UDC 661.185.1

ABOUT THE RESULTS OF CALCULATION OF ADSORPTION FOR LIQUID-CRYSTALLINE NANODEMULSIFIERS ON THE BASIS OF THE OXIALKYLENE BLOCK COPOLYMERS

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Received 16.04.2018

To calculate the adsorption (Γ_i) of liquid-crystalline nanodemulsifiers on the basis of poly(ethylene oxide)–poly(propylene oxide) block copolymers on the liquid-gas interface, the Frumkin equation: $\Gamma_i = \Gamma_m [1 - \exp(\Delta\sigma_i / RT\Gamma_m)]$ was used. Correlation equations are proposed for calculating the minimum surface tension of block copolymers at maximum adsorption (Γ_m) depending on the molecular mass and the degree of hydroxyethylation of the corresponding oligomers, and a general adsorption isotherm in the coordinates $\Gamma_i - (-\Delta\sigma)$ is constructed and some comments, interpretations and conclusions based on the results of the research are presented.

Keywords: the Frumkin equation, adsorption at the liquid–gas interface, the liquid-crystalline nanodemulsifiers, surfactants.

Introduction

Recently, reagents in nanoliquid-crystal state on the basis of surfactants have been applied in oil production to increase oil recovery from reservoirs [1]. The authors of [2] for the first time have developed a liquid-crystal nanodemulsifier (LCND) for primary oil preparation (POP), i.e. for the destruction of water-oil emulsions such as water in oil (W/O). It is known that during oil production the water-oil [reverse (W/O)] and oil-water [direct (O/W)] emulsions are formed. In practice preparation of oil uses a thermochemical (temperature and demulsifiers) method to destroy oil emulsions. Consequently, the temperature and the characteristic of demulsifiers are the main physico-chemical parameters at POP for each oil deposit. In many cases poly(ethylene oxide)–poly(propylene oxide) block copolymers (PEOPPOBC) based on dihydric and polyhydric alcohols are used as the active phase of demulsifiers. In demulsifying processes, the effectiveness of demulsifiers along with other parameters is determined also by the adsorption properties of the main components of demulsifiers, depending on the degree of hydroxyethylation (α) and the molecular weight (M) of the molecules of the PEOPPOBC. The relationship between these parameters has been little studied. For calculation of adsorption isotherms, should be

used the most convenient expressions on the basis of known and generally accepted adsorption equations. It is known that one of the requirements imposed on modern generations of demulsifiers is the possibility to regulation the magnitude of the surface tension, which plays an essential role in the processes of destruction of oil emulsions. Therefore, calculation of adsorption isotherms for the main components of demulsifiers is of interest. Calculation of the adsorption isotherms at the liquid-gas interface can be realized by the Shishkovsky and Frumkin equations. To calculate the theoretical isotherms of the surface tension of the surfactants A.Abramson has proposed an expression with the transformation of the Shishkovsky equation [3, 4]:

$$\sigma_i = \sigma_0 - RT\Gamma_m \ln[(1 + (C_i\delta/\Gamma_m) \cdot \exp(W/RT))], \quad (1)$$

where C_i is the current concentration; Γ_m is the maximum adsorption; W is the adsorption work,

$$W = \beta \cdot 10^3 \cdot m, \quad (2)$$

where β is the Traube number $\beta=4.22$ [4]; 10^3 is the transition coefficient from kJ to J; m is the number of carbon atoms in alkyl radicals of non-ionic surfactants; σ_0 is the surface tension of water at 298 K (72.75 mJ/m²); δ is the thickness of the adsorption layer, $\delta \approx 0.9$ nm; σ_i – current surface tension corresponding to the current concentration value C_i ; Γ_m is determined by the method

of tangents. However the formula of the adsorption work (2) is not related to the adsorption itself and has a too simplified form. Therefore, studie of the adsorption properties of liquid-crystal nanodemulsifiers on the basis of oxy-alkylene block copolymers using the most acceptable and simple form of the adsorption equations have a particular topicality in the field of oil-field chemistry for the creation of highly effective demulsifiers in the oil emulsions.

