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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

DEVELOPMENT AND IMPLEMENTATION OF NANODEMULSIFIERS FOR THE PURIFICATION OF FORMATION WATER FROM OIL IN CONDITIONS PRIMARY PREPARATION OF OIL

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GENERAL CHARACTERISTICS OF THE WORK

The relevance of the topic and degree of elaboration. At each oil field, the produced fluid (oil+water+gas+mechanical impurities), is separated from the gas in the shop of primary preparation and pumping of oil (ShPPPO) then subjected to thermochemical treatment to remove water, salts and mechanical impurities within existing standards. Thereafter commercial oil is transported to refineries, and the produced formation water is utilizated of in reservoir pressure maintenance (RPM) systems[1]. In RPM systems, along with other requirements, the content of oil and oil products in the composition of formation water should not exceed a value of 50 mg/dm^3 [2]. Reverse demulsifiers, which are proposed for the treatment of produced water from oil, have not been confirmed by oil-field technology. In recent years, some new brands of the latest generation demulsifiers have appeared on the world market, including: "DEMTROL" ("Dow" company, USA), "DMO" ("Baker Hughes", USA), "Condem" ("Champion Technologies, Inc", USA), "Randem" (company "Rauan Nalko", USA-Kazakhstan), "Kemelix" ("Croda oil and gas" company, England), F-929, F-940, R-11, X-2647 (companies "Toho Chemical Industry Co., Ltd"; "Iskra Industry" and others, Japan), "Dissolvan" (Hoechst, Basf, Clariant, Almatıneftexim, Germany), demulsifiers of the company "Auby" France, demulsifiers of Shanghai Trustin Chemical Co., Ltd, Qingdao Aurora Chemical Co., Ltd, Shanghai Jianying Chemical Co., Ltd. and others. China, "SNPX" ("NİİNefteprom xim" company, former "Soyuznefteprom xim" company, Russia), "Flek" (Flek company, Russia), liquid-crystalline nano-demulsifier TND (Tyumen State University, Russia), "Khazar" ("IPChP" MSE of Azerbaijan), "Alkan" ("IPChP" MSE of Azerbaijan), "ND"

[1] Mirələmov, H.F. Neftin, qazın boru kəmərləri ilə nəqli. Dərslik./ H.F.Mirələmov, Q.Q.İsmayılov. - Bakı: NQETLİ, - 2010. - 505 s.

[2] ОСТ 39-225-88 (ОСТ 39-133-81). Вода для заводнения нефтяных пластов. Требования к качеству. doi: gostrf.com/normadata/1/4293836/4293836522

(Research and Design Institute Oil and Gas, SOCAR). Among the demulsifiers studied, there are few nanodemulsifier reagents, and there is not enough information on the necessary level of purification of produced water from oil and oil products by the demulsifiers used. On the other hand, there is a need for the development on the basic requirements for demulsifiers and on the mechanisms of destruction and aging of oil emulsions on the level of the oil-field nanotechnology.

Recently, at the modern level of development of oilfield nanoecotechnologies, theories have been formed that are fundamentally different from the classical concepts of crude oil. Thus, the well-known Russian scientist I.N. Evdokimov and others have confirmed with numerous research studies that the dispersion phase in oil emulsions has a natural nanostructure[3]. Therefore, to weaken counteractions of nanostructured barriers in oil emulsions, demulsifiers must also have a nanostructure. It is from this perspective that the development and implementation of nanodemulsifiers for the purification of oil from formation water and formation water from oil in the conditions primary preparation of oil (PPO) is one of the priority direct of the oilfield nanoecotechnology. The dissertation have been fulfilled out on the basis of the scientific and technical cooperation agreement between Azerbaijan State Oil and Industry University (ASOIU) and the "International Oil Services Kazakhstan" LLP. For the first time an attempt was made to study a problem at the level of oilfield nanoecotechnology. In this regard, the results of the analytical analysis of scientific works of many scientists (V.M.Abbasov, A.I. Hasanov, S.M. Abbaszade, E.Sh. Abdullaev, F.S.Ismailov, B.A. Suleymanov, G.I. Kelbaliyev, S.R.Resulov, Q.Q.İsmayılov, D.N.Levchenko, G.N. Pozdnishev, A.A. Abramzon, A.I. Rusanov, I.N. Evdokimov, T.A.Fedushak, S.Axmadi, D.G.Siganov, F.M.Xutoryanskiy, N.Y.Bashkirseva, O.Y.Sladovskaya,

[3] Евдокимов, И.Н. Нанотехнологии управления свойствами природных нефтегазовых флюидов. Учебное пособие. / И.Н. Евдокимов. - М.: МАКС Пресс, - 2010. - 364 с.

V.Garstein, U.Muller, Ch.Deghenhard, D.N.Mingazov, B.T.Mullaev, L.P.Semihina, B.R.Faxrutdinov, R.F. Hamidullin, H.Abdurahman, R.Cato, Y.Fu, C. Gabriel, B.Marie, A.Mosayebi, V.Ramesh, E.H.Diyarov, J. Wu, G. Yin, F.Wang, C. Carsten and others) devoted to the development, research and application of demulsifiers in the conditions of primary preparation of oil, made it possible to determine the relevance, goals and objectives of the work.

The object and subject of the research. The choice of research objects and their functional tasks are directly focused on the purpose of the dissertation topic. The development of nanodemulsifier compositions is based on the synergistic effect of research objects. In this work, non-ionic surfactants were given importance as research objects. Complex obligations for new generation deemulsifiers with synergistic properties: for the first time in PPO conditions, it was determined that polynanostructured "IKHLAS" type reagents are environmentally and technologically highly efficient deemulsifiers, desuspensioners, depressors, dehydrators, inhibitory polyfunctional deemulsifiers for oil emulsions, suspensions and oil colloids. These qualities of demulsifiers has been the focus of attention in the justification of objects. **The subject** of the research is related to the main points that ensure the purification oil from formation water and formation water from oil in the conditions primary preparation of oil: development of requirements that allow the evaluation of demulsifiers; study of structure-property relationships that provide efficient solvent, active phase and wetting additives selection for proposed demulsifiers; laboratory, experimental tests and implementation of nanodemulsifiers of the brand "IKHLAS" with optimal composition for a number of oil fields.

