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Surface Pressure Is One of the Main Criteria for Evaluating the Effectiveness of Nano Demulsifiers for Destruction of Reverse and Direct Emulsions

Nugmanov A.K¹., Dashdiyeva T.K².

Director of company «International Oil Services Kazakhstan», Aktau, Kazakhstan¹ Doctoral student of the Azerbaijan State University of Oil and Industry, Baku, Azerbaijan²

ABSTRACT. In the work by the authors for the first time found, that surface pressure is one of the main criteria for evaluating the effectiveness of nano demulsifiers for destruction of reverse and direct emulsions. In the calculations, were used the classical adsorption equations.

KEYWORDS: nanodeemulsifier "IKHLAS"-1, oilfield nanotechnology, the criteria for the evaluation of demulsifiers, surface pressure.

I. INTRODUCTION

Recently, reagents with nano liquid crystal states based on surfactants have also been applied in oil production to increase oil recovery from reservoirs [1]. The authors of [2] for the first time were developed a liquid-crystal nanodemulsifier for primary preparation of oil (PPO), i.e. for the destruction of water-oil emulsions such as water in oil (W/O). It is known that during oil production are formed the water-oil [reverse (W/O)] and oil-water [direct (O/W)] emulsions. 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 in PPO for each oil deposit. In many cases, poly(ethylene oxide)-poly(propylene oxide) block copolymers (PEOPOBS) based on dihydric and polyhydric alcohols are used as the active phase of demulsifiers. Therefore, in almost all oilfields, PPO is carried out using by the thermochemical method.

II. LITERATURE SURVEY

On the basis of the accumulated world experience in the area of PPO, some requirements for demulsifiers are formulated [3]:

- Demulsifiers should be effective, i.e. should provide high quality of the prepared oil with a minimum specific consumption, the minimum time of sludge, at the minimum temperature.
- The demulsifier must have a large surface activity from the phase in which it is introduced.
- The reagent must be well dissolved in one of the phases, i.e. be water or oil soluble. Although this division is purely arbitrary, for any reagent contains both hydrophilic and hydrophobic groups in strictly balanced proportions. It would be more correct to say that the reagent should be well dispersed in the dispersion medium.



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- Demulsifier molecules should have good peptizing properties in order to adsorb on armor shells to cause their loosening.
- The demulsifier molecules should have a high wetting effect on the elements of armor, without which it is impossible to transfer solid particles into the dispersion medium.
- Demulsifier reagents should be low viscosity liquids that don't exfoliate during prolonged storage and don't freeze at low temperatures.
- Demulsifier molecules should not form strong films, i.e. should not be stabilizers of the emulsion of the opposite type, and should also be cheap, transportable, universal, should not affect the commodity properties of oil and significantly change their properties with changing external conditions.
- Reagents must ensure high quality of the separated water, allowing it to be used in the RPM system without additional preparation.
- Reagents must ensure high quality of the separated water, allowing it to be used in the maintain reservoir pressure system without additional preparation.
- The demulsifiers should not cause corrosion of pipes and equipment and reduce the effectiveness of the action of all other reagents.
- Demulsifiers should not coagulate in formation waters.
- Demulsifiers should have certain antifoam properties.
- It is desirable that the demulsifiers are oil soluble and non-ionic substances.
- It is desirable that the demulsifiers help prevent the deposition of salts and mechanical impurities in process equipments and communications. The demulsifiers should facilitate the concentration of mechanical impurities at the interface oil water.

It should be noted that the adsorption characteristics as the main colloid-chemical criteria for assessing the effectiveness of demulsifiers were not reflected in the lists of the above requirements for 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 PEOPOBS. 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 and surface pressure (π), 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 surface tension of the surface tension (4,5): $\sigma_i = \sigma_o - RT\Gamma_m \ln[(1+(C_i\delta/\Gamma_m) \cdot exp(W/RT)]$ (1)

where - C_i is the current concentration; Γ_m is the maximum adsorption; W is the adsorption work, $W = \beta \cdot 10^3 \cdot m$ (2), where β is the Traube number β =4.22 [5], 10^3 is the transition coefficient from kJ to J; m is the number of carbon atoms in alkyl radicals of nonionic surfactants; σ_o is the surface tension of water at 298 K (72.75 mJ/m²); d 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, studies of the adsorption properties of liquid crystal nanodemulsifiers based on oxyalkylene 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 for the oil emulsions.



