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Evaluation of a new composition for enhanced demulsification and deposit control in resinous heavy oil emulsions

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Original scientific paper

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Abstract
This study investigates the demulsification performance, deposit formation tendencies, and rheological behaviour of highly resinous crude oils and their blended systems using both an individual demulsifier and a newly formulated laboratory composition. Crude oils X (27% water), Y (32% water), and Z (48% water), along with the mixed emulsions A (31% water) and B (41% water), were examined under various temperatures, reagent dosages, and a demulsification duration of 120 minutes. The individual demulsifier ND-12 and the newly developed W composition were evaluated by determining their optimal dosages and comparing the resulting amounts of residual and ballast water after treatment.
Under conditions of 60°C and 120 minutes, the optimal ND-12 dosage was found to be 200 g/t for X, 800 g/t for Y, and 1000 g/t for the highly stable Z emulsion, reducing ballast water to 0.05%, 0.07%, and 0.8%, respectively. Owing to its strong aggregative and kinetic stability, the Z emulsion was also analyzed at 85°C, where the required ND-12 dosage decreased to 750 g/t, and ballast water was reduced fourfold to 0.2%. For the mixed A (X+Z) and B (Y+Z) systems, the optimal ND-12 dosages at 60°C were 1300 g/t and 1100 g/t, achieving ballast water levels of 0.3% and 0.2%, respectively.
Compared with individual reagents, the laboratory-prepared W composition showed higher demulsification efficiency, reduced sediment formation related to asphaltenes, resins and paraffins, and a significant decrease in viscosity. The required reagent dosage for treating mixed oil emulsions A and B was reduced by about 2.5 times, while viscosity decreased by approximately six times. In addition, the application of the composition produced a multifunctional inhibiting effect on crude-oil complications by preventing the formation of a structural network of C_{17}–C_{40} fractions, which are prone to rapid solidification.

Keywords:
water-oil emulsion; demulsification; paraffin hydrocarbons; resinous crude oil; deposit

1. Introduction

It is well known that the stability of water-oil emulsions is primarily governed by the presence of high-molecular-weight compounds, naphthenic acids, and various surfactants that are intentionally injected into wells to enhance oil recovery. These high-molecular-weight components, particularly resins and naphthenic acids, are predominantly present in resinous crude oils, especially those with high viscosity and density (**Matiev et al., 2018a; Choi and Chan 2019**).

Resinous crude oils contain large quantities of resins and asphaltenes, which play a crucial role in the formation and stabilization of water-oil emulsions. These compounds create strong interfacial films around water droplets, leading to the development of highly stable emulsions that are exceptionally difficult to separate. The intense mixing of formation water with resinous crude oil under fluctuating production conditions (pressure and temperature variations) further increases emulsion stability, making phase separation even more challenging and resource-intensive (**Oh et al., 2002; Jia et al., 2021**).

Moreover, the abundant dispersed particles found in resinous crude oils, including resins, asphaltenes, and paraffin components, not only enhance emulsion stability, but also induce severe rheological complications. The interaction among these particles leads to the formation of complex three-dimensional network structures, which act as natural emulsifiers and significantly elevate the apparent viscosity of the emulsion systems. This rheological complexity results in higher flow resistance, reduced pumpability, and increased operational difficulties during processing and transportation (**Ma et al., 2022; Yonguep et al., 2022; Al-Sabagh et al., 2006; Huang et al., 2024**).

Traditional thermal approaches alone are often insufficient to destabilize emulsions formed from resinous

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crude oil, as the permissible operational temperature of 60°C generally fails to achieve effective separation. Although higher temperatures, such as 85°C, can improve demulsification rates, they are not always favorable due to potential negative impacts on oil quality and increased costs. Consequently, chemical demulsifiers and, more recently, specially designed synergistic compositions have emerged as effective solutions for treating resinous crude oil emulsions (**Lim et al., 2015; Thompson et al., 1985; Al-Sabagh et al., 2008; Mushtaq et al., 2023**).

Additionally, emulsions originating from different resinous crude oils are often mixed in storage tanks, resulting in systems with highly variable compositions and water contents. This blending further complicates the rheological behaviour and overall stability of the emulsions, making their treatment a critical issue for the oil industry (**Hu et al., 2018; Chen et al., 1994; Matiev et al., 2018b; Singh and Pulikkal, 2024**).

Considering these challenges, selecting reagents with superior surface activity relative to the interfacial tension of resinous crude oil and evaluating their performance under controlled laboratory conditions is an essential and urgent focus in the field of demulsification research (**Tan et al., 2007; Jinxin et al., 2006; A-Sabagh et al., 2003**).

This study aimed to comprehensively evaluate how different reagents and synergistic compositions influence the demulsification performance, deposit reduction, viscosity behaviour, and paraffin hydrocarbon distribution in water-oil emulsions with different stability characteristics.

2. Materials and Methods

In this study, a comprehensive set of laboratory-based experimental methods was employed to investigate the demulsification behaviour, deposit formation, viscosity changes, and paraffin hydrocarbon distribution in water-oil emulsions derived from resinous crude oil samples. The overall methodology included the stages as follows.

2.1. Selection and preparation of oil samples

Crude oil samples were collected from three different fields: Surakhani (X), Pirallahi (Y), and Umbaki (Z). Each oil emulsion used as the object of study differs in composition, namely in the content of high molecular weight components, as well as in the aggregate and kinetic stability of the emulsion. Furthermore, since it is known that another factor complicating emulsion breaking at oil terminals is oil blending, mixed oil A was prepared by blending oils X and Z, while mixed oil B was prepared by blending oils Y and Z. The selection of these combinations is based on the fact that numerous studies have shown that the main reason for increased complexity of emulsified crude oils is the mixing of highly stable emulsions with easily separable emulsions. For this reason, the combination of oils X and Y was not considered in this study. The initial water content of 27%, 32%, and 48% was not arbitrarily chosen; these values correspond to the natural water saturation levels observed during field sampling. To obtain reproducible laboratory emulsions, water-oil mixtures were homogenized using a high-shear mixer at 2000 rpm for 10 minutes, a widely used condition for preparing laboratory emulsions of viscous crude oils (**Jennings and Weispfenning, 2005; Ala and Daraboina, 2024; Sjöblom, 2006a**).

