Evaluation of the effectiveness of commercial demulsifiers based on polyoxyalkylated compounds in relation to oil and water emulsions of the Sarybulak oilfield

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Water and oil emulsion formation is a natural process that takes place during oil recovery and processing. Heavy oils of Kazakhstan form highly stable oil emulsions mostly stabilized by a high content of asphaltenes, resins, and other surface-active components. Oil-in-water emulsions initiate the corrosion of equipment and cause transportation issues. Dewatering of oil emulsions is economically reasonable and requires universal techniques which could be applied to any sort of oil. In this study, the chemical composition of crude oil from the Sarybulak oilfield was determined, and commercial demulsifiers of Basorol brand were applied to these water-in-oil emulsions. The natural stabilizers content (asphaltenes and resins) was determined and correlated with IR-spectrum data. Finally, the effectiveness of demulsifiers is compared and explained according to their structures. It has been found that the higher the relative solubility number of the demulsifier, the better water-in-oil emulsion separation efficiency and dewatering mechanism was assumed. Results of water separation showed that Basorol PE-10400 and PE-10500 are the most effective, with DE of 96% and 91%, respectively, for 30% (vol.) water-in-oil emulsion at 60°C during 1-hour treatment.

Keywords*:* oil emulsion; chemical demulsification; water-in-oil emulsion; dewatering of crude oil emulsion; heavy oils.

Сарыбұлақ кен орнының су-мұнай эмульсияларына қатысты полиоксиалкилденген қосылыстар негізінде коммерциялық деэмульгаторлардың тиімділігін бағалау

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Судағы мұнай эмульсиясының түзілуі – мұнайды өндіру және өңдеу кезінде орын алатын үдеріс. Қазақстанның ауыр мұнайлары негізінен асфальтендер, шайырлар және басқа да беттік-активті компоненттермен стабилизацияланған тұрақтылығы жоғары мұнай эмульсияларын түзеді. Судағы мұнай эмульсиялары жабдықтардың коррозиясын және тасымалдау мәселелерін тудырады. Мұнай эмульсияларын сусыздандыру экономикалық тұрғыдан тиімді және мұнайдың кез келген түріне қолданылатын әмбебап әдістерді қажет етеді. Бұл зерттеуде Сарыбұлақ кен орнының шикі мұнайының химиялық құрамы анықталды. Осы мұнайдың модельді эмульсияларын сусыздандыру үшін жұмыста Basorol маркалы коммерциялық деэмульгаторлар қолданылды. Табиғи тұрақтандырғыштар асфальтендер мен шайырлардың мөлшері анықталды және ИҚ-спектрінің деректерімен салыстырылды. Әртүрлі деэмульгаторлардың тиімділігі салыстырылды және олардың құрылымымен корреляцияланды. Деэмульгатордың салыстырмалы ерігіштігі неғұрлым жоғары болса, мұнайдағы су эмульсиясының бөліну тиімділігі соғұрлым жоғары болатыны анықталды және сусыздандыру механизмі ұсынылды. Ең жақсы деэмульсиялау нәтижесін, яғни 96% және 91% бұзылу тиімділігін сәйкесінше Basorol PE-10400 және PE-10500 30%-дық (көлем.) мұнайдағы су эмульсиясына 60°С-та кезінде 1 сағат әсер ету барысында көрсетті.

Түйін сөздер: мұнай эмульсиясы; химиялық деэмульсификация; мұнайдағы су эмульсиясы; мұнай эмульсиясының сусыздануы; ауыр мұнай.

