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#### **Abstract:**

The article discusses the features of the synergistic effect of the compositions of nanodemulsifiers in the purification of oil from water and water from oil. Synergy is caused by the composition of non-ionic surfactants of ethoxylated esters of n-aliphatic acids and the block of copolymers of oxyalkylene esters with polyhydric alcohols. Theoretical predictions for synergistic compositions were confirmed by laboratory results. **Key Words:** Synergy, Nanodemulsifiers & Non-Inogenic Surfactants

#### **Introduction:**

The unsatisfactory quality of the oil preparation technology of some fields (including the "Zhetybaimunaygaz" JSC "MMG") is accompanied by the emergence of emulsion systems of oil of secondary origin - these are intermediate emulsion layers, trap oil, barn oil, oil sludge and other production wastes. The above abnormal oils, as dispersed systems containing a high amount of mechanical impurities and being highly water-concentrated emulsions, are not only incompatible with each other, but are not comparable in their basic physicochemical parameters and rheological parameters with traditional oils and their emulsions. Under oil production conditions, especially in the later stages of field exploitation, in the wells are formed reverse wateroil emulsions (W/O), direct oil-water emulsions (O/W) and multiple (medium) emulsions (W/O/W) [1]. Multiple emulsions in oil field practice are called intermediate layers or hard destructible water-oil emulsions (HDWOE) [2]. HDWOE in old fields are often mixed with hard destructible water-oil suspensions (HDWOS). In 2014, the "IKHLAS"-1 nanodemulsifier successfully passed the experimental-industrial tests (EIT) at the "Zhetybaimunaygaz" field of "MMG" JSC of RK and for the first time in the conditions of primary oil preparation, were achieved positive results in wastewater treatment from oil at not exceeding 50 mg/l [2] . For the development of this direction, the authors have envisaged plans to create new modifications of the nanodemulsifier "IKHLAS"-1 for the deep purification of oil (including HDWOE) from water and waste water from oil. HDWOE are characterized by a high content of various mechanical impurities with different degrees of dispersion (i.e., they possess polydispersity), which form the basis of the protective adsorption layers (PAL) around the globules of the dispersed phases. PAL consists of a mixture of natural surface-active substances (surfactants such as naphthenic acids; asphaltenes; resins and other polar components of oil) and nano-, microsize mechanical impurities of organic and inorganic origin (clay, ferrous sulfide, calcium carbonate, sand, silt, salts, porphyrins, high melting paraffins, etc.). Protective adsorption layers have increased viscosity and elasticity that prevent the merging of colliding water droplets [3]. Therefore, molecules of additives to the demulsifiers should have a high wetting effect on the elements of the PAL, without which it is not possible to transfer solid particles into the volume of the dispersion medium [4]. As additives, wetting agents for demulsifiers mainly use NS, which have certain advantages compared with other types of surfactants: salt resistance; cold demulsification; the ability to regulate the hydrophilic-lyophobic balance (and thereby wetting ability) by changing the length of the alkyl radical (m), as well as the number of oxyethylene units (n) in the molecule [5, 6]. Consequently, the selection of optimal values of m and n can provide synergies for the compositions of nonionic surfactants + demulsifier in the preparation of emulsion systems of oil of secondary origin. Therefore, conducting targeted research to establish a correlation relationship a type: structure-propertythe use of nonionic surfactants (as wetting agents in the composition of demulsifiers) is one of the priority and topical issues of modern oilfield chemistry [7].

#### **Experimental Part:**

As the research methods, we used the bottle test method, tensiometric and spectrophotocolorimetric methods [8]. The demulsifying ability of the demulsifiers was determined by the method of bottl-tests, and the values residual water content in the oil were installed on the Dean-Stark facility in accordance with GOST 2477-65, as well as using the "Laboratory crude oil hydrometer" instrument of the LCO-L [9]. The concentration of oil in wastewater was determined on a SPECTROPHOTOMETER UV755B (China, Shanghai, YK Scientific). A micrometric stalagmometer was used as a tensiometric method [10]. The wetting angle and

the work of adhesion were determined in accordance with the generally accepted method [11]. The values of the critical concentration of micelle formation were established as break points from concentration isotherms of surface tension [6].