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III. EXPERIMENTAL AND THEORETICAL PART

Surface tension was determined with the help of a stalagmometer according to the project of the URI (Ufa Research Institute) [5]. To calculate the adsorption isotherms of the surface tension of the surfactant, the most convenient expression was used, by transforming the Frumkin equation :

 $\sigma_{i} = \sigma_{o} + RT\Gamma_{m}ln(1 - \Gamma_{i}/\Gamma_{m})$ (3) The equation (3) solve with respect to Γ_{i} : $ln(1 - \Gamma_{i}/\Gamma_{m}) = \Delta\sigma/RT\Gamma_{m}$ $1 - \Gamma_{i}/\Gamma_{m} = exp(\Delta\sigma/RT\Gamma_{m})$ $\Gamma_{i} = \Gamma_{m} [1 - exp(\Delta\sigma/RT\Gamma_{m})]$ (4) where $\Delta\sigma = \sigma_{i} - \sigma_{o} < 0.$ (5)

Block copolymers ethylene and propylene oxides based on glycerin having a liquid crystal state were investigated as nonionic surfactants. The minimal theoretical area (ω_{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 [4-6]. For block copolymers of oxides of ethylene and propylene based on glycerol (BSOEPBG) $\omega_{min}=78\cdot10^{-20} \text{ M}^2$ [7], then the corresponding value of the maximum theoretical adsorption ($\Gamma_{m(th)}$) is determined by the generally accepted formula [8]:

 $\Gamma_{m(th)}=1/(\omega_{min}\cdot N_A)=2.130\cdot 10^{-6} \text{mol/m}^2$, where N_A is the Avogadro number. Isotherms are often calculated at T=298K. Therefore, equations (4) for the calculation of the adsorption of BSOEPBG can be presented in a more simplified form: $\Gamma_i = 2.130\cdot 10^{-6}[1-\exp(\Delta\sigma \cdot 0.189)]$ (6)

IV. RESULTS AND THEIR DISCUSSION

The experimental maximum values of the adsorption value (Γ_{im}) for the BSOEPBG 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 (ie, there is no surfactant in the system), Γ_i =0, and in the critical state of the BSOEPBG, when there are no interfaces between the liquid and the gas [9] σ_i =0, $\Delta\sigma$ =0-72.75= -72.75 mJ/m², then $\Gamma_i\approx2.130\cdot10^{-6}$ mol/m². The solution of equation (6) at σ_i =0 has only theoretical value, it will be more correct to comment on the corresponding extreme condition as follows: at σ_i =0, $\Gamma_i \rightarrow \Gamma_m$ =2.130·10⁻⁶mol/m². Consequently, Γ_i for BSOEPBG 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 calculate from equation (6) the maximum experimental values of adsorption (Γ_{im}) for each BSOEPBG [depending on the molecular weight (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 least squares method was obtained the correlation equations (7) \div (11) of the type σ_m = f(α) for each oligomergomologist of the BSOEPBG separately; at M = const (Table 1). In Figures 1 and 2 show the linear relationships σ_m = f(α) $\mu \sigma_m$ = f(M). It should be noted that the results are obtained within the molecular weight of 2500-6000, and the degree of hydroxyethylation is 10-50%. On the basis of equations (7) \div (11), was obtain a more generalized equation with two variables ($\alpha \neq M$) for calculating σ_m :

 $\sigma_{\rm m} = 40.8 - 0.0034 \cdot M + (0.203 + 0.285 \cdot 10^{-4} \cdot M) \cdot \alpha$