Experimental and theoretical parts

Surface tension was determined by a stalagmometer according to the project of the USRI (Ufa Scientific Research Institute) [4]. To calculate the adsorption isotherms of the surface tension of the surfactant, the most convenient expression was used, by transforming the Frumkin equation:

$$\sigma_i = \sigma_0 + RT\Gamma_m \ln(1 - \Gamma_i/\Gamma_m) \quad (3)$$

The equation (3) solve with respect to Γ_i :

$$\ln(1 - \Gamma_i/\Gamma_m) = \Delta\sigma/RT\Gamma_m$$

$$1 - \Gamma_i/\Gamma_m = \exp(\Delta\sigma/RT\Gamma_m)$$

$$\Gamma_i = \Gamma_m[1 - \exp(\Delta\sigma/RT\Gamma_m)], \quad (4)$$

where

$$\Delta\sigma = \sigma_i - \sigma_0 < 0. \quad (5)$$

Block copolymers ethylene and propylene oxides on the basis of glycerin having a liquid-crystal state were investigated as nonionic surfactants. The minimal theoretical area ($\omega_{\min}$) at the liquid-gas interface per one surfactant molecule in a saturated monomolecular adsorption layer is determined by the cross-sectional area of the molecules of the solute [3–5]. For block copolymers of oxides of ethylene and propylene based on glycerol (BCOEPG) $\omega_{\min} = 78 \cdot 10^{-20} \text{ m}^2$ [6], then the corresponding value of the maximum theoretical adsorption ($\Gamma_{m(th)}$) is determined by the generally accepted formula [7]:

$$\Gamma_{m(th)} = 1/(\omega_{\min} \cdot N_A) = 2.130 \cdot 10^{-6} \text{ mol/m}^2,$$

where N_A is the Avogadro number. Isotherms are often calculated at $T = 298 \text{ K}$. Therefore, equations (4) for the calculation of the adsorption of BCOEPG can be presented in a more

simplified form:

$$\Gamma_i = 2.130 \cdot 10^{-6} [1 - \exp(\Delta\sigma \cdot 0.189)]. \quad (6)$$

Results and discussion

The experimental maximum values of the adsorption value (Γ_{im}) for the BCOEPG and the corresponding deviation Γ_{im} from $\Gamma_{m(th)}$ are also of interest. Let us consider the extreme conditions for equation (6). At $\Delta\sigma = 0$ (i.e. there is no surfactant in the system) $\Gamma_i = 0$, and in the critical state of the BCOEPG, when there are no interfaces between the liquid and the gas $\sigma_i = 0$ [8], $\Delta\sigma = 0 - 72.75 = -72.75 \text{ mJ/m}^2$, then $\Gamma_i \approx 2.130 \cdot 10^{-6} \text{ mol/m}^2$. The solution of equation (6) at $\sigma_i = 0$ has only theoretical value, it will be more correct to comment on the corresponding extreme condition as follows: at $\sigma_i \rightarrow 0$, $\Gamma_i \rightarrow \Gamma_m = 2.130 \cdot 10^{-6} \text{ mol/m}^2$. Consequently, Γ_i for BCOEPG practically reaches its maximum theoretical value $\Gamma_{m(th)}$, when under research the systems by properties are closer to the critical state of liquids. For calculation from equation (6) the maximum experimental values of adsorption (Γ_{im}) for each BCOEPG [depending on the molecular mass (M) and degree of oxyethylation (α)], it is necessary to have data on σ_m (minimum surface tension at Γ_{im}). On the basis of experimental data on σ_m , by the least squares method there were obtained the correlation equations (1)–(5) of the type $\sigma_m = f(\alpha)$ for each oligomer homologies of the BCOEPG separately; at $M = \text{const}$ (Table 1). In Figures 1 and 2 show the linear relationships $\sigma_m = f(\alpha)$ и $\sigma_m = f(M)$. It should be noted that the results are obtained within the molecular weight of 2500–6000, and the degree of hydroxyethylation is 12–50%. On the basis of equations (1)–(5), was obtain a more generalized equation with two variables (α и M) for calculating σ_m :