2.2. Determination of emulsion stability (bottle test)

The stability of the prepared emulsions was evaluated using the conventional bottle test method. Emulsions were transferred into graduated glass cylinders and allowed to settle under static conditions at laboratory temperature (~20°C). The volume of the separated water phase was recorded over 24 hours. This test provided baseline information about the inherent stability of each emulsion before adding demulsifiers and follows established procedures commonly applied in crude-oil emulsion studies (**Gasimzade, 2024**).

2.3. Investigation of demulsifier effect

The effectiveness of the commercial demulsifier ND-12 and the newly synthesized W composition (ND-12 + chloroprene + isopropanol) was examined across concentrations of 200–1400 g/t. The main experiments were performed at 50°C and 60°C, reflecting typical field operating temperatures. For the most stable emulsions, particularly Z, an additional test was performed at 85°C to understand behaviour under accelerated separation conditions. During each experiment, the demulsification process was monitored for 2 hours. After settling, the residual water and ballast water contents were determined gravimetrically. This approach enables quantitative evaluation of demulsifier efficiency and is consistent with standard methodologies reported in literature (**Jennings and Weispfenning, 2005**).

Depending on the demulsification ability of the tested demulsifier, the amount of separated water is recorded at various time intervals. The amount of separated water is calculated using the following mathematical relation (**Gasimzade, 2024**):

$$X\% = \frac{w}{w_0} 100\% \quad (1)$$

where, w_0 is the initial water content in the emulsion (mg), w is the separated water content during demulsification (mg).

Additionally, the amount of ballast water in the oil sample after demulsification is calculated using the following empirical formula, which incorporates the amount of water separated from the emulsion and the

Table 1: Physicochemical properties of oils

Parameters	X	Y	Z	Methods of conducting analyses
Water content, %	27	32	48	Dean–Stark method according to GOST 2477 using an AKOV-10 (T-AKOV-10) distillation unit with condenser and calibrated receiver, manufactured in the Russian Federation (LOIP)
Density, g/cm³	862.9	916.8	947.5	Pycnometric density determination according to GOST 3900 using PZh-2 laboratory pycnometers and a temperature-controlled bath, manufactured in the Russian Federation (Khimlaborpribor)
Asphaltene, %	1	3	8	Asphaltene precipitation and gravimetric determination according to GOST 11858-66 using standard laboratory extraction, filtration, and drying equipment (e.g. ARN-2 extraction apparatus), commonly manufactured in the Russian Federation and other CIS countries
Resin, %	10	15	12	Adsorption liquid chromatography (SARA fractionation) using laboratory chromatographic columns, baths, and standard adsorbents, manufactured in the Russian Federation
Paraffin, %	2	4	7	Paraffin content determination according to GOST 11851 using a controlled cooling and filtration laboratory setup, manufactured in the Russian Federation

initial water content of a 100 gram oil sample at various temperatures over a two-hour period (**Gasimzade, 2024**):

$$S\% = \frac{m_1 - m_2}{m_3 - m_2} 100\% \quad (2)$$

where, S is the percentage of ballast water after demulsification, (%), m_1 is the amount of water in the oil before demulsification (mg), m_2 is the amount of water separated from the oil during demulsification, (mg), m_3 is the initial mass of the emulsion sample before demulsification, (mg), $m_3 - m_2$ is the mass of the remaining (treated) emulsion after water separation (mg).

All demulsification experiments were conducted at the SOCAR Oil Pipelines Department laboratory (Baku, Azerbaijan).

2.4. Viscosity analysis

Viscosity measurements were performed to analyze the flow behaviour of the oil phases before and after demulsification. A rotational rheometer (Anton Paar MCR series or equivalent) was used, equipped with a concentric cylinder geometry. Measurements were carried out at 20°C under varying shear rates (1–1000 s^{-1}) to simulate different flow conditions. The results provided insights into the impact of demulsifiers and compositions on the rheological properties of resinous crude oils (**Nurullayev et al., 2020a; Aveyard et al., 1990**). A rotational rheometer (Anton Paar MCR series) was used to analyze the flow behaviour of the oil phases. Measurements were carried out at the SOCAR Oil Pipelines Department laboratory (Baku, Azerbaijan).

2.5. Deposit analysis (cold finger test)

To evaluate deposit formation, a cold finger apparatus was used. Oil and emulsion samples were subjected to controlled cooling cycles to simulate pipeline and processing conditions. The amount of asphaltene-resin-paraffin deposits (ARPD) was measured gravimetrically after exposure. Inhibition efficiency of ND-12 and W composition was calculated by comparing deposit mass in treated and untreated samples (**Padula et al., 2020; Yaghy et al., 2021; Peng et al., 2018; Ismayilov et al., 2022**). Deposit formation experiments were performed at the SOCAR Oil Pipelines Department laboratory (Baku, Azerbaijan).

2.6. Paraffin hydrocarbon distribution analysis

The detailed composition of paraffin hydrocarbons in the deposits was analyzed using gas chromatography-mass spectrometry (GC/MS). The deposits were extracted and dissolved in n-hexane, then filtered and injected into a GC/MS analysis was performed using a Shimadzu GCMS-QP2020 system equipped with a capillary column (TR-5MS, 30 m length, 0.25 mm ID). The operating temperature program included an initial hold at 80°C followed by a ramp to 300°C. This analysis allowed quantification of short-chain (C_{12}–C_{16}) and long-chain (C_{17}–C_{40}) paraffin fractions, providing insight into molecular transformations induced by different demulsification treatments (**Nurullayev et al., 2020b; Huang et al., 2003; Ganeeva et al., 2016; Ismayilov et al., 2025; Ismayilov et al., 2017**). GC/MS analysis of paraffin hydrocarbons was carried out at the Intertek International Laboratory (Baku, Azerbaijan).

2.7. Data analysis and evaluation

The collected data were statistically analyzed to determine the effects of temperature, reagent concentration, and composition type on demulsification efficiency, deposit reduction, viscosity stabilization, and paraffin hydrocarbon profile. Comparative evaluations were performed among individual oils and mixed systems to identify optimal treatment strategies for resinous crude oil emulsions (**Jennings and Weispfenning, 2005; Wang et al., 2025; Zu et al., 2024**).