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 [10]. Knowing the values of σ_m , one can calculate $\Delta\sigma$ ($-\Delta\sigma=\sigma_o-\sigma_m=\pi$) according to (5), and then using the equation (6) it is possible to determine the maximum experimental values of adsorption (Γ_{im}) for each liquid-crystal nanodemulsifiers (LCND) type BSOEPBG, that correspond to a saturated monomolecular adsorption layer at the interface liquid-gas. The results are shown in

(12)



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Table 2. In conclusion, using equation (6), it is possible to calculate and construct a common isotherm for the investigated LCND (Fig. 3). The liquid crystal state of the nanodemulsifiers studied is shown in Figs. 4 and 5. On the presented tables and figures can be made the following comments, interpretations and conclusions:

- As follows from Table 1, the σ_m values for the investigated liquid-crystal nanodemulsifiers are in the range of 23.9÷46.0 mJ/m². The lower limit of the interval determines the hydrophobicity, and the upper limit determines the hydrophilicity of the LCND molecules of the BSOEPBG type. If in the equation (12) to put $\alpha=0$ and M=6000, then $\sigma_m=20.4$ mJ/m², this is a high level of hydrophobicity for silicone liquids, for which the surface tension is 20-21 mJ/m² [10]. Indeed, the polypropylene glycol chain with M≥6000 exhibits maximum hydrophobicity. For the LCND type BSOEPBG investigated, the maximum surface tension (46.0 mJ/m^2) is between the surface tensions of polyethylene glycol (43.5 mJ/m² [11]) and of ethylene glycol (46.7 mJ/m² [12]). This also quite is logical at α =50% and M=2500, in the molecule of the BSOEPBG, 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 (7) and (12) 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/m² [13], and for polypropylene glycol with a molecular weight of 3000 the surface tension is in the range of 30.9-31.7 mJ/m² [14] those the value of $\sigma_{\rm m}$ 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 BSOEPBG 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 [5].
- In contrast to oxyethylated ethers of n-aliphatic alcohols [9], for the investigated LCND's based on the BSOEPBG (M=2500-6000, α=10-50%) obtained rectilinear dependences of the type σ_m= f(α) µ σ_m= f(M) (Fig. 1 and Fig. 2). Since for σ_m=f(α) the maximum value of σ_m is limited to 46.7 mJ/m², for subsequent oligomer homologues at M=2500; α> 50%, the minimum surface tension should not exceed 46.7 mJ/m². This was confirmed by experimental data (σ_m=46.4 mJ/m²) for oligomer-homologue M=2500; α=80%. An analogous picture is obtained for correlation expressions of the type σ_m=f(M) in the case of LCND based on an oligomer-gomologue with M=7000; α=10%, in this case σ_m=23.2 mJ/m². Consequently, the expression (11) is satisfied only for was studed homologous series of the BSOEPBG.
- As follows from the data in Table 2, for the majority studied of oligomer-homologues, the maximum . experimental adsorption (Γ_{im}), calculated from the modified Frumkin equation (4), reaches to value of the $(\Gamma_{m(th)}=2.130\cdot10^{-6} \text{mol/m}^2)$. Using equation (6) has been constructed maximum theoretical adsorption general adsorption isotherm in the coordinates $\Gamma_i - \Delta \sigma$ (Fig. 3) for the investigated LCND's on the based BSOEPBG. On this common isotherm there are 35 isotherms of the investigated LCND's. The general isotherm consists of two parts: 1) $\Gamma_i=2.117 \div 2.130 \cdot 10^{-6} \text{mol/m}^2$ the interval corresponds to the values of Γ_{im} for each LCND; 2) 2.117 \cdot 10⁻⁶ mol/m²> Γ_i >0 \cdot 10⁻⁶mol/m². 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 an necessary, but not an sufficient. This condition practically is satisfied for many of the investigated LCND. Therefore, there are arises some difficulties at the adsorption justifications for selecting the high-performance composition of the BSOEPBG 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 at the interface of liquid - gas (π) at Γ_m should be not less than 40 mJ/m², i.e. $\pi \ge 42$ mJ/m². It is known that $\pi = -$