Research purpose and objectives. Development and implementation of nanodemulsifiers for the purification of oil from formation water, and formation water from oil in the conditions of PPO. To achieve this purpose theoretical and experimental research were oriented for solving the following main tasks:

– development of innovative nanodemulsifiers with ecological and technological support, simultaneously breaking both reverse and direct emulsions;

– development of requirements for demulsifiers to determine their effectiveness;

– substantiation of n-aliphatic alcohols of certain composition as optimal demulsifier solvents by studying property-structure relationships;

– study of the necessary colloidal-chemical properties for liquid-crystalline nanodemulsifiers based on oxyalkylene BC;

– bringing to the attention of new colloidal-chemical concepts that can distinguish surfactats-demulsifiers from other surfactats in order to ensure high ecological and technological efficiency of modern generation demulsifiers;

– development of new mechanisms for the breakdown and aging of oil emulsions;

– conducting laboratory and experimental-industrial tests (EIT) for oil purification from formation water, and formation water from oil using nanodemulsifiers, in the conditions of PPO at various oil fields, calculation of economic efficiency based on the results of the EIT and the introduction.

Research methods. Research methods described in chapter II were used in the work of the dissertation: the method of preparative tube chromatography was applied in the separation and identification of oxyethylene ethers of n-aliphatic acids from the technical mixture, as wetting additives; decomposition of water-oil emulsions was carried out by thermo-chemical method; the “bottle-test” method was used to determine the efficiency of demulsifiers; the degree of removal of formation water from oil was carried out by spectrophotometric method; surface tension and interfacial tension were measured in a stalagmometer and a dynamic viscosity was measured at the viscometer.

The main provisions defended:

– IKHLAS brand nanodemulsifiers with ecological and technological support, protected by an innovative patent;

– a system of innovative requirements for demulsifiers at the level of oil and gas nanoecotechnologies in order to qualitatively evaluate any demulsifier;

– results of the study of property-structure, property-property relationships related to the selection of additives with optimal solvent, active phase and wetting properties for nanodemulsifiers proposed for the purpose of cleaning oil from formation waters and formation waters from oil under PPO conditions;

– new mechanisms of the breakdown and aging of oil emulsions;

– results of laboratory and experimental-industrial tests conducted in different oil fields in the direction of cleaning oil from formation water and formation water from oil under PPO conditions and scientific novelty discovered based on research results.

Scientific novelty: The dissertation work deals with the solution of an urgent scientific problem of environmental, technological, economic importance, using innovative nanoecotechnologies for purification oil from formation water and formation water from oil, as well as preventing the formation of hazardous oil nanocolloids in the conditions of PPO:

1. For the first time, nanodemulsifiers have been developed and protected by an innovative patent, which fully ensures the purification of oil from produced waters and produced waters from oil in PPO conditions from an ecological and technological point [2].

2. Numerous requirements have been developed for demulsifiers at the level of oilfield nanoecotechnologies, which allow for the qualitative evaluation of any demulsifier.

3. The concept of the critical value (C_9) of the number of carbon atoms (m) in the aliphatic chain for n-aliphatic alcohols has been introduced, at values of $m \geq 9$ the viscosity increases sharply.

4. In the conditions of PPO, as potential sources of environmental pollution,, in comparison with extremely high-stability, difficult-to-break water-oil emulsions, suspensions and

viscoelastic systems based on water-oil emulsions, “IKHLAS” nanodemulsifiers have demonstrated inhibitory properties.

5. For a more complete assessment of the efficiency of the demulsifier, the surface pressure (π) quantity and the corresponding optimal value ($\pi \geq$40-42 mC/m^2) are proposed as a colloidal-chemical criterion.

6. Based on the analogy of inverse emulsions with oil-collecting reagents on the water surface, a new destruction mechanism of oil emulsions is proposed; the simultaneous destruction mechanism of water-oil and oil-water emulsions and was developed a new aging mechanism of paraffinic oil emulsions.

7. For organic substances as demulsifiers of surfactants nature, a liquid-crystalline (and crystalline liquid) type hybrid aggregate state of the substance has been visually detected.

8. For the first time in the practice of nanoecotechnology based on nanocolloidal chemistry, used in PPO conditions, were introduced some a new concepts type: polynanobarrier property of crude oil, (hard to destroy water-oil suspensions) HDWOS, desuspensifiers, complex interaction of oxyalkylene block copolymers simultaneously with hydrophilic and hydrophobic phases of oil emulsions such as intramolecular surface activity (IMSA), as another source of environmental pollution, colloidal barns.

Theoretical and practical significance of research. In the research results, the registration of liquid crystals (and crystalline liquids) as nanodemulsifiers used in the work as a new hybrid aggregate state of matter can be used in the future to identify other types of hybrid aggregate states for organic substances. Incorporating the term desuspensioner into applied colloid chemistry may be useful in PPO technologies and other areas. The property-structure and property-property correlations found for demulsifier compositions can be used in some theoretical work. Ecological and technological efficiency of “IKHLAS” nanodemulsifiers has been confirmed by laboratory, EIT and application results. It was determined that along with the complete neutralization of oil nanocolloids, which are sources of pollution for the environment, the demulsifier

consumption was reduced by 1.7-3 times, and the consentration of oil in formation waters was reduced by 2.5-25 times to the value of 28-48 mg/dm^3.

The personal contribution of the author It is related to active participation in all stages of the work: determining the relevance, goals and tasks of the work based on the literature review; justification of research objects; conducting laboratory studies and tests; determination of scientific innovations; compilation of articles and theses; discussion of the results, preparation of the dissertation.

Research approval and implementation. The main provisions of the dissertation work have been presented in the scientific-technical council of the Azerbaijan State Oil and Industry University, in the scientific seminars of the "Petrochemical Technology and Industrial Ecology" department, as well as in the following international and regional conferences and forums discussed:

1. International Forum-Competition of Young Scientists “Problems of Subsoil Use”/ Saint Petersburg Mining University, Saint Petersburg: April 19-21, 2017.
2. XVII Republican Scientific Conference of Masters dedicated to the 94th anniversary of national leader Heydar Aliyev, SSU, Sumgayit: May 11-12, 2017.
3. International scientific-technical conference dedicated to the 100th anniversary of academician B.K.Zeynalov, Baku: June 29-30, 2017.
4. International scientific-practical conference “Prospects for innovative development of oil refining and petrochemistry” dedicated to the 110th anniversary of Academician V.S.Aliyev, Baku: October 9-10, 2018 (2 theses).
5. International Conference “Nagiyev readings” dedicated to the 110th anniversary of Academician Murtuza Nagiyev, Baku: November 30-31, 2018.
6. International Scientific Conference “Current problems of modern chemistry”, dedicated to the 90th anniversary of the Institute

of Petrochemical Processes named after Y.H.Mammadaliyev of ANAS, Baku: October 2-4, 2019.