$$\sigma_m = 40.8 - 0.0034M + (0.203 + 0.285 \cdot 10^{-4}M)\alpha. \quad (7)$$

As follows from the data in Table 1, the deviations of the calculated values on σ_m relatively respect to experimental data do not exceed $\pm 0.8\%$. Similar expressions were obtained for n -aliphatic alcohols [9]. Knowing the values of σ_m , one can calculate $\Delta\sigma$ ($-\Delta\sigma = \sigma_0 - \sigma_m$) according to (5), and then using the equation (6) it is possible

Fig. 1. Dependencies of the $\sigma_m = f(\alpha)$ for liquid-crystal nanodemulsifiers with different molecular masses, M : 1 – 2500, 2 – 3500, 3 – 4000, 4 – 5000, 5 – 6000.

Fig. 2. Dependencies of the type $\sigma_m = f(M)$ for liquid crystal nanodemulsifiers at different α , %: 1 – 10, 2 – 12, 3 – 15; 4 – 20; 5 – 30; 6 – 40; 7 – 50.

Fig. 3. The total isotherm of adsorption of liquid crystal nanodemulsifiers, calculated from the Frumkin equation: $\Gamma_i = \Gamma_m [1 - \exp(\Delta\sigma/RT\Gamma_m)]$.

to determine the maximum experimental values of adsorption (Γ_{im}) for each liquid-crystal nanodemulsifiers (LCND) type BCOEPG, that correspond to a saturated monomolecular adsorption layer at the interface liquid-gas. The results are shown in Table 2. In conclusion, using equation (6), it is possible to calculate and construct a common isotherm for the investigated LCND (Figure 3). The liquid crystal state of the nanodemulsifiers studied is shown in Figures 4 and 5. On the offered Tables and Figures can be made the presented below comments, interpretations and conclusions:

- As follows from Table 1, the σ_m values for the investigated liquid-crystal nanoemulsifiers are in the range of 23.9–46.0 mJ/m². The lower limit of the interval determines the hydrophobicity, and the upper limit – the hydrophilicity of the LCND molecules of the BCOEPBG type. If in the equation (7) to put $\alpha=0$ and $M=6000$, to $\sigma_m=20.4$ mJ/m². This is a high level of hydrophobicity for silicone liquids, for which the surface tension is 20–21 mJ/m² [9].

Fig. 4. External visibility of some "IKHLAS" LCND samples from bottles in vertical and horizontal positions: 1 – "IKHLAS" LCND-6003-20, 2 – "IKHLAS" LCND-5003-15, 3 – "IKHLAS" LCND-4003-10.

Fig. 5. Pronounced crystalline states for a liquid crystal nano-demulsifier of the type LCND formed on the walls of a glass beaker: (1) – "IKHLAS" LCND-6003-20, (2) – "IKHLAS" LCND-5003-15, (3) – "IKHLAS" LCND-4003-12, (4) – "IKHLAS" LCND-3500-10.

Indeed, the polypropylene glycol chain with $M \geq 6000$ exhibits maximum hydrophobicity. For the LCND type BCOEPG investigated, the maximum surface tension (46.0 mJ/m^2) is between the surface tensions of polyethylene glycol (43.5 mJ/m^2 [10]) and of ethylene glycol (46.7 mJ/m^2 [11]). This also quite is logical at $\alpha=50\%$ and $M=2500$, in the molecule of the BCOEPG, the terminal polyethylene glycol chain by value of σ_m is approaching to the surface tension of polyethylene glycol and ethylene glycol. On the other hand, at $\alpha=0$ and $M=2500$, formulas (1) and (7) yield $\sigma_m=32.2 \text{ mJ/m}^2$. In this case, a polypropylene glycol chain with a molecular weight of about 2500 contributes its contribution to σ_m . It is known that for propylene glycol the surface tension is 36 mJ/m^2 [12], and for polypropylene glycol with a molecular weight of 3000 the surface tension is in the range of $30.9\text{--}31.7 \text{ mJ/m}^2$ [13] those the value of σ_m in the case $\alpha=0$ and $M=2500$, is between the surface tension of polypropylene glycol and propylene glycol. Thus, there is a definite genetic link between the LCND type BCOEPG molecule and its constituent components (polyethylene glycol, ethylene glycol and polypropylene glycol, propylene glycol). The similar interrelationships for oxyethylated *n*-aliphatic alcohols were discovered by the authors of [4].