Table 2: Demulsification of oil samples at different temperatures

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
T=50ºC							
1	X	9.7	13.8	17.9	24.3	2.7	3.6
2	Y	8.9	14.2	18.4	23.9	8.1	10.6
3	Z	3.8	6.2	8.6	10.0	38	42.2
T=60ºC							
1	X	10.5	16.8	21.5	26.6	0.4	0.5
2	Y	9.6	15.7	20.5	25.1	6.9	9.2
3	Z	8.6	12.5	17.4	25.5	22.5	30.2

Table 3: Demulsification of oil samples with the participation of ND-12 demulsifier at a concentration of 50°C

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C=200 g/t							
1	X	16.4	22.6	25.8	26.9	0.1	0.14
C=800 g/t							
1	Y	19.6	24.8	28.7	31.5	0.5	0.7
C=1000 g/t							
1	Z	23.2	30.9	35.8	41.4	6.6	11.3

To achieve the research objective under laboratory conditions, oil samples X (27% water), Y (32% water), and Z (48% water) were selected. Notably, oils Y and Z form stable emulsions, whereas oil X produces an unstable emulsion. The physicochemical properties of these samples are summarized in **Table 1**.

All initial physicochemical parameters presented in **Tables 1–10** were not arbitrarily selected. The water content values (27%, 32%, 48%) correspond to the natural water-cut measured in the respective oil fields using the Dean–Stark method. Density, asphaltene, resin, and paraffin contents correspond to the real production characteristics of these oils, which were confirmed by laboratory analyses. The selection of these specific oils was intended to cover a wide stability range, from low-stability emulsions (X) to highly stable emulsions (Z), thereby enabling systematic comparison across all experimental stages.

The physicochemical characteristics of the studied oil samples are shown in **Table 1**. Sample X, with lower asphaltene and resin content, formed unstable emulsions, while Y and Z, with higher levels of heavy components, produced more stable systems.

3. Results and discussions

Following this characterization, the next stage of the study focused on evaluating the demulsification behaviour of the samples at different temperatures. The demulsification times (0.5–2 h) were selected according to standard bottle test procedures described in ASTM D4007 and GOST 6370, allowing time-dependent observation of separation kinetics. The results of this investigation are summarized in **Table 2.**

Table 2 presents the demulsification results of these samples at 50°C and 60°C. At 50°C, sample X showed the highest separation efficiency and lowest residual water. Increasing the temperature to 60°C improved separation in all samples, though Y and Z retained higher residual water, confirming their stronger emulsion stability. In demulsification studies, a higher amount of residual water after chemical treatment is a widely accepted indicator of stronger emulsion stability. Stable water–oil emulsions typically exhibit small water droplet sizes, a dense interfacial film enriched with asphaltenes and resins, and slow coalescence kinetics. These structural features prevent efficient water release even when temperature and demulsifier concentration are increased (**Schramm and Kutay, 2009; Sjöblom, 2006b; Schramm, 1992**). In the present study, samples Y and Z contained a higher proportion of polar resins and asphaltenes (see **Table 1**), which is consistent with their higher residual water levels, confirming that they form more stable emulsions compared with sample X.

After assessing the general demulsification behaviour at different temperatures, further detailed experiments were conducted to evaluate the performance of the ND-12 demulsifier at optimal concentrations for each oil sample at 50°C and 60°C. The summarized results of these optimized demulsification tests are presented in **Tables 3** and **4**.

Table 4: Demulsification of oil samples with the participation of ND-12 demulsifier at a concentration of 60°C

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C=200 g/t							
1	X	17.9	22.8	26.8	26.96	0.04	0.05
C=800 g/t							
1	Y	23.9	27.5	29.8	31.95	0.05	0.07
C=1000 g/t							
1	Z	30.8	39.1	44.2	47.6	0.4	0.8

Table 5: Demulsification of Umbaki oil sample at 85ºC with the participation of ND-12 demulsifier

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C_{reag}=750 g/t							
1	Z	33.5	41.3	45.6	47.91	0.09	0.2

As shown in **Tables 3** and **4**, increasing ND-12 concentration and temperature notably improved water separation in all samples. At 50°C, higher dosages enhanced separation, though sample Z maintained higher residual water, indicating strong stability. At 60°C, demulsification further improved; sample X achieved near-complete separation with minimal residual water, while Y and Z also showed significant reductions. Sample Z reached its best efficiency at 1000 g/t but still had higher residual levels than X and Y. These results confirm that higher reagent dosage and temperature are essential for effective demulsification, especially for stable resinous crude oil emulsions.

To evaluate the influence of temperature on demulsification performance, the results obtained at 50°C (see **Table 3**) and 60°C (see **Table 4**) were compared. A direct deviation analysis between corresponding samples shows the improvement achieved at elevated temperature. For sample X (200 g/t), the amount of separated water after 2 h increased from 26.9% to 26.96%, corresponding to a deviation of +0.22%. For sample Y (800 g/t), the separation increased from 31.5% to 31.95%, giving a deviation of +1.42%. For the most stable emulsion (sample Z, 1000 g/t), separation increased from 41.4% at 50°C to 47.6% at 60°C, resulting in a deviation of +15.0%. This clearly demonstrates that temperature elevation has its strongest effect on highly stable systems rich in resins and asphaltenes, such as sample Z. The term "near-complete separation" is used for sample X because the residual water after 2 h decreased to 0.04–0.05% at 60°C, which falls within the range typically considered as almost fully dehydrated crude oil in standard demulsification practice. According to established literature on demulsifier performance (**Jennings and Weispfenning, 2005; Schramm and Kutay, 2009**), residual water levels below 0.1% are widely accepted as indicative of near-complete water separation under laboratory conditions. The values obtained for sample X therefore satisfy this definition, confirming that the emulsion formed by oil X is weak and readily destabilized at moderate temperature. At 60°C, the demulsification performance showed a clear improvement across all samples, which is quantitatively supported by comparing the values in Tables 3 and 4. For sample X, the amount of separated water increased from 26.9% (50°C) to 26.96% (60°C) at 200 g/t, while the residual water decreased from 0.10% to 0.04%, corresponding to a 60% reduction in residual water content. A similar trend was observed for sample Y: at 800 g/t, separation increased from 31.5% to 31.95%, and residual water decreased from 0.5% to 0.05%, demonstrating a tenfold improvement in water removal efficiency. The most pronounced enhancement occurred in sample Z. At 1000 g/t, the separated water increased from 41.4% to 47.6%, representing a 15% relative improvement, while residual water decreased from 6.6% to 0.4%, corresponding to a 93.9% reduction. These deviations clearly confirm that the higher temperature accelerates droplet coalescence and destabilizes the interfacial film, resulting in significantly greater demulsification efficiency.