7. XVI International Scientific and Practical Conference International Trends in Science and Technology", Warsaw: 31 august, 2019.

8. International Scientific and Technical Conference dedicated to the 100th anniversary of ASOIU, Baku: May 7-8, 2020.

9. International conference on actual problems of chemical engineering, dedicate to the 100th anniversary of the Azerbaijan State Oil and Industry University, Baku: December 24-25, 2020.

10. Online scientific conference of young researchers and doctoral students dedicated to the 98th anniversary of the birth of the National Leader Heydar Aliyev. ASOIU, Baku: May 21, 2021.

11. 1st International Scientific and Practical Internet Conference "Discovering New Horizons in Science and Prospects for Implementation of Innovations". Ukraine, Dnipro: July 7-8, 2022.

The annual actual and expected economic benefits from the application of the "IKHLAS" nanodemulsifiers proposed in the dissertation are $604.402 and $2.27 million, respectively (the relevant act is presented in Appendix 5 to the dissertation).

The name of the organization in which the dissertation work was carried out. The dissertation work was completed at the Department of "Petrochemical Technology and Industrial Ecology" of the Azerbaijan State Oil and Industry University.

Structure and volume of dissertation: The introduction to the dissertation consist of 13305, chapter I - 42264, chapter II - 27872, chapter III - 63525, chapter IV - 62718 signs, totaling 211493 signs were explained in the text were signed.

MAIN WORK CONTENT

The introduction substantiates the relevance of the topic, defines the goals and objectives of the research, provides information about the main provisions put forward for defense, the scientific novelty of the work, the theoretical and practical significance, the

approval and application of the research, the personal contribution of the author and the scope of the work.

The first chapter presents an analytical literature review on the topic. The information on demulsifiers of leading companies in the field (DEMTROL brand owned by Dow Company, Randem brand owned by Rauan Nalco Company, Kemelix brand owned by Croda Oil and Gas Company, England, reverse demulsifiers owned by the People's Republic of China, Dissolvan brand manufactured by Hoechst, Basf, Clariant Companies in Germany,; TND brand with liquid-crystal properties owned by Tyumen State University in Russia and ND brand owned by Khazar, MSERA IPChP, Alkan MSERA IPChP, Alkan LTD Company, Oil Gas Scientific Research Project Institute, SOCAR in Azerbaijan ND brand) was systematically analyzed from an ecological and technological perspective, and positive aspects and shortcomings were highlighted. In conclusion, the numerous results of the review made it was possible to determine the relevance of the topic at the level of oil and gas nanotechnologies, its goals and objectives.

The second chapter describes research objects and methods. In this work, as research objects, importance was given to the non-ionic surfactants. The highly efficient deemulsifiers, desuspensifiers of “IKHLAS” type reagents with polynanostructure has been the focus of attention in the justification of research objects. As research methods were used: preparative tube chromatography in the separation and identification of oxyethylene esters of n-aliphatic acids from the technical mixture, thermo-chemical in the breakdown of water-oil emulsions, “bottle-test” in determining the demulsifiers efficiency, spectrophotometric for the purification of formation water from oil, stalagmometric in determining the surface-tension and interphase tension, viscometric in measurement of the dynamic viscosity.

In the third chapter, from the point of view of theoretical studies, the requirements imposed for the demulsifiers, are interpreted in more detail the results of the research works performed with the aim of substantiating the synergistic constituent components

of nanodemulsifiers used for the treatment of oil from formation waters and formation waters from oil by means of "IKHLAS" nanodemulsifiers in the conditions of the primary preparation of oil. During the preparation of a systematic analytical review on the research direction based on literature data, the requirements[4] for the demulsifiers, with additions (13-42) were reworked for the first time at the level of environmentally friendly oil-field nanoecotechnologies (abbreviated) [16]:

1. The demulsifier molecules should have a high wetting effect on the components of MAL.

2. Demulsifiers they must provide high ecological quality of formation water.

3. The value of surface pressure for demulsifier should be at least 40-42 mJ/m^2.

4. The demulsifier must have a high speed action.

5. With the aim of to break down oil emulsions and other ecologically dangerous oil nanocolloids under PPO conditions, demulsifiers should have a polynanostructure.

6. Demulsifiers should not exhibit emulsifying properties that lead to ecological and technological negatives, regardless of their a special consumptions.

7. The most suitable solvents for nanodemulsifiers are critical nanoemulsions, which with the active phase can produce a synergistic effect.

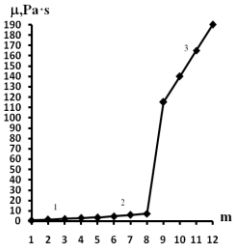
8. Active phase molecules of eco-friendly demulsifiers should easily overcome the polynanostructured barriers existing in the dispersion medium and dispersed phase.

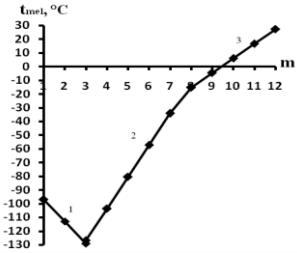
9. Demulsifiers should be desuspensifier for oil-based suspensions, and inhibitors and solvents for gas hydrates.

10. In terms of highly efficient environmental and technological support compared to oil emulsions, demulsifier surfactants must have intramolecular surface-activity.

[4] Требования к деэмульгаторам https://studfile.net/preview/4522165/page:2/

For the first time, based on the correlation relation $\mu=f(m)$ for alcohols, the concept of critical value of the number of carbon atoms ($m_{cr}=9$) in the aliphatic chain was introduced. At the value of $m_{cr}=9$, the dynamic viscosity (μ) is sharply increased (~16 times), whereas $m_{cr}<9$ the average increase value of m_{cr} at each unit increase price is ~1.5 times (graph 1) [1,16]. Based $t_{mel}=f(m)$ on the property-structure correlation regularities m=1-5 type alcohols are considered expedient as solvents in ecologically and technologically highly efficient nanodemulsifiers that can clean oil from formation water and formation water from oil (graph 2). For the first time, the quantity of surface pressure (π) was proposed as a colloid-chemical criterion for a more complete evaluation of the demulsifier efficiency from the ecological and technological point of view [12, 16, 17].