Оценка эффективности коммерческих деэмульгаторов на основе полиоксиалкилированных соединений в отношении водонефтяных эмульсий месторождения Сарыбулак

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Образование водонефтяной эмульсии – естественный процесс, происходящий при добыче и переработке нефти. Тяжелые нефти Казахстана образуют высокоустойчивые нефтяные эмульсии, в основном стабилизированные за счет высокого содержания асфальтенов, смол и других поверхностно-активных компонентов в нефти. Эмульсии нефть/вода вызывают коррозию оборудования и другие проблемы при транспортировке. Обезвоживание нефтяных эмульсий экономически целесообразно и требует универсальных приемов, применимых к любому виду нефти. В данном исследовании был определен химический состав сырой нефти месторождения Сарыбулак и были применены коммерческие деэмульгаторы марки Basorol для разделения модельных эмульсий. Присутствие природных стабилизаторов, таких как асфальтены и смолы, было определено и сопоставлено с данными ИК-спектра. Эффективности деэмульгаторов были сравнены и соотносены с их структурой. Было обнаружено, что чем выше относительная растворимость деэмульгатора, тем эффективнее разделение эмульсии вода/нефть, а также предложен механизм обезвоживания. Результаты деэмульгирования показали, что Basorol PE-10400 и PE-10500 являются наиболее результативными с эффективностью разделения 96% и 91% соответственно для 30% (об.) эмульсии вода-в-масле при 60°С в течение 1 часа обработки.

Ключевые слова: нефтяная эмульсия; химическое деэмульгирование; эмульсия вода-в-масле; обезвоживание нефтяной эмульсии; тяжелая нефть.

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1. Introduction

The destruction of water-in-oil emulsions is one of the most serious difficulties in the collection, transportation, and processing of oil, and it necessitates varied solutions depending on the stage of oilfield exploitation [1-3]. Long-term operation of oil fields leads to the formation of stable water-in-oil emulsions with high water content (20-97%). The chemical composition and physical properties of the oil in a certain oilfield determine the stability of an emulsion during the extraction or pumping of the oil [4]. Some types of emulsions are well separated by simple gravity settling of water phase at heating, while others are rapidly divided into two phases under the action of chemical reagents, thermal or electrical treatment [5-6].

The destruction of water-in-oil emulsions occurs due to colloidal physical processes. The demulsifier displaces or dissolves the existing natural stabilizer, changes the wettability of natural emulsifying components and allows them to move from the water/oil interface into the oil or water phase [7].

The composition of produced oil varies depending on the oilfield, its density and the content of asphalt-resinous substances and paraffins that determines the need to expand the range of new technological developments and demulsifiers [8].

Heavy oils produced in Kazakhstan are characterized by high density and a large number of asphaltenes, resins, and high molecular weight paraffins [9], and crude oil forms stable water-in-oil emulsions [10]. The formation of stable water-in-oil emulsions leads to higher costs for transportation and causes the corrosion of the equipment [1]. Therefore, the dewatering of oil emulsions is an urgent problem for our country.

In this research, samples of oil from the Sarybulak oilfield of the East Kazakhstan region and its water-in-oil model emulsions were studied. Series of Basorol brand compounds were chosen because of their diversity and wide range of RSN numbers. The aim of the research is to define the effect of composite demulsifiers based on polyoxyalkylated compounds on the breaking down of oil emulsions. In addition to determining the physicochemical parameters of oil samples, the degree of dewatering of model oil emulsions was determined using a series of commercial demulsifiers of the Basorol brand to select the most effective demulsifying agent for high viscous oil emulsions with strong stability and clarify the mechanism of breaking down.

2. Experiment

2.1 Materials and methods

Oil emulsions produced from the Sarybulak oilfield located in Zaisan district of the East Kazakhstan region were analyzed (Table 1) according to Standard test methods.

A Series of commercial products of BASF Company, Germany, known as Basorol demulsifiers were tested (Table 2).

Density was identified using an areometer according to ST RK 1319-2004 (Table 1). Dynamic viscosity was measured using Brookfield digital rheometer model DV-III+. Water content was measured using the Dean-Stark distillation method. International analysis standards were followed during oil and emulsions density (ST RK 1319-2004) and chloride salts composition (GOST 21534-76) analysis.

"Spectrum - 65" Fourier IR, (Perkin Elmer) at 4000-450 cm^{-1} diapason spectrometer was used for crude oil functional groups determination.