- In contrast to oxyethylated ethers of *n*-aliphatic alcohols [8], for the investigated LCND's on the basis of BCOEPG ($M=2500\text{--}6000$, $\alpha=10\text{--}50\%$) obtained rectilinear dependences of the type $\sigma_m=f(\alpha)$ и $\sigma_m=f(M)$ (Figures 1 and 2). Since for $\sigma_m=f(\alpha)$ the maximum value of σ_m is limited to 46.7 mJ/m^2 , for subsequent oligomer homologues at $M=2500$ and $\alpha>50\%$, the minimum surface tension should not exceed 46.7 mJ/m^2 . This was confirmed by experimental data ($\sigma_m=46.4 \text{ mJ/m}^2$) for oligomer-homologue with $M=2500$ and $\alpha=80\%$. An analogous picture is obtained for correlation expressions of the type $\sigma_m=f(M)$ in the case of LCND based on an oligomer-homologue

with $M=7000$ and $\alpha=10\%$. In this case $\sigma_m=23.2 \text{ mJ/m}^2$. Consequently, the expression (5) is satisfied only for was studied homologous series of the BCOEPG.

- As follows from the data in Table 2, for the majority of studied oligomer-homologues, the maximum experimental adsorption (Γ_{im}), calculated from the modified Frumkin equation (4), reaches to value of the maximum theoretical adsorption ($\Gamma_{m(th)}=2.130 \cdot 10^{-6} \text{ mol/m}^2$). Using equation (6) general adsorption isotherm has been constructed in the coordinates $\Gamma_i-(-\Delta\sigma)$ (Figure 3) for the investigated LCND's on the based BCOEPG. On this common isotherm there are 35 isotherms of the investigated LCND's. The general isotherm consists of two parts: 1) $\Gamma_i=2.117\text{--}2.130 \cdot 10^{-6} \text{ mol/m}^2$ the interval corresponds to the values of Γ_{im} one for each LCND; 2) $2.117 \cdot 10^{-6} \text{ mol/m}^2 > \Gamma_i > 0 \cdot 10^{-6} \text{ mol/m}^2$. In this interval, Γ_i assumes the current values. It should be noted that for the selection of the most highly efficient LCND, the condition $\Gamma_{im} \rightarrow \Gamma_{m(th)}$, is necessary, but not sufficient one. This condition practically is satisfied for many of the investigated LCND. Therefore, there arise some difficulties at the adsorption justifications for selecting the high-performance composition of the BCOEPG as an LCND. In our opinion, as a colloid-chemical criterion for evaluating the effectiveness of demulsifiers, one more requirement for demulsifiers should be introduced: surface pressure (π) at Γ_m should be not less than 42 mJ/m^2 , i.e. $\pi \geq 42 \text{ mJ/m}^2$. It is known that $\pi = -\Delta\sigma$. As follows from Table 2, 9 oligomeric homologues correspond to this condition, 6 LCND of them are the leaders in terms of π ($\approx 45\text{--}49 \text{ mJ/m}^2$). Demulsifiers at $\pi \geq 42 \text{ mJ/m}^2$ are able to easily destroy the adsorption protective sheath (APSh) around water globules in water-oil emulsions and also the APSh around oil globules in oil-water emulsions, as a result of which there are achieved standard levels of purification oil from water (residual water content is not more than 0.5%) and water purification from oil (not more than

50 mg/dm³). These demulsifiers can be called also dispersants with respect to the APSH. Thus, for the first time we have proposed a method for estimating the efficiency of demulsifiers by determining their surface pressure, in order to verify the satisfaction of the condition $\pi \geq 42 \text{ mJ/m}^2$.