#### **Results and their Discussion:**

As a multiple oil emulsion, was used trap oil (TO, mixture of HDWOE and HDWOS) from the water tank VST-2 at the shop primary preparation of oil (ShPPO) of the "Zhetybaimmunaygas" field of JSC "MMG". Laboratory studies were conducted with the IKHLAS -1 demulsifier (which was developed jointly by specialists "IOS Kz", JSC "Mangistaumunaigas" of the Republic of Kazakhstan and Azerbaijan State University of Oil and Industry) in comparison with the RANDEM base demulsifier-2219 (Nalco USA). As the objects of the study, individual ethoxylated ethers were used, which were isolated and identified from the mixture of oligomers of homologues by the method of preparative column chromatography using critical nanoemulsions [12]. In studies of the wetting angle were determined on the hydrophilic surface of quartz glass and on a glass plate coated with a layer of paraffin. In our work, we used the Young formula, which makes it possible to determine the work of adhesion (W<sub>A</sub>) through of the wetting angle ( $\theta$ ) [6]:

$$
W_A = \sigma_{mLG}(1 + \cos\theta) \tag{1}
$$

Using formula (1), table 1 presents a systematic qualitative assessment of the magnitude of the wetting of liquids and the work of adhesion on a solid hydrophilic surface.



Table 1: Qualitative assessment of the values of wetting of liquids and adhesion work on a hydrophilic solid surface

As follows from table 1, full wetting occurs when  $\cos\theta =1$ . A similar table can be obtained for a hydrophobic surface. Consequently, the maximum synergistic effect of the wetting agent in the composition of the demulsifier when cleaning oil from water and when cleaning waste water from oil can be achieved with the use of nonionic surfactants, for which the condition  $\cos \theta = 1$  on the hydrophilic and hydrophobic surface is satisfied. Table 2 shows the values for some of the colloidal chemical properties for the studied nonionic surfactants as wetting additives to the demulsifier "IKHLAS" -1 (which, by the authors was patented by the Republic of Kazakhstan patent [13]), as well as the results of the bottle tests. At figure 1 are presented the inversely proportional dependences of the minimum surface tension for the investigated nonionic surfactants at a critical micelle concentration (CMC). By the method least squares, obtained the following correlation dependences of the property-structure type of nonionic surfactants (in particular, on the length of the hydrocarbon radical of nonionic surfactants) at obtained for the same length of the oxyethylene chain:



$$
\sigma_{mLG} = 59.9 - 1.53 \cdot m \qquad \text{when } n = 16 = \text{const}
$$
 (3)  
\n
$$
\sigma_{mLG} = 59.9 - 1.53 \cdot m \qquad \text{when } n = 16 = \text{const}
$$



Figure 1: Dependence  $\sigma_{mIG}$  on m for  $C_{10}EO_m$ : 1 - n=8; 2 - n=12; 3 - n=16.

Since the dependence  $\sigma_{mLG} = f(m)$  gives parallel straight lines, the slope tangent (tga) will be a constant value, regardless of the value of n. Indeed, according to equations (2)  $\div$  (4) tga = 1.53 = const. In the table 3 for  $\sigma_{mLG}$  (minimum surface tension with CMC) are presented the experimental and calculated values, as well as the corresponding deviations that are within the experimental error. The maximum deviation is  $\pm$  0.88. Table 3 also shows the data on the values of CMC for the representatives of the investigated homologous series of ethoxylated esters of n-aliphatic acids. In semi-logarithmic coordinates are presents the dependencies  $ln(CMC \cdot 10^6) = f(m)$ , for which the following empirical expressions are established:

 $ln(CMC \cdot 10^6) = 27.1 - 1.451$  $n=8=const$ Table 2: Data on the colloid-chemical properties of NS, and the effectiveness of the compositions of  $MS + "IKHI A S" 1 50$ 