Water-in-oil emulsions were prepared by mixing crude oil and saline water (1% wt. NaCl) and homogenized using IKA T 10 basic ULTRA-TURAX homogenizer at 18000 rpm for 10 min at 25°C. Emulsions were prepared with different water content (5%, 10%, 20%, 30%, 40%, 50%) by adding 1% wt. NaCl solution and homogenised afterwards. NaCl is added to mimic naturally-produced crude oil emulsions, and chosen concentration is explained by the fact that higher concentrations can promote dewatering [17-18]. The density of emulsions was determined at a room temperature of 25°C.

Demulsification efficiency (DE) was calculated according to the following equation:

$$\mathrm{DE} = \frac{Volume\ of\ separated\ water}{Initial\ volume\ of\ added\ water} \bullet 100\%$$

To evaluate demulsification efficiency, 0.06-0.09 grams of demulsifier was introduced and followed by thorough mixing for 5 min by means of IKA T 10 basic ULTRA-TURAX homogenizer at 8000 rpm at 25°C. This amount of demulsifier was chosen in order to achieve the minimum demulsifier consumption per ton of oil emulsion [19]. Under these conditions, the amount of the demulsifier varies between 0.13-0.2% (wt.).

The mixtures of emulsions with demulsifiers were thermostated for 1 hour and centrifuged for 15 minutes at 2000 rpm. All measurements were repeated three times with each sample.

3. Results and Discussion

The presence of high molecular weight paraffins, asphaltenes and resins identified for a sample of the Sarybulak oilfield (Table 1) contributes to the stabilization of water globules in oil emulsions. It is known that oil can be divided into three groups according to the tendency to form emulsion: highly stable emulsion, medium stable emulsion, low stable emulsion, depending on the composition of the resin, asphaltenes and physical and chemical characteristics [20].

Table 1 – Physico-chemical properties of oil sample

Parameters	The Sarybulak oil	Standard
$\rho_{25°}$ (kg/m^3)	905	ST RK 1319-2004
μ, dynamic viscosity, mPa·s	1520	ASTM D 445-1997
Water content (%)	0 or traces, less than 0.3 mL	ST RK 1314-2004
Salts content (%)	3.25	GOST 21534-76
Mechanical impurities (%)	0.0	GOST 6370-83
Asphaltenes (%)	0.2	GOST 11858
Resins (%)	8.0	GOST 11858

Table 2 – Commercial demulsifiers used for oil emulsions separation and their characteristics [11-16]

Demulsifier	Type	Structure/functional groups	[1]RSN	Other parameters
Basorol L 121	Nonionic surfactant	Block polymer based on propylene oxide and ethylene oxide	9.4	White viscous liquid
Basorol PE10400	Nonionic surfactant	Block copolymers in which the central polypropylene glycol group is flanked by two polyethylene glycol groups	24.1	White, waxy solid, viscosity
Basorol P DB-9390	Treater/dryer	Alkoxylated polyethylenimines	13.4	Active component 75-90%, Viscosity 740 cPs, 50°C. Soluble in isopropanol and aromatic solvent (10% product). Insoluble in water and kerosene
Basorol K 3800	Water dropper/Treater/dryer	Tri-functional amine alkoxylates	9.4	Viscosity 1370 cPs, 20°C
Basorol PE 10500	Nonionic surfactant	Block copolymers in which the central polypropylene glycol group is flanked by two polyethylene glycol groups	31.5	White, waxy solid, viscosity
Basorol P DB-9393	Treater/dryer	Alkoxylated polyethylenimines	5-7	Active component 80-100%, Viscosity 500-2000 cPs, 20°C. Soluble in isopropanol and aromatic solvent (10% product). Insoluble in water and kerosene

[1]RSN (relative solubility number) is determined by the demulsifier solubility in water

According to the results of the study, Sarybulak oil is medium stable in terms of the amount of high molecular weight components, and this oil sample is highly stable in terms of density and viscosity (Table 1).