- Demulsifiers for the destruction of water–oil and oil–water emulsions (under the conditional names "IKHLAS-1"÷"IKHLAS-42" on the basis of the above oligomer homologues), developed by us were defended by a patent [14]. Currently, "IKHLAS-1" and "IKHLAS-2" as LCND are being successfully introduced in five oilfields of the Republic of Kazakhstan, where before were used by the demulsifiers of the USA, Russia and Germany.
- Figure 4 shows three bottles with samples of the liquid-crystal (LC) state of nanodemulsifiers of the "IKHLAS" type: 1 – "IKHLAS" LCND-6003-20; 2 – "IKHLAS" LCND-5003-15; 3 – "IKHLAS" LCND-4003-10. The locations of the vials in the vertical and horizontal positions demonstrate the liquid states of the substance. By the visual observations revealed for the first time crystalline states for the liquids under study at the interface with the surface of the chemical dishes (Figure 5). There is an adsorption accumulation of LC crystals from the volume of the system. In the LC volume, the crystalline state is observed with the help of various methods and instruments, including microscopes. Some authors have established nanodimension LC [15, 16]. Consequently, nanocrystalline nuclei in the LC volume have a high surface activity, due to which corresponding adsorption processes occur. This is a specific aggregate state of matter, in which it manifests the properties of a crystal and a liquid at the same time. Each of the compounds of liquid crystals behaved like a liquid in its mechanical properties and as a crystalline solid body - according to the optical properties of [15, 16]. Of course, not all substances can be in the liquid crystal state. Most substances can be found only in three, all well-known aggregate states. Some

organic substances possessing complex molecules, in addition to the three named states, can form the fourth aggregate state – liquid crystal. Depending on the type of ordering of the molecular axes, liquid crystals are divided into three varieties: nematic, smectic and cholesteric. Another term for the name of liquid crystals (LC) is also used. This is the "mesophase", which literally means "intermediate phase". By external states, the above liquid crystals belong to the smectic form of the LC. This state occurs when the crystals of certain substances are melted. When melting, a liquid crystal phase is formed, which differs from ordinary liquids. This phase exists in the range from the melting point of the crystal to a somewhat higher temperature, when heated to which the liquid crystal passes into the ordinary liquid. What differs the liquid crystal from a liquid and a usual crystal and what makes it similar to them? Like a conventional liquid, liquid crystals also have fluidity and take the shape of the vessel into which it they placed. This is different from the crystals known to all. However, in spite of this property, which unites it with liquidity, it possesses the property characteristic of crystals. This is the ordering in the space of the molecules that form the crystal. True, this ordering is not as complete as in ordinary crystals, but nevertheless it significantly affects the properties of liquid crystals, which distinguishes them from ordinary liquids. Incomplete spatial ordering of molecules forming a liquid crystal is manifested in the fact that in liquid crystals there is no complete order in the spatial distribution of the centers of gravity of molecules, although a partial order can be. This means that they do not have a rigid crystal lattice. However, our studies show that after draining the LC from the glass, with time, real crystals appear on the surface of the used chemical containers like a snowball [Figure 5 (1), (2), (4)]. The crystals found are readily in polar solvents (in water, low-molecular alcohols, etc.) and immediately disappear from the walls of the glass dishes, and in the medium of the nonpolar

organic solvent the visibility of the crystals remains [Figure 5 (3)]. Consequently, the phenomenon associated with the transition from mesocrystals to ordinary traditional crystals was first observed. If we take into account the fact that at a certain temperature there is a transition from liquid crystals to a liquid state, we must also have the possibility of a transition from liquid crystals to a crystalline state, as evidenced by the results of the presented studies.