Graph 1. Dependence $\mu=f(m)$ **Graph 2. Dependence $t_{mel}=f(m)$**

The new version of the mechanism destruction of oil emulsions, is justified by the analogy between the actions of oil-collecting reagents (OCR)[5] and demulsifiers of oil emulsions (in abbreviated) [17]:

- The active phase of the OCR and of demulsifiers mainly consist of nonionic surfactants, the main colloid-chemical criterion is surface pressure (designation of surface pressure for OCR: π_{OCR}; for

[5] Гумбатов, Г.Г. Применение ПАВ для ликвидации аварийных разливов нефти на водной поверхности. / Г.Г. Гумбатов, Р.А.Дашдиев. - Баку: Элм, - 1998. – 210 с.

demulsifiers: π_d), conditions for both reagents: $\pi_{OCR} \geq 35$ mJ/m^2; $\pi_d \geq 40\text{-}42$ mJ/m^2 [12, 17] ($\pi = 72.75 - \sigma_m$);

- In the both mechanisms of action occur: competitive adsorption; competitive spreading; competitive displacement of components from MAL and, at the end, localization of film oil and destruction of emulsions.

In work, for the first time, also the mechanism of simultaneous decomposition of water-oil, oil-water emulsions was given [22]. It is known that during the synthesis of oxyethylated esters of n-alcohols, acids and other compounds, depending on the number of oxyethylene atoms in the molecule (n), a certain statistical mixture of oligomeric homologues is formed. which are characterized by the regularity of the distribution of monomers [7]. This mixture is characterized by a certain regularity of distribution according to the n value of oligomer homologues. When polynanostructured "IKHLAS" demulsifiers are dosed to crude oil, oligomeric homologues are distributed in water-oil and oil-water emulsions. More hydrophobic components with relatively low n values are distributed in water-oil emulsions, and more hydrophilic components with relatively large n values are distributed in oil-water emulsions. As a result, simultaneous decomposition of water-oil and oil-water emulsions occurs according to the requirements of the current standard [22]. It is for these reasons that polynanostructured "IKHLAS" demulsifiers are designed for the simultaneous decomposition of water-oil and oil-water [2]. For emulsions based on high-paraffin oils in the frozen state, rapid aging and an increase in stability are explained for the first time by the fast adsorption of nanosized salt crystals from the dispersed phase into MAL coatings.

The values of minimum surface tension (σ_m) should be known in determining π in order to select for technological and ecological purposes effective LCND (liquid crystal nanodemulsifier) "IKHLAS" reagents for breaking oil emulsions. With the purpose of calculating σ_m an empirical expression is proposed for nanodemulsifiers of the "IKHLAS" mark [8, 12]:

$$\sigma_m = 40.8 - 0.0034 \cdot M + (0.203 + 0.285 \cdot 10^{-4} \cdot M) \cdot \alpha. \quad (1)$$

Where M – is molecular weight; α – is degree of oxyethylation.

With the aim to ecological control the purification of formation water from oil, for the first time a correlation expression $C_{oil}=f(\sigma_M)$ of the type property-property for the investigated LCND was established based on the experimental values of σ_M):

$$C_n = 10.3\sigma_M + 36.1. \quad (2)$$

Where C_{oil} - is the concentration of oil in the formation water, mg/dm^3; σ_M - interfacial tension, mJ/m^2; a constant value of 36.1 mg/dm^3 can be estimated as the average value of the maximum purification levels for highly efficient LCND samples ($\sigma_M=0$ or $\sigma_M \rightarrow 0$). The expression (2) allows for the establishment a new of correlation relationship: property-property-efficiency of applying. These results were also confirmed by industrial tests in the "Karajanbasmunai" field in 2022 (C_{oil}=36 mg/dm^3) [22].

In figure 1 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 bottles in the vertical and horizontal positions demonstrate the liquid states of the substance. Images on vials confirm the appearance of these substances in crystals. Therefore, for the studied compounds, for the first time is confirm the liquid-crystal (and crystal-liquid) state of matter with by visual method. "IKHLAS" reagents were called nanodemulsifiers because of the fact that the nanostructured demulsifiers studied in the case contain also nano-sized liquid crystals. In addition to the known aggregations of states accepted in the world terminology, is also hybrid aggregation state possible. In (crystalline) state this regard, the liquid crystals can be viewed as hybrids of liquid and solid. In some fields (for example, in the oil fields of the Republic of Kazakhstan such as "Zhetybay" "Uzen", "Tenge Oil & Gas", "Tasbulat", "Karajanbasmunai", etc.) the technology primary preparation of oil, is accompanied with formation of certain ecologically dangerous oil emulsions. These systems, called oil residues, are made up of oil nanocolloids.

Prevention of the formation of oil colloids and they processing, which are associated with negative environmental and technological manifestations, are difficult problems of oil-field chemistry [2, 4, 18].

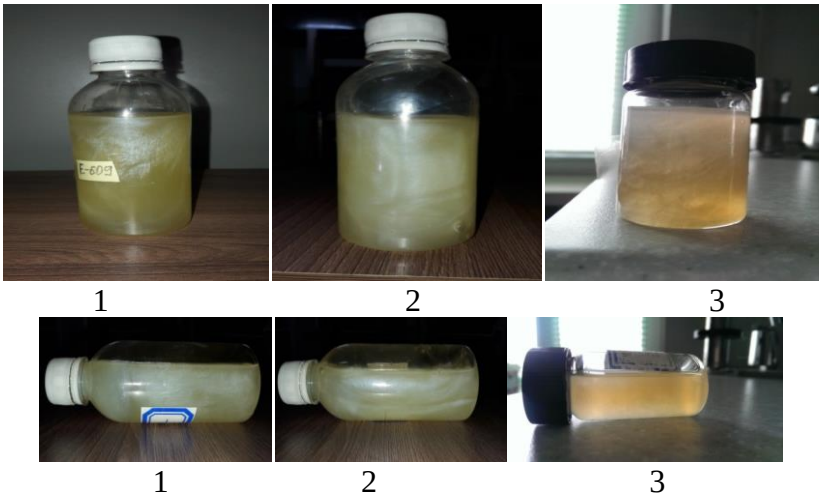


Figure 1. Appearance of some "IKHLAS" LCND samples in vertical and horizontal positions: 1 - "IKHLAS" LCND- 6003-20; 2 – "IKHLAS" LCND-5003-15; 3 – "IKHLAS" LCND-4003-10.