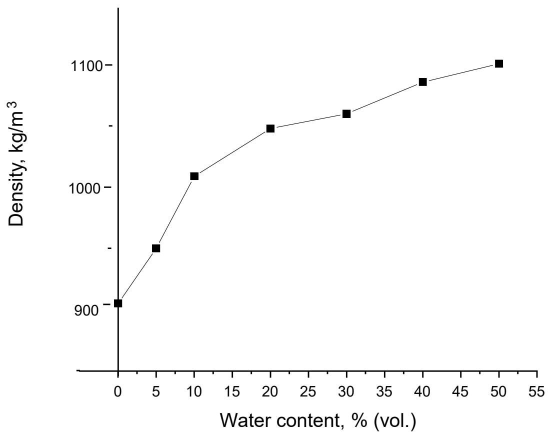

Figure 1 – Dependence of the density of w/o emulsions on the amount of water

Separation efficiency rises with increasing water content in emulsions due to the relatively high difference in densities of water and oil phase. To test this dependence densities of emulsions were measured. Densities of water-in-oil emulsions increase as water percentage increases (Figure 1). It is clearly shown that the increase of water water content leads to an increase of density. Asphaltenes densities vary from 1170 kg/m^3 to 1520 kg/m^3 and due to the presence of high molecular weight asphaltene aggregates, the density of the emulsions is higher than 1000 kg/m^3 [21].

IR spectroscopic method was used to study the qualitative composition of stabilizing components of the oil. The spectrum of the sample was determined. The intensity of peaks corresponding to hydroxide groups (bands at 3122.10 and 1400.05 cm^{-1}) is moderate, which indicates a low water content in the oil and corresponds to the physicochemical parameters defined above. Absorption bands belonging to double-bonded and condensed benzene rings were also observed in the spectrum (bands at 1605.26 and 825.03 cm^{-1}) (Figure 2). IR spectrum for oil from the Sarybulak oilfield shows the presence of long-chain paraffin structures of normal and branched structures (bands at 2924.43; 2854.05; 1458.98; 1384.31 cm^{-1}).

According to the IR spectrum, there are sharp, specific peaks that may be produced by functional groups present in high molecular weight components of oil – asphaltenes and resins (Table 3). The asphaltene molecule contains fragments of condensed hydrocarbons, heterocyclic, alicyclic hydrocarbons [12].

Table 3 – Frequencies of functional groups determined by IR spectrum analysis of oil sample from the Sarybulak oilfield

Detected wave numbers, cm^{-1}	Possible functional groups
618.95	υ-C=O
1384.31	δ-$C(CH_3)_2$
1400.05	δ-OH
1458.98	δ-$C(CH_3)$
1605.26	υ C=C
2854.05	υ-CH_2
2924.43	υ-CH_2
3122.10	υ-OH

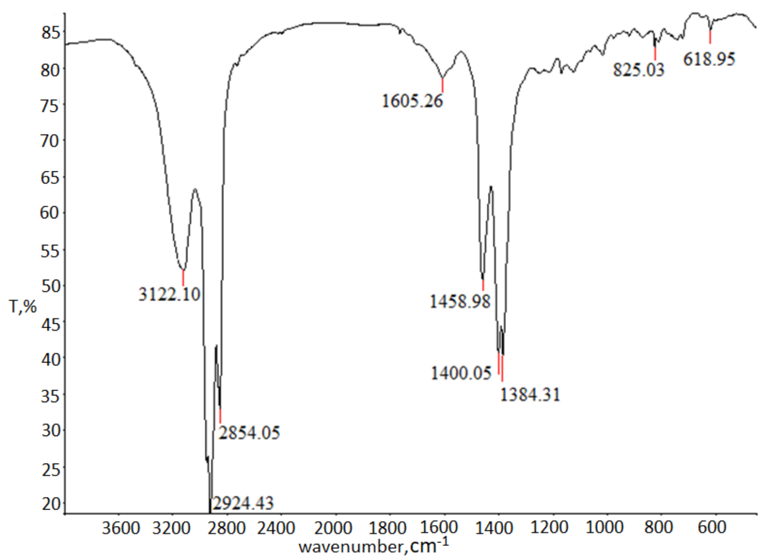

Figure 2 – IR-spectrum of oil samples from the Sarybulak oilfield

The values of the dynamic viscosity of the emulsion samples at different temperatures and different percentages of water were measured at intervals of 5°C from 25°C to 60°C (Figure 3).