To further evaluate demulsification under extreme conditions, additional tests were performed on the most stable Umbaki (Z) oil sample at 85°C using ND-12 at the optimal concentration, where C_{reag} denotes the reagent concentration and C_{comp} denotes the composition concentration. The results are presented in **Table 5**.

As shown in **Table 5**, at 85°C and an optimal demulsifier concentration of 750 g/t, sample Z demonstrated markedly improved separation performance. After 2 hours, the amount of separated water reached 47.91%, while the residual water content decreased to 0.09% and the ballast water content to 0.2%. These results confirm that elevated temperature substantially accelerates the breakdown of highly stable resinous emulsions and sig-

Table 6: Some physicochemical properties of oil mixtures

Parameters	A	B	Methods of conducting analyses
Water content, %	31	41	Dean–Stark method according to GOST 2477 using an AKOV-10 (T-AKOV-10) distillation unit with condenser and calibrated receiver, manufactured in the Russian Federation (LOIP)
Density, g/cm³	968.1	894.7	Pycnometric density determination according to GOST 3900 using PZh-2 laboratory pycnometers and a temperature-controlled bath, manufactured in the Russian Federation (Khimlaborpribor)
Asphaltene, %	7	2	Asphaltene precipitation and gravimetric determination according to GOST 11858-66 using standard laboratory extraction, filtration, and drying equipment (e.g. ARN-2 extraction apparatus), commonly manufactured in the Russian Federation and other CIS countries
Resin, %	13	14	Adsorption liquid chromatography (SARA fractionation) using laboratory chromatographic columns, baths, and standard adsorbents, manufactured in the Russian Federation
Paraffin, %	6	5	Paraffin content determination according to GOST 11851 using a controlled cooling and filtration laboratory setup, manufactured in the Russian Federation

Table 7: Demulsification of oil mixtures at 50ºC with the participation of ND-12 demulsifier

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C_{reag}=1200 g/t							
1	B	19.0	27.5	34.4	40.7	0.3	0.5
C_{reag}=1400 g/t							
1	A	18.0	21.6	26.8	30.6	0.4	0.6

Table 8: Demulsification of oil mixtures at 60ºC with the participation of ND-12 demulsifier

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C_{reag}=1100 g/t							
1	B	19.2	29.1	36.0	40.9	0.1	0.2
C_{reag}=1300 g/t							
1	A	17.5	22.5	27.6	30.8	0.2	0.3

nificantly reduces the amount of water remaining dispersed within the oil phase.

Although the improvement is notable, such high temperatures are not typically feasible in field operations due to increased energy consumption and the risk of altering crude oil quality. Therefore, laboratory testing at 85°C was performed strictly to understand the limiting behaviour of the most stable emulsion (sample Z) under extreme conditions.

The conducted investigations demonstrate that the breakdown mechanism of crude oil emulsions rich in resins and asphaltenes is directly dependent on temperature and on the strength of the interfacial film formed around the water droplets. In such emulsions, the high concentrations of resins and asphaltenes increase the elasticity of the interfacial layer and hinder the collision and subsequent coalescence of droplets. Experiments performed in the range of 50–60°C confirm that although droplet mobility increases at these temperatures, the interfacial film remains highly resistant; therefore, demulsification becomes possible only at very high demulsifier dosages. This approach is not considered economically practical, as it results in increased reagent consumption and prolonged processing time.

Raising the temperature to 85°C weakens the surface tension and structural strength of the interfacial film, which sharply enhances droplet coalescence and leads to significantly faster demulsification. The results obtained for highly stable emulsions, such as the Z crude oil sample, clearly indicate that temperature elevation is the primary factor ensuring the accelerated separation of the aqueous phase. However, the use of such high temperatures may negatively affect certain quality characteristics of the crude oil and require additional energy input under field conditions. Therefore, from an operational standpoint, the main objective is to identify a demulsifier capable of effectively weakening the interfacial film at lower temperatures (50–60°C). This makes the determination of an optimal demulsifier for use within this moderate temperature range a priority for practical application.

Table 9: The composition and mole ratio of the mixture

W	ND-12 + Chloroprene + Isopropanol	4:0.75:0.25

Table 10: Demulsification of oil mixtures at 50ºC with the participation of the composition.

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C_{comp}=700 g/t							
1	B	20.4	26.8	35.3	40.9	0.1	0.2
C_{comp}=800 g/t							
1	A	18.7	24.4	28.6	30.8	0.2	0.3

Table 11: Demulsification of oil mixtures at 60ºC with the participation of the composition.

№	Oil samples	Amount of separated water, %				Residual water amount, %	Ballast water amount, %
Demulsification time, hour		0.5 h	1.0 h	1.5 h	2.0 h		
C_{comp}=450 g/t							
2	B	20.5	30.4	37.3	40.92	0.08	0.1
C_{comp}=650 g/t							
1	A	18.9	25.2	29.3	30.9	0.1	0.1

To further investigate how blending affects demulsification performance, two mixed oil systems were prepared by combining individual crude samples: mixture A (X + Z) and mixture B (Y + Z). Their key physicochemical properties are summarized in **Table 6**, enabling a comparison between individual oils and mixed systems under identical experimental conditions.

As shown in **Table 6**, mixture A (X + Z) contains less water but exhibits higher density and a greater proportion of asphaltenes, indicating a composition with stronger intermolecular interactions and higher structural complexity. In contrast, mixture B (Y + Z) has a higher water content together with lower density and asphaltene levels, which reflects a comparatively less compact and more flowable system.

To evaluate how these compositional differences influence separation behaviour, demulsification tests for both mixtures were performed using ND-12 at elevated concentrations at 50°C and 60°C. The corresponding results are summarized in **Tables 7** and **8**.