demulsifier	<b>NS</b> (wetting agent)	$(110T$ INILAN $-170T$ The composition of the demulsifier, %			CMC NS.	$\beta$	$\sigma_{mLG}$ NS,	$\theta$ NS, $^{\rm o}$	
		<b>NS</b>	act. phase	solvent	mol/l	traube coefficient	mJ/m <sup>2</sup>	qua gl par	
"Randem"-2219					÷.	$\overline{\phantom{a}}$			
IKHLAS-1-50		$\overline{\phantom{a}}$	50	50				$\overline{\phantom{a}}$	$\overline{\phantom{a}}$
"IKHLAS"-1-50-1	$C_8EO_8$	3	50	47	$47.28 \cdot 10^{-1}$	4.16	43.4	28	44
"IKHLAS"-1-50-2	$C_8EO_{12}$	$\overline{3}$	50	47	$25.22 \cdot 10^{-1}$	4.16	46.3	$\theta$	65
"IKHLAS"-1-50-3	$C_8EO_{16}$	3	50	47	$13.47 \cdot 10^{-1}$	4.15	47.6	$\mathbf{0}$	89
"IKHLAS"-1-50-4	$C_{10}EO_8$	3	50	47	$27.30 \cdot 10^{-2}$	4.15	40.4	47	$\mathbf{0}$
"IKHLAS"-1-50-5	$C_{10}EO_{12}$	$\overline{3}$	50	47	$14.57 \cdot 10^{-2}$	4.15	43.4	$\mathbf{0}$	$\boldsymbol{0}$
"IKHLAS"-1-50-5	$\overline{C}_{10}EO_{12}$	3	50	47	$14.57 \cdot 10^{-2}$	4.15	43.4	$\Omega$	$\mathbf{0}$
"IKHLAS"-1-50-5	$\overline{C}_{10}EO_{12}$	3	50	47	$14.57 \cdot 10^{-2}$	4.15	43.4	$\Omega$	$\mathbf{0}$
"IKHLAS"-1-50-5	$\overline{C_{10}EO}_{12}$	3	50	47	$14.57 \cdot 10^{-2}$	4.15	43.4	$\mathbf{0}$	$\boldsymbol{0}$
"IKHLAS"-1-50-5	$C_{10}EO_{12}$	3	50	47	$14.57 \cdot 10^{-2}$	4.15	43.4	$\mathbf{0}$	$\theta$
"IKHLAS"-1-50-6	C <sub>10</sub> EO <sub>16</sub>	3	50	47	$7.78 \cdot 10^{-2}$	4.16	44.5	$\Omega$	12
"IKHLAS"-1-50-6	$\overline{\mathrm{C}}_{10}\mathrm{EO}_{16}$	3	50	47	$7.78 \cdot 10^{-2}$	4.16	44.5	$\mathbf{0}$	12
"IKHLAS"-1-50-6	$\overline{C}_{10}EO_{16}$	3	50	47	$7.78 \cdot 10^{-2}$	4.16	44.5	$\theta$	12
"IKHLAS"-1-50-6	$C_{10}EO_{16}$	3	50	47	$7.78 \cdot 10^{-2}$	4.16	44.5	$\Omega$	12
"IKHLAS"-1-50-6	$C_{10}EO_{16}$	3	50	47	$7.78 \cdot 10^{-2}$	4.16	44.5	$\mathbf{0}$	12
"IKHLAS"-1-50-7	$C_{12}EO_8$	3	50	47	$15.86 \cdot 10^{-3}$	4.48	37.6	76	$\Omega$
"IKHLAS"-1-50-8	$C_{12}EO_{12}$	$\overline{3}$	50	47	$8.46 \cdot 10^{-3}$	4.10	40.6	42	32
"IKHLAS"-1-50-9	$C_{12}EO_{16}$	3	50	47	$4.52 \cdot 10^{-3}$	4.14	41.5	19	92
"IKHLAS"-1-50-10	$C_{14}EO_8$	3	50	47	$7.89 \cdot 10^{-4}$	4.10	34.0	92	$\Omega$
"IKHLAS"-1-50-11	$C_{14}EO_{12}$	3	50	47	$4.21 \cdot 10^{-4}$	4.10	37.3	71	25
"IKHLAS"-1-50-12	$C_{14}EO_{16}$	3	50	47	$2.25 \cdot 10^{-4}$	4.09	38.4	47	77
"IKHLAS"-1-50-13	$C_{16}EO_8$	3	50	47	$4.70 \cdot 10^{-5}$	4.17	31.2	99	$\Omega$
"IKHLAS"-1-50-14	$\overline{C}_{16}EO_{12}$	$\overline{3}$	50	47	$2.50 \cdot 10^{-5}$	4.17	34.3	80	19
"IKHLAS"-1-50-15	$\overline{C_{16}}\overline{E}O_{16}$	3	50	47	$1.34 \cdot 10^{-5}$	4.17	35.4	68	61
"IKHLAS"-1-50-16	$C_{18}EO_8$	3	50	47	$2.70 \cdot 10^{-6}$	4.22	28.0	114	$\Omega$
"IKHLAS"-1-50-17	$C_{18}EO_{12}$	3	50	47	$1.44 \cdot 10^{-6}$	4.22	31.2	82	13
"IKHI AS"-1-50-18	$C_{\infty}$ FO <sub><math>\infty</math></sub>	$\mathcal{R}$	50	47	$0.77,10^{-6}$	422	323	73	27