To achieve high quality demulsification of oil colloids, including hard to destroy water-oil emulsions (HDWOE) of creating new modifications of "IKHLAS" nanodemulsifiers with the addition of wetting components are included in the dissertation problems [7, 20, 21]. New modifications of "IKHLAS" nanodemulsifiers with the addition of wetting components such as certain ethoxylated esters of n-aliphatic acids ($C_{10}EO_{12\div16}$) with the properties of highly effective demulsifiers and inhibitors for HDWOE and O/W, which are used at some oil fields of the RK, in PPO conditions [16, 18].

The results of laboratory and experimental-industrial tests carried out with "IKHLAS" nanodemulsifiers are given in the **fourth chapter** of the dissertation in comparison with the base demulsifiers.

Chapter four is interpreted in a concise manner, because the material is quite large. Below in laconic form are the laboratory and EIT results for some oil fields.

"ZhetybayMunayGaz" oil field

Laboratory test results. The base demulsifier is Randem-2219 (Rauan Nalko, USA). In laboratory tests, 42 new samples of patent-protected nanodemulsifiers "IKHLAS-1"÷"IKHLAS-42" were used as research objects. Demulsifiers specimens consist of nanomicellar compositions of colloid and non-colloidal surfactants of different composition, low-molecular mass and relatively high molecular weight [2]. Evaluation of demulsifier efficiency from the technological and environmental point of view was carried out by the "Bottle-test" method according to known standards. The results of laboratory tests show that the efficiency of the "IKHLAS" on the properties of C_{rw} (residual water in oil, %), C_{rs} (residual salt in oil, mg/dm^3), C_{rmi} (residual mechanical impurities in oil, %), C_{ro} (residual oil in water, mg/dm^3) is respectively: 29-425, 55-462, 10-490, 13-42 times in comparison with the base demulsifier (dissertation 4.1) [3]. The maximum efficiency values are manifested for the "IKHLAS-1" nanodemulsifier. Therefore, it was proposed to conduct an EIT "IKHLAS-1" at the "Zhetybay" field.

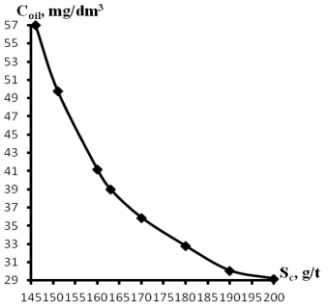
EIT results. EIT of the "IKHLAS-1" mark nanodemulsifier were carried out at the "Zhetybay" field in the ShPPPO during 03.11.14-03.12.14, based on the results of the aforementioned laboratory tests. Table 1 provides comparative information on some technological and ecological indicators based on EIT results with the base demulsifier. As can be seen from Table 1, the polyfunctional (PF) "IKHLAS-1" nanodemulsifier provides high technological and ecological quality indicators of commodity oil and formation waters by breaking up both reverse and straight emulsions at a high level under PPO conditions. The optimal thermochemical regime (S_c=160g/t; T=65-70^{o}C) is determined for the regulation of oil concentration in formation water (C_{oil}) by the dependences of $C_{oil}=f(S_c)$, $C_{oil}=f(T)$, based on EIT results (graph 3, 4). The test of demulsifier "IKHLAS-1", in contrast to the base demulsifier

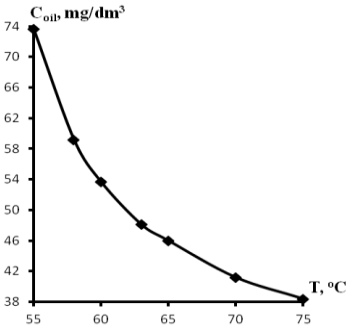
"Randem-2219" has shown himself not only a good demulsifier of ecologically dangerous of HDWOE, but also the best inhibitor for HDWOE. The official commission recommended using the "IKHLAS-1" with S_c=160 g/t on "Zhetybay" field [3, 4, 14].

Table 1

EIT results of nanodemulsifier "IKHLAS-1"

Parameters	"Randem-2219"	"IKHLAS-1"	Efficiency, times
Specific consumption of the demulsifier, g/t	204	178	1.15
Minimum and maximum concentration of chloride salts in running samples VST mg/dm^3	41-460	12-151	3-3.4
Minimum and maximum concentration of water in running samples VST №13, %	0.12-1.2	0.03-0.27	2.2-4
Dominant concentration of chloride salts in control oil, mg/dm^3	45-703	28-45	1.5-15.6
The average daily consumption of Volga river water, m^3	777	595	1.31
Number of cases that do not meet the standards in the 1-month control analysis	26	1	26
Average oil concentration in the waste water, which is sent to MRP, mg/dm^3	600-1400	30-50	20-28
HDWOE formation volume, m^3/day	100	0	many





Graph 3. Dependence $C_{oil}=f(S_c)$ **Graph 4. Dependence $C_{oil}=f(T)$**

“Tenge Oil & Gas” oil field

Laboratory tests results. The base demulsifier is “Dissolvan V 5748” (Germany). Results of laboratory tests on ecological and technological indicators with water-oil emulsions (QP-2, QU-1), belonging to “Tenge Oil & Gas”: In QP-2, the residual water in the oil and the oil in the formation water for “IKHLAS-1” respectively are 5-33 and 12-31 times less than the base demulsifier “Dissolvan V 5748”; In QU-1 the residual water in the oil and the oil in the formation water for “IKHLAS-1” respectively are 5-33 and 13-32 times less than for base demulsifier (dissertation 4.2).

EIT results (11.05.17-05.06.17). The results EIT (12.05.17) on the of record concentrations of water (0.06%) and chloride salts (10.5 mg/dm^3) for the first time to confirm the high level of effectiveness of the IKHLAS-1 nanodemulsifier on QP-2. On the QU-1 section of “Tenge Oil & Gas” also was carried out EIT (05.27.17-05.06.17). Based on the results of EIT, we can conclude that the efficiency of the “IKHLAS-1” nanodemulsifier on water (%) and chloride salts (mg/dm^3) in oil is 14 and 18 times higher than that of the base demulsifier. As an ecological advantage, it was determined that the concentration of oil in formation waters decreased 18 times from an average of ~830 mg/dm^3 to ~46 mg/dm^3. “IKHLAS-1” since 2018 has been used on “Tenge Oil & Gas” field [18, 19].

“Phystech II” oil field

Laboratory tests results. The base demulsifier is “SNPX-4315D” (Russia). The results obtained on the basis of laboratory test data on ecological and technological indicators: of residual water in oil and residual oil in formation water, in comparison with base demulsifier “SNPX-4315D” for “IKHLAS-1” it is 10-23 and 6.9-8.4 times less (table 2).