As shown in **Tables 7** and **8**, increasing both the ND-12 dosage and the temperature resulted in higher separation efficiency for the mixed oil systems. At 50°C, mixture B (Y + Z) responded more readily to the demulsifier, reaching residual and ballast water levels of 0.3% and 0.5% at 1200 g/t. The reagent concentration required to achieve significant demulsification of oil A, a blend of oils X and Y, was relatively high (1400 g/t). This is attributed to the higher asphaltene content of oil A, which maintains the emulsion in a more stable state compared with oil B. An increase in temperature to 60°C led to an enhanced demulsification rate. Mixture B achieved optimal results at a lower concentration (1100 g/t), with residual and ballast water contents reduced to 0.1% and 0.2%, respectively. Mixture A also showed enhanced performance at 1300 g/t but still retained marginally more residual water than mixture B, which reflects the higher resistance of this mixture arising from its greater proportion of heavy polar components. These findings confirm that both temperature elevation and increased demulsifier dosage are crucial for effective treatment of mixed resinous crude oil emulsions, with mixture composition playing a significant role in determining optimal conditions.

Following the tests with ND-12, a new synergistic composition labeled W was prepared to enhance demulsification efficiency. The composition and its mole ratio are presented in **Table 9**.

To evaluate the performance of the newly prepared W composition, demulsification tests were further conducted on the oil mixtures at 50°C and 60°C. The summarized results are presented in **Tables 10** and **11**.

As shown in **Tables 10** and **11**, the W composition demonstrated superior demulsification performance at both temperatures and lower concentrations compared to ND-12 alone. At 50°C, mixture B achieved the lowest residual and ballast water contents (0.1% and 0.2%) at 700 g/t, while mixture A showed good performance at 800 g/t. At 60°C, even better results were obtained: mixture B reached minimal residual water (0.08%) and ballast water (0.1%) at only 450 g/t, whereas mixture A achieved similarly low values at 650 g/t. These findings highlight the strong synergistic effect of the W composition, enabling effective emulsion breaking with reduced reagent dosage and confirming its potential as a promising alternative to conventional demulsifiers for resinous crude oil emulsions.

The comparison of the demulsification of oil mixtures at 60°C using the composition versus the individual demulsifier is shown in **Figures 1** and **2**.

To assess the rheological behaviour of the treated oil samples, viscosity measurements were carried out at 20°C under varying shear rates. Viscosity values shown in all rheological curves represent the dynamic viscosity of the crude oil samples, measured using a rotational viscometer at 20°C. The curves correspond to the optimal reagent

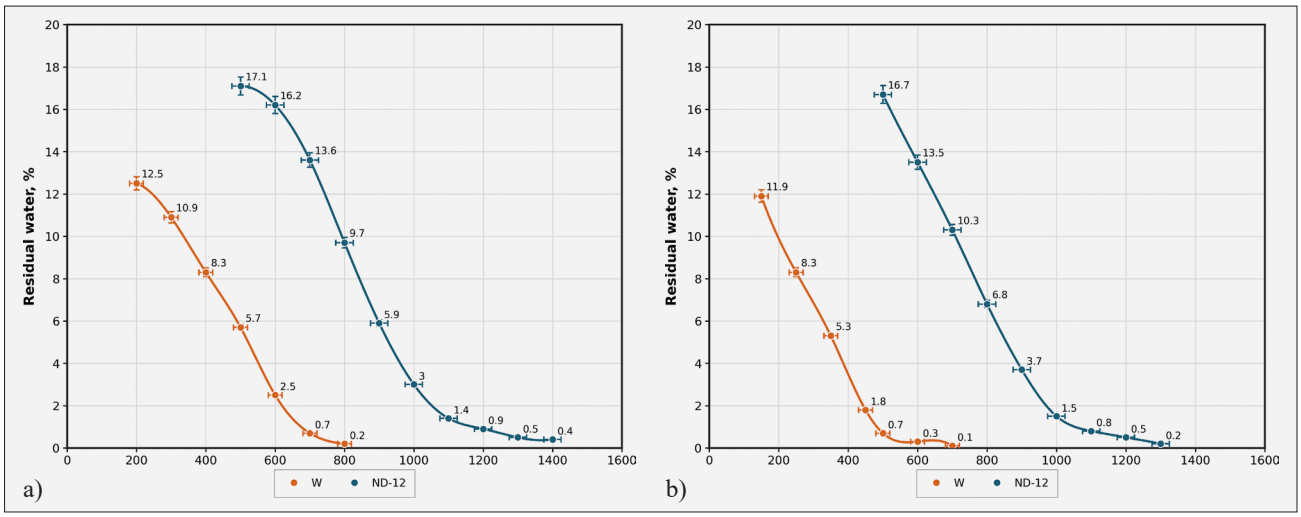


Figure 1: Dependence of ballast water amount on the concentration of ND-12 reagent and composition during the demulsification of oil A a) t=50°C, b) t=60°C.