Conclusion

As a colloid-chemical criterion for estimating the effectiveness of demulsifiers, it was first proposed to use the surface pressure (π) at Γ_m with the following condition: $\pi \geq 42 \text{ mJ/m}^2$, where $\pi = -\Delta\sigma$. As follows from Table 2, to this condition corresponds to 9 oligomergomologs, 6 LCND from them are the leaders in terms of π values ($\approx 45\text{--}49 \text{ mJ/m}^2$). Demulsifiers with $\pi \geq 42 \text{ mJ/m}^2$ can easily destroy the armouring shell around water globules in water-oil emulsions, as well as the armor shell around the oil globules in the oil-water emulsions, resulting in the achievement of the standard levels of oil cleaning from water (residual water content no more than 0.5%) and cleaning water from oil (residual oil content no more than 50 mg/dm^3). Demulsifiers for the destruction of water-oil and oil-water emulsions (under the conventional names "IKHLAS-1"÷"IKHLAS-42" on the basis of the above oligomerhomologs) were defended by a patent [13]. Currently, "IKHLAS-1" and "IKHLAS-2" as an LCND, are being successfully implemented at five oilfields of the Republic of Kazakhstan, where before the demulsifiers of the USA, Russia and Germany used to work.

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OKSİALKİLEN BLOKSOPOLİMERLƏRİ ƏSASINDA OLAN MAYE-KRİSTAL NANODEEMULQATORLARI ÜÇÜN ADSORBSİYANIN HESABLANMA NƏTİCƏLƏRİ HAQQINDA

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Poli(etilenoksid)–poli(propilenoksid) əsasında olan maye-kristal nanodeemulqatorların adsorbsiyasının hesablanması üçün Frumkin tənliyindən istifadə edilmişdir: $\Gamma_i = \Gamma_m [1 - \exp(\Delta\sigma_i / RT\Gamma_m)]$. Bloksopolimerlərin maksimal adsorbsiya qiymətlərinə (Γ_m) uyğun olan minimal səthi gərilmə qiymətlərinin müvafiq oliqomerlərin molekul kütləsindən və oksietilləşmə dərəcəsindən asılı olaraq hesablanması üçün korrelyasiya tənlikləri təklif edilməklə yanaşı $\Gamma_i - (-\Delta\sigma)$ koordinatlarında adsorbsiyanın ümumi izoterması qurulmuş və tədqiqat nəticələri üzrə bəzi şərhlər, interpretasiyalar və nəticələr verilmişdir.

Açar sözlər: Frumkin tənliyi, maye-qaz səth bölgüsündə adsorbsiya, maye-kristal nanodeemulqatorlar, səthi-fəal maddələr.

О РЕЗУЛЬТАТАХ РАСЧЕТА АДСОРБЦИИ ДЛЯ ЖИДКОКРИСТАЛЛИЧЕСКИХ НАНОДЕЭМУЛЬГАТОРОВ НА ОСНОВЕ ОКСИАЛКИЛЕНОВЫХ БЛОКСОПОЛИМЕРОВ

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Для расчета адсорбции (Γ_i) жидкокристаллических нанодэмульгаторов на основе блоксополимеров поли(этиленоксида)–поли(пропиленоксида) на границе раздела жидкость–газ нами использовано уравнение Фрумкина: $\Gamma_i = \Gamma_m [1 - \exp(\Delta\sigma_i / RT\Gamma_m)]$. Предложены корреляционные уравнения для расчета минимального поверхностного натяжения блоксополимеров при различных значениях Γ_m в зависимости от молекулярной массы и степени оксизетилирования соответствующих олигомеров, построена общая изотерма адсорбции в координатах $\Gamma_i - (-\Delta\sigma)$ и приведены некоторые комментарии, интерпретации и выводы по результатам исследований.

Ключевые слова: уравнение Фрумкина, адсорбция на границе раздела жидкость–газ, жидкокристаллические нанодэмульгаторы, поверхностно-активные вещества.