EIT results (12.04.17-17.04.17).

Information about base technology: “Phystech II” AC oil production is 430 tonnes per day. The specific consumption of the

basic demulsifier is S_c=221 g/t. The temperature of the demulsion is 65-75°C. The sedimentation period is 40-60 minutes.

Conclusions: The "IKHLAS-1" nanodemulsifier has been successfully used in the "Phystech II" field since 2018 with a specific consumption of 70 g/t and a temperature decrease of 5°C.

Scientific novelty: Inhibition of bottom-up sediments of HDWOE and HDWOS mixtures during EIT and application "IKHLAS-1" is confirms its as a highly efficient polyfunctional reagent (demulsifier-desuspensifier-inhibitory) of with respect to the corresponding ecologically dangerous oil colloids [18].

Table 2

Laboratory test results with the water-oil emulsions by "Phystech II" field (abbreviated)

Demulsifier	S_c, g/t	T, °C	τ*, min	Water, %		T. ef, times	C_{oil}, mg/dm^3	E. ef times
				initial	residue			
"SNPX-4315D"	220	75	50	29	0.30	–	174	–
"SNPX-4315D"	150	70	50	29	4.2	–	316	–
"SNPX-4315D"	100	70	50	29	5.7	–	388	–
"IKHLAS-1"	220	75	50	29	0.03	10	25	6.9
"IKHLAS-1"	150	60	50	29	0.18	23	39	8.1
"IKHLAS-1"	100	60	50	29	0.30	19	46	8.4

*settling duration; T. eff – Technological efficiency; E. eff – Ecological efficiency

"TetisAralGaz" LLP "Akkulka" oil field

Laboratory tests results presented in table 3. As follows from table 3, the residual chloride salts in the oil and as an ecoligical factor the oil in the formation water for "IKHLAS-1" respectively are 4.5 and 3.5 times less than base demulsifier "DMO-86520" ("Baker Hughes" USA).

EIT results (14.08.17-26.08.17).

The base technology data is presented in Table 4 S_c=69.6 g/t. As shown in Table 4, the dominance of chloride salts for "DMO-86520" in oil samples taken from the VST-700 (commercial) tank is

201-250 mg/dm^3. Table 5 presents the results of "IKHLAS-1" during EIT in accordance with table 4. As can be seen from table 5, the interval dominant concentration of chloride salts for "IKHLAS-1" is 100-150 mg/dm^3 (92.3%). Therefore, the technological efficiency of the "IKHLAS-1" nanodemulsifier is 6.5 times higher than that of "DMO-86520", and as an ecoligical factor, of the oil concentration in formation waters is 3.5 times higher, i.e. reduction has been achieved from 62-137 mg/dm^3 to 24-28 mg/dm^3 (table 3). Since october 2017, "IKHLAS-1" is used at the "Akkulka" field [17, 18].

Table 3

Comparative laboratory test results of "IKHLAS-1" and "DMO-86520"

Specific consumption (S_c), g/t		Formation water in oil, %		Chloride salts in oil, mg/dm^3		Ef-y, times	C_{oil}, in formation water, mg/dm^3		Ef-y, times
DMO	"IKS"	DMO	"IKS"	DMO	"IKS"		DMO	"IKS"	
70	70	0	0	285	59	4.8	62	24	2.6
60	60	0	0	329	74	4.4	70	25	2.8
50	50	0	0	380	83	4.5	105	27	3.8
42	42	0	0	422	98	4.3	137	28	4.9
average value				**354**	**78**	**4.5**	**93.5**	**26**	**3.5**

Note: Ef-y – Efficiency; DMO: "DMO-86520" demulsifier; "IKS": "IKHLAS-1"

Table 4

Information on intervals dominant concentration of chloride salts (mg/dm^3) in commercial oil for base technology (01.01.17-31.07.17)

The intervals concentration and percentage of analysis quantity, mg/dm^3						
100-150	151-200	**201-250**	251-300	301-350	351-400	401-500
13 (14.1%)	24 (26.1%)	**25 (27.2%)**	20 (21.8%)	5 (5.4%)	4 (4.3%)	1 (1.1%)

Table 5

Information on intervals dominant concentration of chloride salts (mg/dm^3) in commercial oil during EIT of "IKHLAS-1"

The intervals concentration and percentage of analysis quantity, mg/dm^3						
100-150	151-200	201-250	251-300	301-350	351-400	401-500
12 (92.3%)	1 (7.7%)	0	0	0	0	0

OGPM "Zhaikmunaigas" JSC "EmbaMunaiGas"

The laboratory test results are presented in table 6, which shows the advantages of "IKHLAS-1" compared to the base demulsifier "Dissolvan-4795" in terms of ecological and technological indicators. Technological and ecological efficiency was 12-48 and 3.3-11 times higher, respectively.

Table 6

Laboratory test results (abbreviated)

Demulsifier	S_c, g/t	T, °C	τ, min	Water, %		T. Efficiency, times	C_{oil}, mg/dm^3	Efficiency, times
				initial	residue			
water-oil emulsion belonging to S. Balkimbayev field								
"Dissolvan-4795"	246	25	60	17	0.38	–	59	–
"Dissolvan-4795"	123	25	60	17	4.8	–	490	–
"IKHLAS-1"	246	25	60	17	0.03	12	18	3,3
"IKHLAS-1"	123	25	60	17	0.10	48	43	11

Conclusion: the "IKHLAS-1,2" nanodemulsifiers were proposed for conducting an EIT at OGPM "Zhaikmunaigas" JSC "EmbaMunaiGas".

EIT results. In figure 2 presents distribution diagrams of C_{IDCChS} quantities in relation to IDCChS visually demonstrate the superiority of "IHLAS-1" (after a dual reduction in S_c) during delivery of commercial oil to "KazTransOil". Figure 3 shows diagrams of the distribution of the amount of C_{IDCRW} with respect to

IDCRW in oil samples taken from the “Gran” field”. As can be seen from figure 3, the effectiveness of “IHLAS-1” is 6.4 times higher than that of “Dissolvan-4795”. As an ecological factor in formation waters, a decrease in oil concentration by an average of 8.5-9 times is achieved, i.e. from 170-270 mg/dm^3 to 20-30 mg/dm^3 (table 7 and 8).