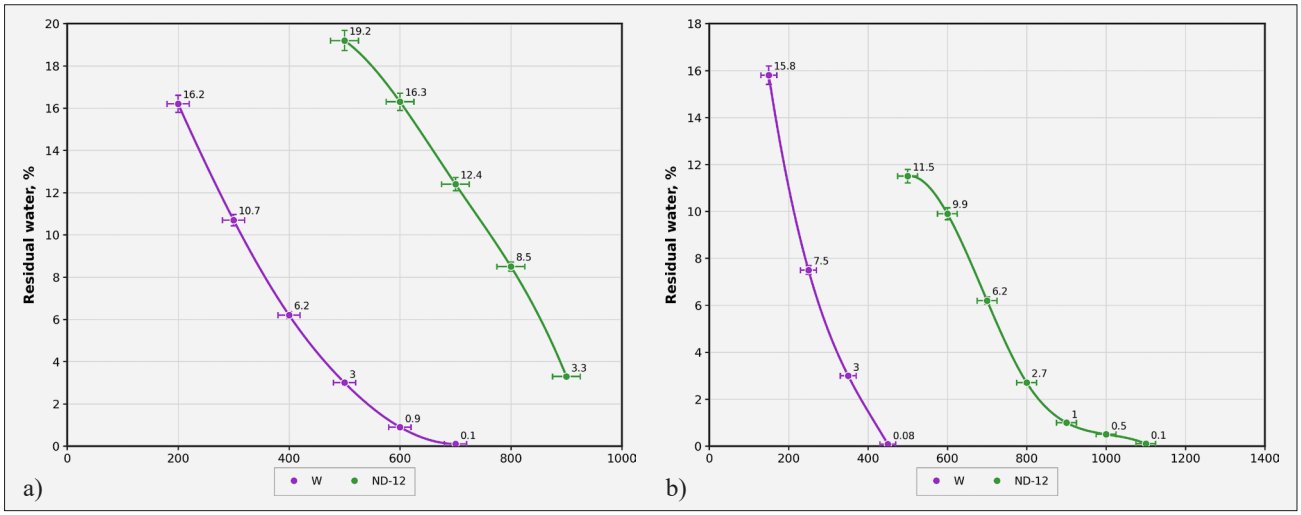


Figure 2: Dependence of ballast water amount on the concentration of ND-12 reagent and composition during the demulsification of oil B a) t=50°C, b) t=60°C.

concentrations, selected after preliminary efficiency screening.The results are illustrated in **Figures 3-6**.

The viscosity versus shear rate curves in **Figures 3–6** show that both oils experience their strongest viscosity drop in the low-shear region (approximately 5–40 s^{-1}). This behaviour is characteristic of resinous and asphaltenic systems, where even mild deformation begins to break the weak associative networks. The wider spacing observed between the concentration curves in this zone indicates that the additives exert their greatest influence when the structural framework of the oil is still largely preserved. As shear rate increases, the curves gradually move closer together, which suggests that mechanical disruption becomes the dominant factor in the breakdown of the internal structure.

For oil A, increasing the ND-12 dosage from 500 to 1400 g/t leads to a steady reduction in viscosity, with the largest deviation appearing at 5.4 s^{-1}. At higher deformation rates, the curves converge, confirming that chemical dosage becomes less critical once the internal network is already weakened by shear. The W composition behaves somewhat differently- its concentration curves remain closer throughout the mid-shear region (around 27–80 s^{-1}), yet it still produces the lowest viscosity values at all deformation rates. Oil B shows a similar general pattern, but its absolute viscosities are lower, most likely because of differences in the stability of resin and asphaltene aggregates. Here again, the W composition results in a narrower spacing between concentration curves, while ND-12 shows a more pronounced dependence on dosage.

The dual mechanism underlying the two systems, namely enhanced demulsification and viscosity reduction, can be attributed to their interactions with both the interfacial film and the bulk structural components of the crude oil. The additives contain amphiphilic molecules that are capable of penetrating the asphaltene-resin layer

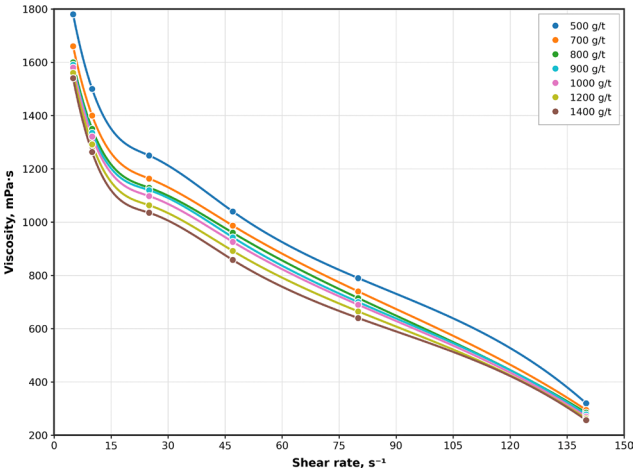


Figure 3: Viscosity changes of oil A under the influence of ND-12 demulsifier at different concentrations and shear rates at 20°C.

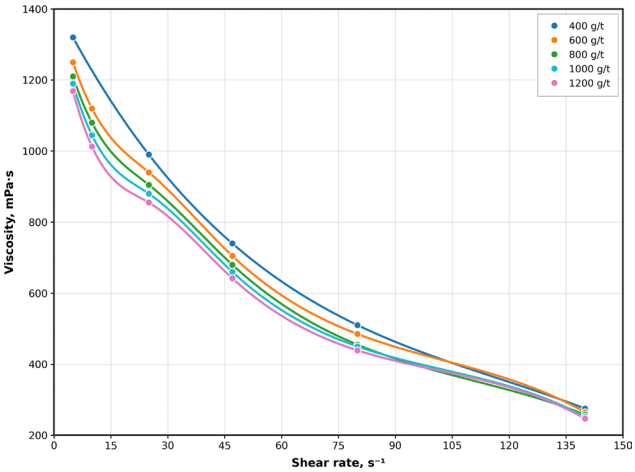


Figure 5: Viscosity changes of oil B under the influence of ND-12 demulsifier at different concentrations and shear rates at 20°C.

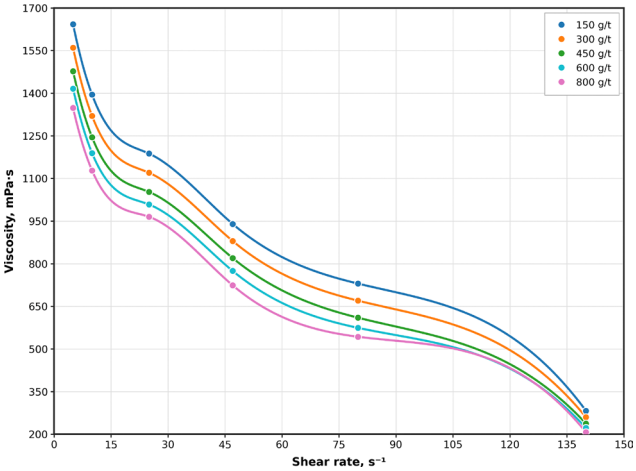


Figure 4: Viscosity changes of oil A under the influence of the W composition at different concentrations and shear rates at 20°C.