 $(5)$ 



Table 3: Experimental and theoretical values of  $\sigma_{mWG}$ , ln(CMC·10<sup>6</sup>) with corresponding deviations



Based on the analytical expressions  $(5) \div (7)$ , a more generalized equation was obtained for characterize the property-structure correlation relationship of nonionic surfactants with two variables (m and n):

 $ln(CMC \cdot 10^6) = 29 - 0.244 \cdot n - 1.498 \cdot m + 0.0059 \cdot m \cdot n$ 

Table 3 shows the experimental and calculated values for  $ln(CMC \cdot 10^6)$ , as well as the corresponding deviations that are within the experimental error. The maximum deviation is  $\pm 1.77$ . The exception is data for representatives of the homologous series of ethoxylated n-octadecylic acid esters  $(C_{18}EO_n)$ , for which the maximum deviations of the calculated data relative to the experimental data are  $\pm$  34.6%. Such high deviations for  $C_1$ <sub>s</sub>EO<sub>n</sub> are probably related to ultra-low values of the expression  $ln(CMC \cdot 10^6)$ . Table 2, 3 and Figure 1, 2 reflect the regularities of the property-structure and property-property for the studied nonionic surfactants, and table 2 additionally reflects the structure-property-application relationship. If pay attention to the relationship  $(m; n)$  - the cosine of the wetting angle (cos  $\theta$ ) - (the effect of cleaning oil from water + the effect of cleaning water from oil), here the variables m; n determine the molecular structure of the nonionic surfactants; cos  $\theta$  is a property of nonionic surfactants, and the expression [the effect of oil purification from water  $(C_{rw})$  + the effect of water purification from oil  $(C_{r_0})$  determines the functional purpose for the use of nonionic surfactants as a wetting agent in the synergistic composition of the nanoemulsifier "IKHLAS"-1. Indeed, as follows from the data in Table 2, the maximum efficiency of  $C_{rw}$  and  $C_{ro}$  values is achieved at  $(cos\theta_{qug} + cos\theta_{par}) = 2$ , and also for  $(cos \theta_{qug} + cos \theta_{par})$   $\rightarrow$  2. Herewith happens the full wetting or excellent wetting (table 1), as well as the complete destruction of the inverse and direct emulsions, resulting in,  $C_{rw} = 0\%$  and  $C_{ro} = 23$  mg/l. The residual concentration of oil in water in the order of 23 mg/l corresponds to the dissolution of hydrocarbons in water at the molecular level, i.e. there is no the direct emulsion, type oil in water. True solutions of hydrocarbons in the composition of the formation waters are not able to create problems during the re-injection of wastewater to maintain reservoir pressure.

The above described optimal synergistic interconnection by property - the use of nonionic surfactants is achieved with the following values of the structural elements of nonionic surfactants  $m = 10$  and  $n = 12 \div 16$ . Thus, the goal in work was achieved. The most optimal compositions of nonionic surfactants  $(C_{10}EO_{12+16})$ were selected as wetting agents for the "IKHLAS"-1nanodemulsifier for destruction HDWOE, accumulated in ShPPO of the Zhetybaimunaygaz JSC of "MMG". With the help of the proposed technology, the first time the maximum effects are achieved during the dewatering of the HDWOE and in the purification of wastewater from oil under conditions of the primary preparation of oil. Thus, the goal in work was achieved. The most optimal synergistic compositions of nonionic surfactants ( $C_{10}EO_{12+16}$ ) were selected as wetting agents for the "IKHLAS"-1 nanodemulsifier for destruction HDWOE, accumulated in ShPPO of the Zhetybaimunaygaz JSC of MMG. With the help of the proposed technology, the first time the maximum synergistic effects are achieved

during the destruction of the HDWOE and in the purification of wastewater from oil under conditions of the primary preparation of oil.



Figure 2: Dependence  $ln(CMC \cdot 10^6)$  on m for  $C_{10}EO_m$ : 1 - n=8; 2 - n=12; 3 - n=16.