Conclusion: From 07.01.19 at the OGPM “Zhaikmunaigas” the nanodemulsifiers “IKHLAS-1, 2” are used [18, 19].

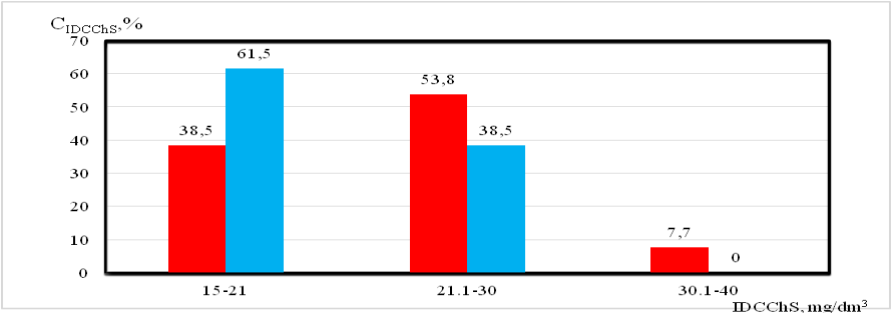


Figure 2. Diagrams for C_{IDCChS} (red: “Dissolvan”; blue: “IKHLAS”)

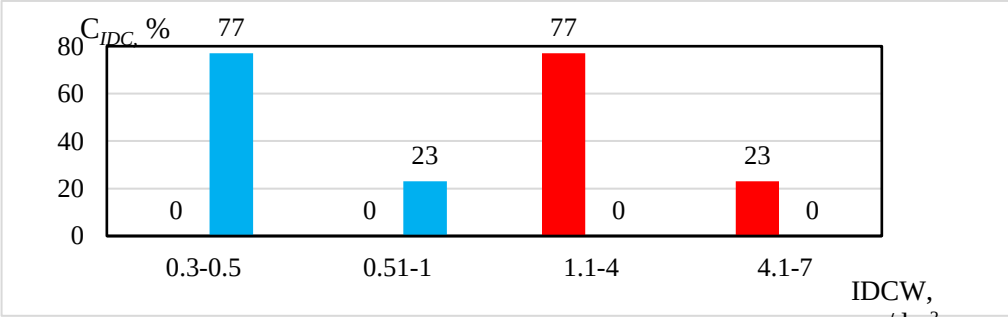


Figure 3. Diagrams for C_{IDCRW} (red: “Dissolvan”; blue: “IKHLAS”)

Table 7

Values of C_{IDCRO} in formation water samples during basic technology

C_{IDCRO}, mg/dm^3						
15-20	20.1-45	45.1-50	50.1-100	100.1-170	**170.1-270**	270.1-400
the number of analyzes (n_i) for each C_{IDCRO}						
0	0	0	2	2	7	1
B= (n_i/n_o)·100, %, where n_o is the total number of analyzes						
0	0	0	16.7	16.7	**58.3**	8.3

Table 8

Values of C_{IDCRO} in formation water samples during the EIT

C_{IDCRO}, mg/dm^3						
15-20	**20.1-30**	30.1-50	50.1-100	100.1-170	170.1-270	270.1-400
the number of analyzes (n_i) for each C_{IDCRO}						
1	10	1	0	0	0	0
B= (n_i/n_o)·100, %, where n_o is the total number of analyzes						
8.35	**83.3**	8.35	0	0	0	0

OGPM "Dossormunaigas" JSC "EmbaMunaiGas"

The results of laboratory tests: Laboratory test results are presented in table 9. Residual water concentration (C_{rw}): in the variant "IHLAS-1" C_{rw} for the "Karsak" ShPPPO, is 2-18 times less than the the base demulsifier Dissolvan-4795; value of C_{rw} 1.5-2 times less for the water-oil emulsions of the "Botahan" field; and the oil concentration in formation waters is 3-3.8; 2.3-2.7 times less than the base demulsifier "Dissolvan-4795" (table 9).

Table 9

Comparative laboratory test results with "Karsak" ShPPPO and "Botakhan" field water-oil emulsions according to ecological and technological factors

Demulsifier	S_c, g/t	T, °C	τ, min	Water, %		T. ef, times	C_n, mg/dm^3	E.ef, times
				C_{pw}	C_{rw}			
water-oil emulsion owned by "Karsak" ShPPPO								
"Dissolvan-4795"	200	63	30	8	0.06	–	75	–
"Dissolvan-4795"	170	63	30	8	0.18	–	94	–
"Dissolvan-4795"	130	63	30	8	1.3	–	131	–
"Dissolvan-4795"	100	63	30	8	2.7	–	146	–
"IKHLAS-1"	200	63	30	8	0.03	2	25	3
"IKHLAS-1"	170	63	30	8	0.03	6	28	3.3
"IKHLAS-1"	130	63	30	8	0.09	14	35	3.7
"IKHLAS-1"	100	63	30	8	0.15	18	38	3.8
water oil emulsion owned by the "Botakhan" field								
"Dissolvan-4795"	52	40	30	33	0.56	–	84	–
"Dissolvan-4795"	40	40	30	33	0.92	–	129	–
"IKHLAS-1"	52	40	30	33	0.38	1.5	36	2.3
"IKHLAS-1"	40	40	30	33	0.45	2	47	2.7

Baseline and TSS results for technological and environmental indicators:

The obtained data are presented in table 10, 11. Comparative results based on the data of tables 10, 11:

– The specific consumption of demulsifier (S_c) was reduced from 193 g/t to 115 g/t by means of "IKHLAS-1" nanodemulsifier in "Karsak" ShPPPO commodity tanks, and the concentration of oil in reservoir waters was reduced from 73 mg/dm^3 to 30 mg/dm^3 (table 10);

– In the technological tanks VST No2, VST No7 in the "Botakhan" field, the efficiency of "IKHLAS-1" nanodemulsifier compared to "Dissolvan-4795" according to the quantities of C_w, C_{Cl}, m_s and S_c respectively is 1.25, 1.5, 1.2, 1.2 was times more (table 11).