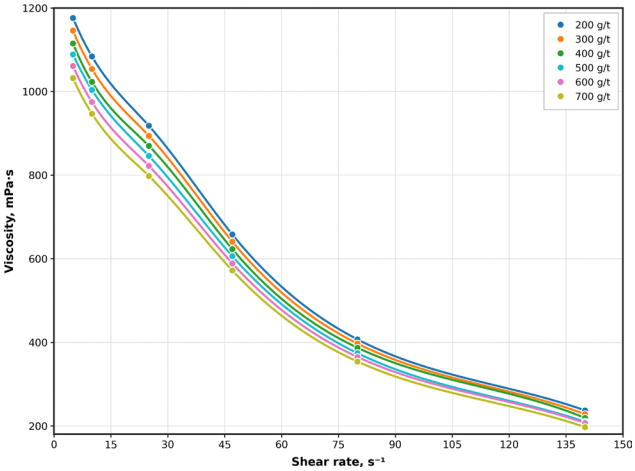


Figure 6: Viscosity changes of oil B under the influence of the W composition at different concentrations and shear rates at 20°C.

surrounding dispersed water droplets. By weakening the π–π stacking and hydrogen bonding that stabilise this film, they facilitate droplet coalescence and promote water separation. At the same time, their adsorption onto polar sites of asphaltenes and resins partially detaches these components from paraffin crystals, decreasing the connectivity of the aggregate network that contributes to high viscosity. The W composition exhibits a stronger effect because it contains more polar functional groups with higher affinity for both interfacial and bulk structural elements, making it more effective in disrupting stabilising interactions within the system.

Altogether, the rheological trends and the underlying molecular interactions show that the W composition produces a more uniform and efficient modification of the structural networks in both oils. This explains why it achieves stronger viscosity reduction while simultaneously enhancing demulsification performance.

To evaluate the impact of reagents on deposit formation, both dry crude oil and water-oil emulsion samples were analyzed. The summarized results are presented in **Table 12**.

As shown in **Table 12**, the application of ND-12 and the W composition significantly reduced deposit (ARPD) amounts in both dry crude oil and water-oil emulsions. In all cases, the W composition achieved higher inhibition efficiency, leading to lower deposit masses compared to ND-12. This effect was more pronounced in emulsion systems, where the W composition reached up to 85% inhibition. These results confirm the superior performance of the W composition in minimizing deposit formation, which is critical for reducing operational challenges such as pipeline blockages and flow assurance problems in resinous crude oil systems. This enhanced performance can be attributed to the amphiphilic nature of the W composition, which improves its interaction at the oil-water interface and promotes more effective disruption of stabilizing structures.

To further analyze the structural characteristics of the deposits, the distribution of paraffin hydrocarbons (C_{12}–

Table 12: Deposit formation in dry crude oil and water-oil emulsions

Sample	Amount of ARPD, g/100 g	ND-12		W	
		amount of ARPD, g/100 g	degree of inhibition, %	amount of ARPD, /100 g	degree of inhibition, %
X (dry crude oil)	20.8	6.86	67	5.62	73%
X (27% emulsion)	18.4	4.23	77	2.76	85%
Y (dry crude oil)	27.4	10.41	62	8.22	70%
Y (32% emulsion)	26.2	7.34	72	5.76	78%
Z (dry crude oil)	35.8	14.32	60	11.46	68%
Z (48% emulsion)	32.1	10.27	68	8.03	75%
A mixed oil (dry crude oil)	40.3	19.34	52	15.31	62
A mixed oil (31% emulsion)	39.8	15.92	60	6.37	84
B mixed oil (dry crude oil)	37.6	16.54	56	13.16	65
B mixed oil (41% emulsion)	37.2	13.02	65	6.7	82

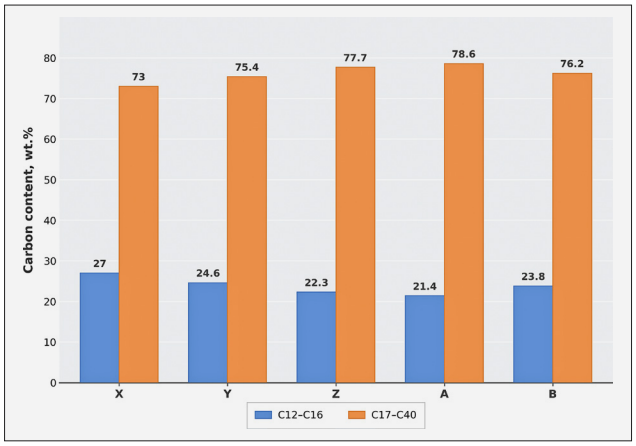


Figure 7. Paraffin hydrocarbon composition in deposits derived from water-oil emulsion samples (ND-12)

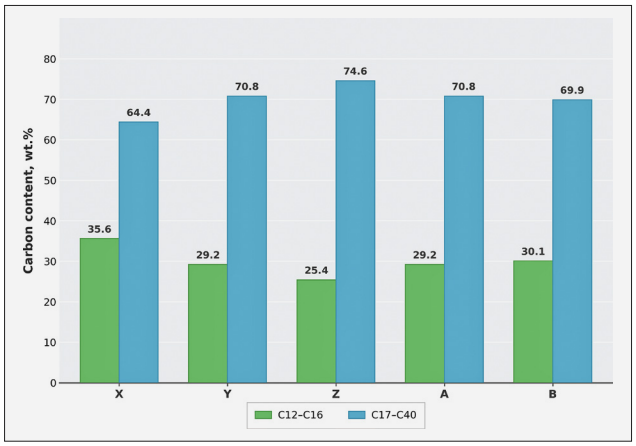


Figure 8. Paraffin hydrocarbon composition in deposits derived from water-oil emulsion samples (W composition)

C_{16} and C_{17}–C_{40}) in the emulsion samples was determined. Importantly, the analysis was conducted on the separated crude oil phase obtained following demulsification of the emulsions. The results are illustrated in **Figures 7** and **8**.

As shown in **Figures 7** and **8**, treatment with the W composition consistently increased the proportion of lower-molecular-weight paraffin hydrocarbons (C_{12}–C_{16}) in all emulsion samples, whereas the corresponding share of heavier paraffins (C_{17}–C_{40}) decreased when compared with ND-12. This shift in the paraffin profile is important from an oil-quality perspective, because a higher content of C_{12}–C_{16} hydrocarbons is typically associated with lower crystallization temperatures, reduced structural stiffness, and improved flowability.

At the molecular level, the observed increase in lighter paraffins suggests that the W composition more effectively interferes with interactions between long-chain paraffins, resins, and asphaltenes. These interactions usually promote the formation of dense, high-viscosity deposits. By weakening these associations, W facilitates the breakdown of larger paraffin clusters and prevents the formation of rigid, tightly packed structures.