The dynamic viscosity of model emulsions made of Sarybulak oil was determined at room temperature of 25°C and elevated temperatures.

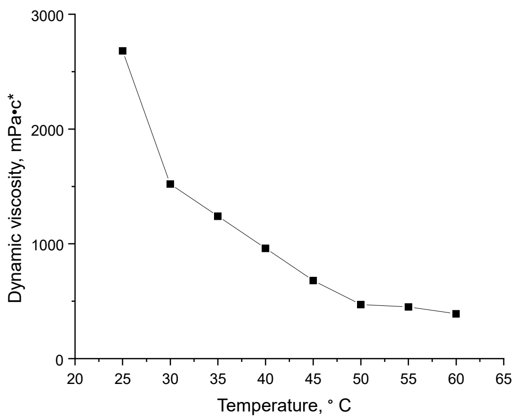

Figure 3 – Temperature dependence of dynamic viscosities of crude oil

From the graph (Figure 4), we see that as the percentage of water in model emulsions increases, so does their viscosity, which is since the increase in water content is related to the formation of hydrogen bonds between increased amounts of water molecules and natural stabilizers polar groups. Eventually, molecular flow decreases and dynamic viscosity rises. In contrast, as the temperature increases, the kinetic energy of the molecules increases too. This is followed by intermolecular forces weakening and promoting molecular flow, decreasing the dynamic viscosity that we can observe in Figure 3 and Figure 4 [22].

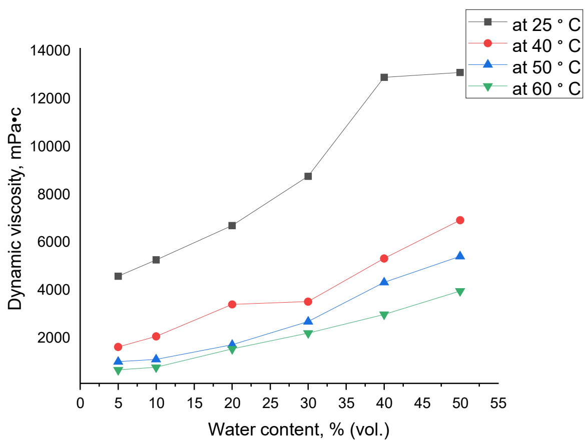

Figure 4 – Dynamic viscosity dependence of w/o emulsion on temperature

To break down the Sarybulak oilfield emulsions, the thermal dewatering of samples was carried out at 40-80°C. However, there was no observed water separation from model emulsions, and that can be explained by the ageing of the emulsion and the effect of natural stabilizers in the emulsion [13]. Therefore, thermal demulsification is not effective for these samples.

The results of physicochemical studies show that natural stabilizers increase the stability of Sarybulak oil emulsions. Therefore, commercial Basorol demulsifiers with a high effect on oil emulsions were used.

A simple addition of demulsifiers does not affect on water separation from emulsions and therefore the thermochemical separation was applied. Thermochemical treatment resulted in large water droplets spread throughout the oil phase without the formation of clear water and oil layers. To separate coalesced water droplets, thermochemical emulsion treatment was followed by centrifugation (Figure 5). Experiments were repeated several times, and separation efficiencies were the same.