As a result of years of world experience in the field oil production, were formulated some basic requirements for demulsifiers in the condition primary preparation of oil: [14]:

- Demulsifiers should be effective, i.e. must ensure high quality of the prepared oil at the minimum specific consumption, minimum sludge time at the minimum temperature;
- The demulsifier must have a large surface activity from the phase in which it is introduced;
- The demulsifier should be well dispersed in the dispersion medium;
- Demulsifier molecules should have good peptizing (grinding) properties in order to adsorb on reserving 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 a dispersion medium;
- The molecules of demulsifiers 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 when change the of externals conditions.
- The demulsifier reagents should be low-viscosity liquids that do not exfoliate during long-term storage and do not solidify at low temperatures;
- The demulsifiers should provide high quality separated water in the conditions of primary oil preparation, allowing it to be used in the reservoir pressure maintenance system without additional preparation;
- The demulsifiers should not cause corrosion of pipes and equipment and reduce the effectiveness of all other reagents;
- The demulsifier should not coagulate in the formation waters;
- The demulsifier must have certain anti-foam properties;
- It is desirable that the demulsifier is oil soluble and non-ionic;
- The demulsifier must have a high speed action;
- Demulsifiers should exhibit thermodynamic and aggregative resistance under various technical, thermal, technological, and climatic conditions for oil preparation;
- With the aim of maximum efficiency of destruction of all types of oil emulsions (reverse emulsions of type W/O; direct emulsions of type O/W; medium emulsions of type W/O/W), taking into account their natural nanostructures, and all types of technological residues (hard to destroy water-oil emulsions (HDWOE), hard to destroy water-oil suspensions (HDWOS), trap oil, granary oil, bottom sediments of process and commercial tanks, trap oil sludge, etc.) primary oil preparation, demulsifiers should have a nanostructure, i.e. must be nanodemulsifiers.
- The surface pressure for nanodemulsifiers should be at least 40 mJ/m<sup>2</sup>;
- Nanodemulsifiers should have liquid-crystalline properties and their liquid-crystalline state can be visually observed;
- The components of the active phase of nanodemulsifiers should show a synergistic effect in the destruction of reverse, direct and medium emulsions;

- Highly effective nanodemulsifiers as a wetting agent should contain a synergistic component from homologous series of low molecular weight nonionic surfactants such as ethoxylated ethers of naliphatic alcohols, acids and other compounds that cause the maximum wetting (or  $cos\theta=0$ , or  $\cos\theta \rightarrow 0$ ) of components of the molecular adsorption layer ("reservation shell");
- The most suitable solvents for nanodemulsifiers are critical nano-emulsions, which with the active phase can produce a synergistic effect;
- The demulsifier with the solvent should not give a visco-elastic systems;
- Molecules of the active phases of nanodemulsifiers should easily overcome of nanostructured barriers in the dispersion medium and in the dispersed phase of oil emulsions;
- Demulsifiers, including nanoemulsifiers should not contain nanopowder components, which can further increase the resistance of oil emulsions;

Side requirements for nanodemulsifiers in the condition primary preparation of oil:

- Nanodemulsifiers may also possess depressant, anticorrosive and bactericidal properties;
- Nanodemulsifiers can also perform the function of a nanodesuspensifier for treating bottom sediments consisting of a mixture of HDWOE and HDWOS;
- Nanodemulsifiers can also perform the function as an inhibitor and solvent of visco-elastic systems in the condition primary preparation of oil;
- Nanodemulsifiers can also perform the function as an inhibitor and solvent of gas hydrates in the condition primary preparation of oil;
- Nanodemulsifiers can also perform function as a neutralizer of hydrogen sulfide and highly dispersed iron sulfide in oil in the condition primary preparation of oil;
- Demulsifiers can also contribute to the prevention of salt deposits and mechanical impurities in the technological equipments;

Requirements number 14-28 formulated by us. Thus, the development and implementation of nanoemulsifiers (as much as possible meet the above requirements) for the treatment of formation water from oil in the conditions of primary oil preparation is one of the priorities directions of oilfield nanotechnology.

# **Conclusion:**

Based on laboratory studies, the most optimal synergistic compositions of nonionic surfactants  $(C_{10}EO_{12-16})$  were selected as wetting agents for the nanodemulsifier "IKHLAS" -1. With the help of the proposed synergistic technology, for the first time, a deep purification of oil (including HDWOE) from water and waste water from oil is achieved under conditions of primary preparation of oil. Therefore, a new synergistic modification of the nanodemulsifier "IKHLAS" -1 is recommended for carrying out an EİT at the field "Zhetybaymunaygaz" of JSC "MMG" RK.

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