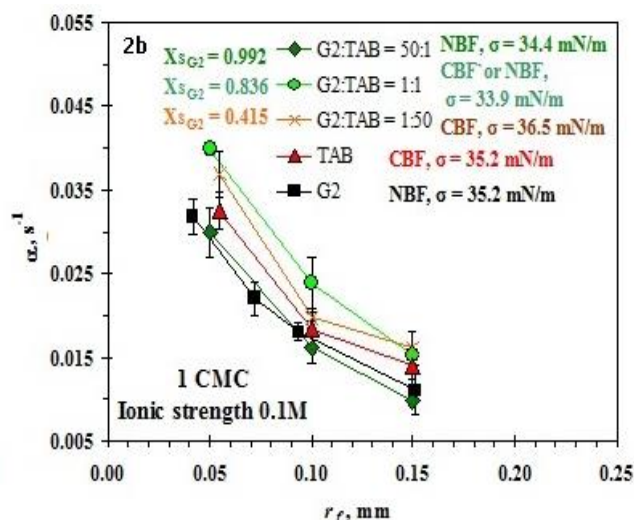


Fig.2 Comparison of the drainage coefficient (α) for foam films stabilized by individual surfactants and their mixtures of molar ratios 1:1 or 50:1: (a) for $C_{12}G_2 + C_{12}E_6$; (b) $C_{12}G_2 + C_{12}TAB$.

Fig. 2b presents data for α of films with close-packed surfaces (1.0 CMC) from a mixture of $C_{12}G_2$ and $C_{12}TAB$. The curves for films from mixtures of molar ratios 1:1 and 1:50 are situated higher than the curves for films from pure $C_{12}G_2$ and $C_{12}TAB$. The films formed from mixed solution 1:1 (after time, sufficient for attaining adsorption equilibrium) and 1:50 are CBF, as are the films from pure $C_{12}TAB$. According to Ref. [17] at these molar ratios the presence of $C_{12}TAB$ in the adsorption layer leads to positively (re)charged film surfaces. Indeed, the added electrolyte 0.1 M should have screened the positive charge, but the fact that such films are formed indicates the presence of some residual electric charge. This charge affects considerably the films equilibrium state, but to a lesser extent the velocity of thinning. That is why we connect the differences found for the thinning of films from these mixtures with the different composition of the adsorption layer, which determines its capability for corrugation. From the data for the molar part of $C_{12}G_2$ (see Fig. 2b) in the mixed adsorption layer (obtained by the procedure of Ingram [11]) is clear that in the film from a mixture with molar ratio 1:1

mixture with molar ratio 1:1 is with surface tension close to that of $C_{12}E_6$, but lower than that for $C_{12}G_2$. Consequently, $C_{12}E_6$ is the dominant component in this mixture. Accordingly, the α values of films from 1:1 mixtures should be similar to those of films from $C_{12}E_6$. Such a tendency emerges in Fig 2a, but only for films of small radii. Thus the films are divided into two groups, according to their drainage coefficients: from $C_{12}G_2$ solutions and a mixture of $C_{12}G_2:C_{12}E_6 = 50:1$; from $C_{12}E_6$ and a mixture of $C_{12}G_2:C_{12}E_6 = 1:1$.



$C_{12}G_2$ again has the dominant role in the interfaces. The molecular interaction parameter in the adsorption layer is evaluated equal to 2.29, which shows that the mixture does not behave as an ideal one. Although the content of $C_{12}TAB$ in the adsorption layer is (relatively) low, its presence there makes the layer non-homogeneous and reduces its coherence. The presence of domains of $C_{12}G_2$ and/or $C_{12}TAB$ modifies the adsorption layer corrugation capability, which we believe to be the reason for the higher values of drainage coefficient. Increase of the amount of $C_{12}TAB$ in the mixture 1:50 adds to the coherence in the layer and decelerates the film thinning.

5. CONCLUSIONS

In the presented study data of the kinetic and equilibrium properties for films from mixed solutions of $C_{12}G_2$ with the nonionic surfactants $C_{12}E_6$, or $C_{12}E_0$ and ionic $C_{12}TAB$ in the presence of electrolyte (0.001 and 0.1M) have been obtained and discussed. The experimental results follow that:

- at low ionic strength the film stability strongly depends on the surfactant nature;

➤ at high ionic strength, the influence of additive nature on the type of equilibrium films (CBF or NBF) manifest only when the films surfaces are covered;

➤ the velocity of thinning of films from mixtures at molar ratio 50:1 correlates with that of the films stabilized by single $C_{12}G_2$ solutions (see Fig.1-2); with the exception of films from mixture $C_{12}G_2:C_{12}E_0 = 50:1$, which was attributed to the low elasticity of the mixed adsorption layer;

➤ the rate of thinning of films from mixtures of molar ratio 1:1 depends on the impact of the species dominating in the adsorption layer; for a mixture of $C_{12}G_2 + C_{12}E_6$ it is determined by $C_{12}E_6$ (see Fig.2a), while for a mixture of $C_{12}G_2 + C_{12}TAB$ both components contribute substantially (see Fig.2b).

Acknowledgement: This paper is supported by two projects:

1. BG051PO001-3.3.06-0003 The Project is realized by the financial support of the Operative Program "Development of the human resources" of the European Social Fund;

2. RD - 08-255/09.03.2015 realized by the Scientific Fund of Konstantin Preslavsky University, Shumen, Bulgaria.

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КИНЕТИЧНИ И РАВНОВЕСНИ СВОЙСТВА НА ПЕННИ ФИЛМИ ОТ СМЕСЕНИ РАЗТВОРИ НА n-ДОДЕЦИЛ-β-D-МАЛТОЗИД С НЕЙОННИ И ЙОННИ ПОВЪРХНОСТНОАКТИВНИ ВЕЩЕСТВА

Д. Ст. Иванова^{1*}, Ж. Кр. Ангарска¹ и Е. Д. Манев²

¹Шуменски университет „Епископ Константин Преславски”, Факултет по природни науки

²Софийски университет „Свети Климент Охридски”, Факултет по химия и фармация

Постъпила на 9 юли, 2015 г.; коригирана на 5 октомври, 2015 г.

(Резюме)

Чрез интерферометричния метод са получени експериментални данни за кинетичните и равновесни параметри на пенни филми от смесени разтвори на n-додецил-β-D-малтозид ($C_{12}G_2$) с хексаетиленгликол монододецил етер ($C_{12}E_6$); додеканол ($C_{12}E_0$) и с додецил триметиламониев бромид ($C_{12}TAB$). Анализирани са влиянието на типа на ПАВ, молното им съотношение и тотална концентрация в сместа върху вида, стабилността и скоростта на изтъняване на филмите при ниска (0.001M) и висока (0.1M) йонна сила. Резултатите показват, че: (i) влиянието на природата на ПАВ върху стабилността на филмите е силно изразено при ниската йонна сила; (ii) скоростта на изтъняване на филмите от смеси с молно съотношение 50:1 е много близка до тази на филмите, стабилизирани само с $C_{12}G_2$; (iii) за филмите, получени от разтвори на смеси с молно съотношение 1:1, скоростта на изтъняване зависи от доминиращия компонент в адсорбционния слой - за сместа $C_{12}G_2 + C_{12}E_6$ - се определя от $C_{12}E_6$, докато за $C_{12}G_2 + C_{12}TAB$ - от двата компонента.