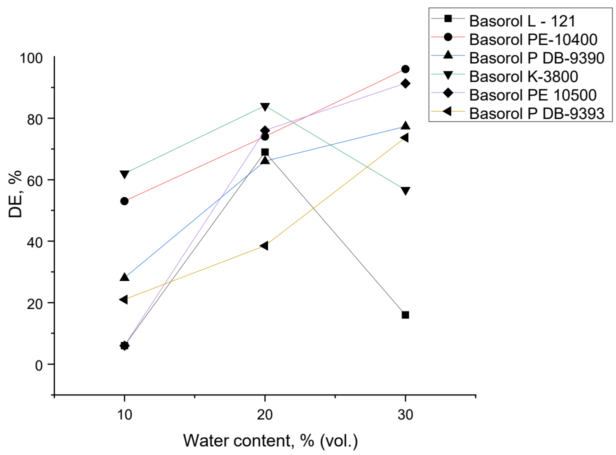

Figure 5 – Demulsification efficiency of series of Basorol demulsifiers

According to obtained results, it is clearly seen that combination of thermochemical treatment and centrifugation results in water phase separation.

The basis of demulsifier choice is its relative solubility number (RSN). RSN value shows how much water could demulsifier bind with. The higher the RSN value, the more hydrophilic the substance [14]. There is an increasing trend of demulsification efficiency when water content rises for all tested demulsifiers except Basorol L-121 and Basorol K-3800. This deviation could be related to demulsifiers' nature, their low RSN values and low molar mass. Namely, Basorol K-3800 is represented as amine alkoxylate, while Basorol L-121 is propylene oxide and ethylene oxide copolymer. These functional groups are classified as less polar ones, and they probably can not promote water droplets coalescence as well as other ones. Even though, we can see that Basorol K-3800 shows DE of 84% for w/o emulsion with 20% water content.

The most efficient demulsifier in this study is Basorol PE-10400 with DE of 96% for 30% w/o emulsion. The reason for such a good effect could be the presence of many polar groups in the copolymer structure and relatively high RSN value. A similar tendency is observed in the case of Basorol PE-10500, which differs only by the percentage of polyethylene glycol in the polymer structure (50%). Furthermore, the average molar mass of tested copolymers directly affects DE. For instance, Basorol P DB-9390 and Basorol P DB-9393 display very close DE values, while in contrast, Basorol PE-10400 and Basorol PE-10500 show higher DE values. In other research [23], Basorol RPE 3110 with a lower RSN value (9.8) and average molar mass was tested onto heavy oil emulsions with 45% DE which aligns with our approach of demulsifier efficiency. It should also be noted that DE of Basorol PBD series is similar to the results in the research of Hajivand and Vaziri [15]. A similar demulsifier, Basorol P DB-9935, attached to the magnetite nanoparticles displayed relatively high efficiency for heavy water-in-oil emulsions with DE of 82% [24].

The common emulsion separation mechanism in the case of using chemical agents considers the substitution of natural stabilizers by compounds with lower molecular weight [16]. So, the separation effectiveness depends on the choice of a suitable demulsifier with appropriate weight and nature.

4. Conclusion

To sum up, we have tested a series of commercial demulsifiers of Basorol brand. The presence of natural stabilizers correlates with IR-spectrum. In order to increase the DE, the thermochemical settling is followed by centrifugation. For the first time, commercial Basorol demulsifier's actions were studied on the high-viscous oil from the Sarybulak oilfield. Results of dewatering demonstrated that 1.2-1.8 g/L of Basorol PE-10400 and PE-10500 are the most effective, with DE of 96% and 91%, respectively, for 30% (vol.) water-in-oil emulsion at 60°C during 1-hour treatment.

Functional groups and the average molar mass of the polymer play the main role in demulsifier choice. Demulsifiers from Basorol PE series demonstrated the highest separation efficiency. Such a high dewatering ability is related to the high RSN values and molecular weight. Because the crude oil of the Sarybulak is characterized by natural surface active components, commercial demulsifiers are not universal, and their choice is dictated by crude oil composition. Moreover, such highly stable emulsions require temperature treatment and centrifugation.

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