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 $\Delta\sigma$. As follows from Table 2, 9 oligomeric homologues correspond to this condition, 6 LCND of them are the leaders in terms of π (\approx 45-49 mJ/m²). Demulsifiers at $\pi \ge 42$ mJ/m² 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 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 us is proposed a method for estimating the efficiency of demulsifiers by determining their surface pressure at the interface of liquid - gas, in order to verify the satisfaction of the condition $\pi \ge 40$ mJ/m².

- 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 [15]. 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.
- In 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 [16, 17]. 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 [16, 17]. 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. Than the liquid crystal differs from a liquid and a usual crystal and than it is similar to them? Like a conventional liquid, liquid crystals also have fluidity and take the shape of the vessel into which it is 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.)



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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.

V. CONCLUSION

As a colloid-chemical criterion for estimating the effectiveness of demulsifiers, it was first proposed to use the surface pressure at the interface of liquid - gas (π) at Γ_m with the following condition: $\pi \ge 40 \text{ mJ/m}^2$, where $\pi = -\Delta \sigma$. As follows from Table 2, to this condition corresponds to 9 oligomergomologists, 6 LCND from them are the leaders in terms of π values (≈ 45 -49 mJ/m²). Demulsifiers with $\pi \ge 42 \text{ mJ/m}^2$ 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/dm³). 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 oligomergomologys) were defended by a patent [15]. 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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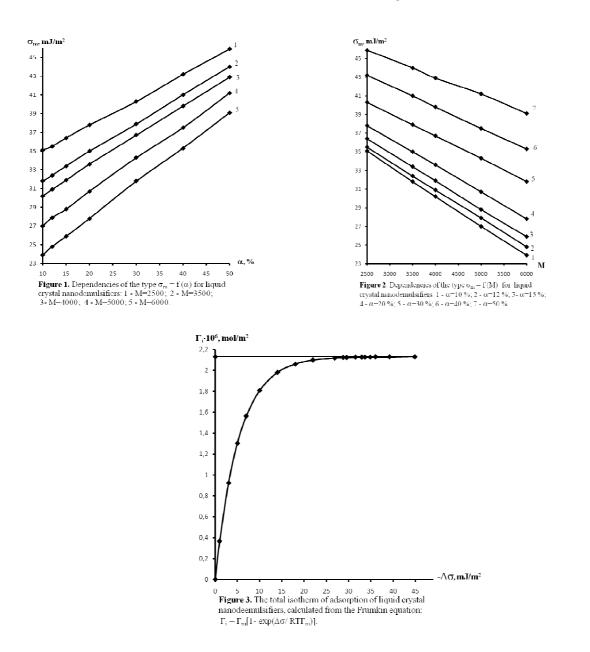
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Table 1: Comparison of the experimental and theoretical values of σ_m , calculated with the help of (7)-(12) at Γ_{im} for the LCND of BSOEPBG type with different values of M and α