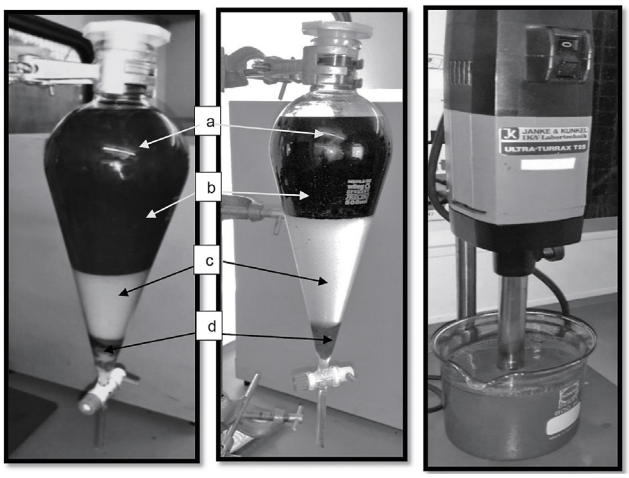
As a result, the deposits formed in the presence of W are less ordered and less prone to agglomeration, which reduces their ability to accumulate on equipment surfaces. This behaviour is consistent with a lower pour point and a reduced tendency toward wax-related flow complications, both of which are critical for maintaining stable operation in resin-rich crude oil systems.

The findings show that the W composition not only improves demulsification and decreases the total amount of deposited material but also modifies the molecular characteristics of the deposits in a way that enhances the operational quality of the crude oil.

The visual effect of the composition and the ND-12 reagent on crude oil A, based on the conducted experiments, is shown in **Figure 9**.

4. Conclusions

1. The results show that the efficiency of demulsification using the individual demulsifier ND-12 strongly depends on emulsion stability and temperature. For single crude oils, the required ND-12 dosage increased from 200 g/t for the least stable emulsion (oil X) to more than 1000 g/t for the most stable system (oil Z). Increasing the temperature to 85°C for oil Z significantly reduced the required dosage and lowered the ballast water content, con-



1) 2) 3)

Figure 9: Crude oil A: 1) effect of ND-12 reagent at 1300 g/t; 2) effect of composition W at 650 g/t; 3) initial crude oil–water emulsion prepared using a high-speed homogenizer; (a) crude oil layer at the top, (b) emulsion zone in the middle, (c) separated water phase below, and (d) sediment layer at the bottom.

firming the dominant role of temperature in destabilizing highly stable emulsions.

2. For mixed crude oil emulsions A and B, substantially higher ND-12 dosages were required to achieve effective separation at 60°C. In contrast, the laboratory-developed W composition demonstrated markedly superior performance, providing effective demulsification at significantly lower dosages. Application of the W composition reduced reagent consumption by approximately 2.5 times and decreased the residual ballast water content to 0.1% in both mixed systems.
3. Rheological and structural analyses further confirmed that the W composition effectively disrupted the associative network formed by resins, asphaltenes, and paraffins. The most pronounced effects were observed at low-shear rates, accompanied by modifications in the distribution of heavy paraffin fractions, reduced sediment formation, and improved flow stability. These findings indicate that synergistic demulsifier compositions represent a promising approach for improving demulsification efficiency and flow assurance in resin-rich crude oil systems.

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SAŽETAK

Procjena nove mješavine za poboljšano deemulgiranje i kontrolu naslaga u emulzijama teške nafte s visokim udjelom smola

Ovo istraživanje ispituje učinkovitost deemulgiranja, sklonost stvaranju naslaga i reološko ponašanje sirovih nafti s visokim udjelom smola i mješovitih sustava koji ih sadržavaju, korištenjem pojedinačnoga deemulgatora i novorazvijene laboratorijske mješavine. Sirove nafte X (27 % vode), Y (32 % vode) i Z (48 % vode), zajedno s miješanim emulzijama A (31 % vode) i B (41 % vode), ispitane su pri različitim temperaturama, količinama reagensa i trajanju deemulgiranja od 120 minuta. Pojedinačni deemulgator ND-12 i novorazvijena mješavina vode W procijenjeni su određivanjem njihovih optimalnih količina i usporedbom dobivenih količina zaostale i balastne vode nakon obrade.
U uvjetima od 60 °C i 120 minuta, optimalna količina ND-12 iznosila je 200 g/t za X, 800 g/t za Y i 1000 g/t za visoko stabilnu emulziju Z, pri čemu je količina balastne vode smanjena na 0,05 %, 0,07 % i 0,8 %. Zbog svoje snažne agregacijske i kinetičke stabilnosti emulzija Z dodatno je analizirana pri 85 °C, gdje je potrebna količina ND-12 smanjena na 750 g/t, a udio balastne vode smanjen je četiri puta, na 0,2 %. Za mješovite sustave A (X + Z) i B (Y + Z) optimalne količine ND-12 na 60 °C iznosile su 1300 g/t i 1100 g/t, pri čemu je udio balastne vode bio 0,3 % odnosno 0,2 %.
U usporedbi s pojedinačnim reagensima laboratorijski pripremljena mješavina vodene pjene W pokazala je veću učinkovitost deemulgiranja, smanjeno stvaranje naslaga povezanih s asfaltenima, smolama i parafinima te znatno smanjenje viskoznosti. Potrebna količina reagensa za obradu mješovitih emulzija nafte A i B smanjena je za oko 2,5 puta, dok je viskoznost smanjena za otprilike šest puta. Osim toga, primjena novorazvijene mješavine proizvela je višenamjenski inhibicijski učinak na probleme povezane sa sirovom naftom sprječavajući stvaranje strukturne mreže frakcija C17-C40, koje su sklone brzom skrućivanju.

Ključne riječi:
emulzija vode u nafti, deemulgiranje, parafinski ugljikovodici, sirova nafta s visokim udjelom smola, naslaga

Author's contribution

Aysel Gasimzade (Associate Professor): conceptualization, investigation and software. **Vali Nurullayev** (Associate Professor): investigation, acquisition of crude oil samples, and definition of the scientific direction.
All authors have read and agreed to the published version of the manuscript.