Table 10

Analysis results for "IKHLAS-1" and "Dissolvan-4795"

Date	V_o, t	Technological VST No1		Commodity VST No10, 12		Demulsifier consumption		C_n mg/ dm^3
		C_{rw}, %	C_{Cl}, mg/dm^3	C_{rw}, %	C_{Cl}, mg/dm^3	kg	g/t	
"IKHLAS-1"								
period TSS	413	0.03	43.9	≈0	47	46	115	30
"Dissolvan-4795"								
02.01.2019		–	–	0.02	47.6	–	–	–
03.01.2019	410	–	–	0.03	65.1	74.4	181	–
04.01.2019	415	–	–	0.04	44.3	83.7	202	–
05.01.2019	415	–	–	0.07	51.2	74.4	179	–
06.01.2019	404	–	–	0	39.8	93.0	230	–
07.01.2019	402	–	–	0	40.7	93.0	231	–
08.01.2019	399	–	–	0.03	53.8	65.1	163	–
09.01.2019	392	–	–	0.03	36.8	93.0	237	–
10.01.2019	399	–	–	0.06	64.5	65.1	163	–
11.01.2019	389	–	–	0.03	37.4	65.1	167	–
12.01.2019	416	–	–	0.03	36.8	74.4	179	–
average value	**404**	**0.03**	**43.7***	**0.03**	**47.1**	**78.1**	**193.2**	**72.9****

Note (tables 9 and 10): *Data corresponding to October-December 2018; ****** has been appointed before EIT;V_o – oil production; C_o - oil concentration in water; S_c - specific consumption of demulsifier; T - botl - temperature in the water bath during the test; τ - settling time in a water bath at temperature T; C_{pw} - the primary concentration of water in crude oil; C_{rw} – residual concentration of water in oil at the end of deemulsification; C_{Cl} - residual concentration of chloride salts in oil at the end of deemulsification.

Table 11

Analysis results for "Dissolvan-4795" and "IKHLAS-1" on VST No 2, 7 technological tank oils ("Botakhan" field) (02.02.19 - 12.02.19)

Date	V_o, t	Technological VST No 2, 7		Demulsifier consumption D_s (m_s və S_c)	
		C_w, %	C_{Cl}, mg/dm^3	m_s, kg	S_c, g/t
1	2	3	4	5	6
Base demulsifier "Dissolvan-4795"					
Before EIT	≈327	0.5	5587	16.9	51.6
"IKHLAS-1" in period EIT					
03.02.2019	≈326	–	–	–	–
04.02.2019	≈326	0.4	4495	15.611	47.8
05.02.2019	≈326	0.3	3211	15.611	47.8
06.02.2019	≈326	0.4	3211	13.575	41.6
07.02.2019	≈326	0,4	3982	13.575	41.6
08.02.2019	≈326	0.4	3853	13.575	41.6
09.02.2019	≈326	0.5	3211	13.575	41.6
10.02.2019	≈326	0.4	3495	13.575	41.6
11.01.2019	≈326	0.5	3853	13.575	41.6
12.01.2019	≈326	0.4	3853	13.575	41.6
average value	**326**	**0.4**	**3684**	**14.0**	**42.9**

CONCLUSIONS

1. Laboratory and industrial tests conducted under PPO conditions have confirmed that the absence of hazardous contaminants, such as HDWOE, HDWOS and visco-elastic systems, indicates that "IKHLAS" branded reagents posses multifunctional properties, including demulsifier, desuspender, depressant, inhibitor distinguishing them from known analogues.

2. Numerous requirements have been developed that can qualitatively evaluate demulsifiers. Patented "IKHLAS"

nanodemulsifiers have been found to sufficiently meet these requirements, surpassing demulsifiers of well-known companies.

3. The surface pressure (π) quantity and its optimal value, $\pi \geq 40\text{-}42$ mC/m^2 have been proposed as criteria for assessing demulsifier efficiencyand. Have been justified C_1-C_5OH alcohols as solvents.

4. “IKHLAS” viscosities are 20-25 mPa·s, densities are 900-910 kg/m^3, interfacial tensions are $\sigma_M=0$ or $\sigma_M \rightarrow 0$. For the “Karajanbasmunai” field oil, the empirical expression between the oil concentration (C_n) and σ_M $C_n = 10.3\sigma_M + 36.1$ was confirmed by EIT in 2022, C_n=36 mg/dm^3 (3 times less than Randem-2208).

5. It has been determined that “IKHLAS-1” does not exhibit the negative emulsifying properties, such as Randem-2208 (USA, Nalco), which is widely used in the region.

6. A new mechanism for the destruction of reverse emulsions, based on the analogy of action with oil-collecting reagents, a mechanism of simultaneous disintegration of water-oil and oil-water emulsions with a regular distribution of hydrophobic and hydrophilic homologues during dosing of polynanostructured “IKHLAS” demulsifiers into crude oil, a mechanism of rapid aging and increased durability in high-paraffin oil emulsions in the frozen state due to the adsorption of nanosalt crystals from the dispersed phase to the MAL have been identified.

7. According to the results of EIT conducted in several oil fields for “IKHLAS”, it has been determined that, compared to the corresponding base technologies, demulsifier consumption was reduced by 1.7 to 3 times, and the oil concentration in the produced water was reduced by 2.5 to 25 times, reaching values as low as 28-48 mg/dm^3. The annual actual and expected economic benefits of the “IKHLAS” nanodemulsifiers on the fields are shown to be $604.402 and $2.272.610, respectively (Appendix 5).

List of published scientific works on the topic of the dissertation work

1. Gasanov, A.A., Dashdiyeva, T.K. On research results of interrelation property–structure for n–alifatic alcohols // Azerbaijan Chemistry Journal, – Baku: – 2016. No1, – p. 44-48.

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Using Critical Nanoemulsions // Russian Journal of General Chemistry, – Saint-Petersburg: – 2017. 87 (8), – p. 1771-1774.

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12. Dashdiyeva, T.K. The main criteria for evaluating the effectiveness of nanodemulsifiers for cleaning oil from produced water and produced water from oil in the conditions of primary oil preparation // Proceedings of the XVI International Scientific and Practical Conference" International Trends in Science and Technology", – Warsaw: – 31 august, – 2019, – p. 43-49.

13. Dashdiyeva, T.K. Isolation and identification of individual oxyethyl esters of n-aliphatic acids using critical

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Personal contribution of the applicant in joint works with co-performers

[3,5,12,13,15,16,17,19,20,23] – have been performed independently.

[1,4,6,7,8,9,10,11,14,18,21,22] – participation in setting issues, conducting laboratory research, participation in publication of materials.

[2] – participation in setting the issue, conducting laboratory research, participation in the development of an invention.

The defense of the dissertation will take place on 30 may 2025 at 15^{00} at the meeting of the Dissertation Council FD 1.38 under the National Aerospace Agency of the Ministry of Defense Industry of the Republic of Azerbaijan.

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