М	$\sigma_m = f(\alpha)$	$\sigma_{\rm m},{ m mJ/m^2}$																				
		α=10%			α=12%			α=15%			α=20%			α=30%			α=40%			α=50%		
		exp	theo	erro	exper	theo	erro	exper	theo	erro	exp	theo	erro									
		eri	reti	r,	iment	reti	r,	iment	reti	r,	eri	reti	r,									
		men	cal	%	al	cal	%	al	cal	%	men	cal	%									
		tal									tal			tal			tal			tal		
2500	$\sigma_m = 32.2 + 0.274 \cdot \alpha$ (7)	35.1	35.0	-0.3	35.5	35.6	+0.	36.4	36.4	0	37.8	37.8	0	40.3	40.5	+0.	43.2	43.3	+0.	45.9	46.0	+0.
							3									5			2			2
3500	$\sigma_m = 28.9 + 0.302 \cdot \alpha$ (8)	31.8	31.9	+0.	32.4	32.5	+0.	33.4	33.4	0	35.0	34.9	-0,3	37.9	38.0	+0.	41.0	41.0	0	44.0	44.0	0
				3			3									3						
4000	$\sigma_m = 27.0 + 0.318 \cdot \alpha$ (9)	30.2	30.4	+0.	30.9	31.0	+0.	31.9	32.0	+0.	33.6	33.5	-0.3	36.7	36.7	0	39.8	39.9	+0.	42.9	43.0	+0.
				7			3			3									2			2
5000	$\sigma_m = 23.5 + 0.354 \cdot \alpha$	27.0	27.2	+0.	27.9	27.9	0	28.8	29.0	+0.	30.7	30.7	0	34.3	34.2	-0.3	37.5	37.6	+0.	41.2	41.1	-0.2
	(10)			7						7									3			
6000	$\sigma_m = 20.4 + 0.374 \cdot \alpha$	23.9	24.1	+0.	24.8	24.9	+0.	25.9	26.0	+0.	27.8	27.9	+0.	31.8	31.6	+0.	35.3	35.4	+0.	39.1	39.1	0
	(11)			8			4			4			3			6			3			

Table 2: The values of σ_m and - $\Delta\sigma$ at Γ_{im} for each liquid crystal nanodemulsifier

α, %	$\sigma_{\rm m}$, mJ/m ² ; - $\Delta\sigma$ (or π), mJ/m ² ; $\Gamma_{\rm im}$ ·10 ⁶ , mol/m ²															
	M=2500			M=3500				M=4000			M=5000		M=6000			
	$\sigma_{\rm m}$	- $\Delta \sigma$, or π	$\Gamma_{\rm im}$	σ_{m}	- $\Delta \sigma$, or π	$\Gamma_{\rm im}$	$\sigma_{\rm m}$	- $\Delta \sigma$, or π	$\Gamma_{\rm im}$	$\sigma_{\rm m}$	- $\Delta \sigma$, or π	$\Gamma_{\rm im}$	σ_{m}	- $\Delta \sigma$, or π	$\Gamma_{\rm im}$	
10	35.1	37.65	2.128	31.8	40.95	2.129	30.2	42.55	2.129	27.0	45.75	2.130	23.9	48.85	2.130	
12	35.5	37.25	2.128	32.4	40.35	2.129	30.9	41.85	2.129	27.9	44.85	2.130	24.8	47.95	2.130	
15	36.4	36.35	2.128	33.4	39.35	2.129	31.9	40.85	2.129	28.8	43.95	2.129	25.9	46.85	2.130	
20	37.8	34.95	2.127	35.0	37.75	2.128	33.6	39.15	2.129	30.7	42.05	2.129	27.8	44.95	2.130	
30	40.3	32.45	2.125	37.9	34.85	2.127	36.7	36.05	2.128	34.3	38.45	2.128	31.8	40.95	2.129	
40	43.2	29.55	2.122	41.0	31.75	2.125	39.8	32.95	2.125	37.5	35.25	2.127	35.3	37.45	2.128	
50	45.9	26.85	2.117	44.0	28.75	2.121	42.9	29.85	2.122	41.2	31.55	2.124	39.1	33.65	2.126	

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Figure 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.

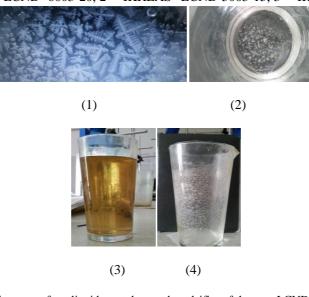


Figure 5. Pronounced crystalline states for a liquid crystal nano-demulsifier of the type LCND formed on the walls of a glass beaker: (1) "IKHLAS" LCND- 6003-20; (2) "IKHLAS" LCND- 5003-15; (3) "IKHLAS" LCND- 4003-12; (4) "IKHLAS" LCND- 3500-10.