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THE RESULTS OF THE SPECIALISTS' AND SCIENTISTS' RESEARCHES

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High-viscosity nanoemulsions of petroleum products in a sand matrix: problems and solutions

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ABSTRACT

Introduction. Oil spills on sandy shores lead to the formation of stable emulsions that are difficult to eliminate. The research is aimed at a comprehensive analysis of the physical and chemical properties of such an emulsion and the development of effective methods for its separation using modern chemical and technological approaches that have aspects of nanolevel interaction at the phase boundary. **Methods and materials.** The object of the study was a sample of contaminated soil. Visual inspection was performed; density and dynamic viscosity were determined in the range of 25–90 °C. The separation of the emulsion was tested by centrifugation (including the use of a demulgator) and water distillation (Dean-Stark method). The content of mechanical impurities was determined gravimetrically, and their composition was determined using Fourier transform IR spectrometry. For demulsification and viscosity reduction, dilution with diesel fuel and solvent was used, followed by centrifugation. **Results and discussion.** The sample was a highly viscous (105 379 MPa•s at 25 °C) emulsion that could not be separated by standard centrifugation. The content of water (33%) and mechanical impurities (23.8%) identified as sand (SiO_2) was determined. The high stability of the emulsion is probably due to the formation of strong interfacial layers. Dilution of the sample with diesel fuel and Nefras-C280 in a 1:1 ratio significantly reduced the viscosity and achieved effective separation into the oil phase and mechanical impurities during subsequent centrifugation. **Conclusion.** It is shown that traditional methods of emulsion destruction are ineffective without preliminary modification of the system. The most effective strategy is chemical dilution followed by thermomechanical treatment, which disrupts the stable nanostructure of the emulsion and facilitates separation. The data obtained are important for developing practical recipes for oil spill response in coastal areas.

KEYWORDS: oil emulsion, nanoscale stabilization, dynamic viscosity, demulsification, mechanical impurities, Dean-Stark method, centrifugation, spillresponse

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THE RESULTS OF THE SPECIALISTS' AND SCIENTISTS' RESEARCHES

Высоковязкие наноэмульсии нефтепродуктов в песчаной матрице: проблемы и решения

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АННОТАЦИЯ

Введение. Разливы нефтепродуктов на песчаных берегах приводят к образованию устойчивых эмульсий, сложных для ликвидации. Исследование направлено на комплексный анализ физико-химических свойств такой эмульсии и разработку эффективных методов ее разделения с применением современных химико-технологических подходов, имеющих аспекты наноуровневого взаимодействия на границе фаз. **Методы и материалы.** Объектом исследования служила проба загрязненного грунта. Проводился визуальный осмотр, определялись плотность и динамическая вязкость в диапазоне 25–90 °С. Разделение эмульсии тестировали методами центрифугирования (включая использование деэмульгатора) и отгонки воды (метод Дина-Старка). Содержание механических примесей определяли гравиметрически, их состав устанавливали с помощью ИК-Фурье спектрометрии. Для деэмульсации и снижения вязкости применяли разбавление дизельным топливом и растворителем с последующим центрифугированием. **Результаты и обсуждение.** Проба представляла собой высоковязкую (105 379 мПа•с при 25 °С) эмульсию, не поддающуюся разделению стандартным центрифугированием. Установлено содержание воды (33%) и механических примесей (23,8%), идентифицированных как песок (SiO_2). Высокая стабильность эмульсии, вероятно, обусловлена образованием прочных межфазных слоев. Разбавление пробы дизельным топливом и «Нефрасом-С2 80» в соотношении 1:1 позволило значительно снизить вязкость и добиться эффективного разделения на нефтяную фазу и механические примеси при последующем центрифугировании. **Заключение.** Показано, что традиционные методы разрушения эмульсии неэффективны без предварительной модификации системы. Наиболее эффективной стратегией является химическое разбавление с последующей термомеханической обработкой, что нарушает стабильную наноструктуру эмульсии и облегчает сепарацию. Полученные данные важны для разработки практических рецептур для ликвидации последствий нефтеразливов в прибрежных зонах.

КЛЮЧЕВЫЕ СЛОВА: нефтяная эмульсия, наноразмерная стабилизация, динамическая вязкость, деэмульсация, механические примеси, метод Дина-Старка, центрифугирование, ликвидация разливов

ИСТОЧНИКИ ФИНАНСИРОВАНИЯ НАУЧНОЙ РАБОТЫ, РЕЗУЛЬТАТОМ КОТОРОЙ СТАЛА ПУБЛИКАЦИЯ: Работа выполнена при поддержке Министерства науки и высшего образования Российской Федерации по соглашению № 075-15-2022-297 в рамках программы создания и развития НЦМУ «Рациональное освоение запасов жидких углеводородов планеты».

ДЛЯ ЦИТИРОВАНИЯ:
Абусал Юсеф А.Ю., Яхин А. Р., Сильнов Д.В., Арасланова Д.И., Горшков В.А. Высоковязкие наноэмульсии нефтепродуктов в песчаной матрице: проблемы и решения. *Нанотехнологии в строительстве.* 2026;18(3):349–362. https://doi.org/10.15828/2075-8545-2026-18-3-349-362. – EDN: UXRVNE.

INTRODUCTION

Pollution of the environment, in particular coastal ecosystems, as a result of oil and oil products spills is one of the most urgent problems of modern ecology and the oil and gas industry [1, 2]. Particularly difficult are cases when oil products mixed with sand and water form stable high-viscosity emulsions that are resistant to traditional cleaning methods [3, 4]. Such systems are characterized by the formation of strong interfacial layers stabilized by nanosized particles of mechanical impurities, which significantly complicates their separation into components [5, 6].

Effective management of the consequences of such spills requires a deep understanding of the physicochemical properties of the resulting emulsions and the development of targeted methods for their demulsification [7, 8]. Existing approaches, including centrifugation, thermal and chemical treatment, often turn out to be insufficiently effective without preliminary modification of the system to reduce viscosity and destroy stabilizing barriers [9, 10].

The aim of this work was a comprehensive study of the properties of a highly viscous oil-sand emulsion selected in the contamination zone, and the development of an effective method for its separation using chemical dilution and thermomechanical methods.

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Methods and materials

Figure 1 shows the appearance of the obtained samples from different collection points.

Table 1 shows the result of the analysis of the appearance and characteristics of the samples.

Analysis of the presented tabular data shows a significant heterogeneity of contamination at various sampling points. The nature of the samples varies from thick black viscous masses to pasty and solid aggregate states, which indicates differences in the aging processes of petroleum products, evaporation of volatile fractions, and their interaction with the soil environment. The presence of characteristic odors of bitumen and organic solvents indicates a multi-component composition of the pollutant. For an in-depth study, a sample with a maximum mass and pronounced homogeneity — " Large Pond, point No. 2 (mark +3.3 m)" — was selected for subsequent determination of physical and chemical characteristics and fractionation into components (oil, water phase, solid impurities).

Separation of the emulsion by centrifugation

The method of separation of the emulsion system is based on the centrifuge effect and includes the following steps: preheating the sample to 80 °C followed by mixing

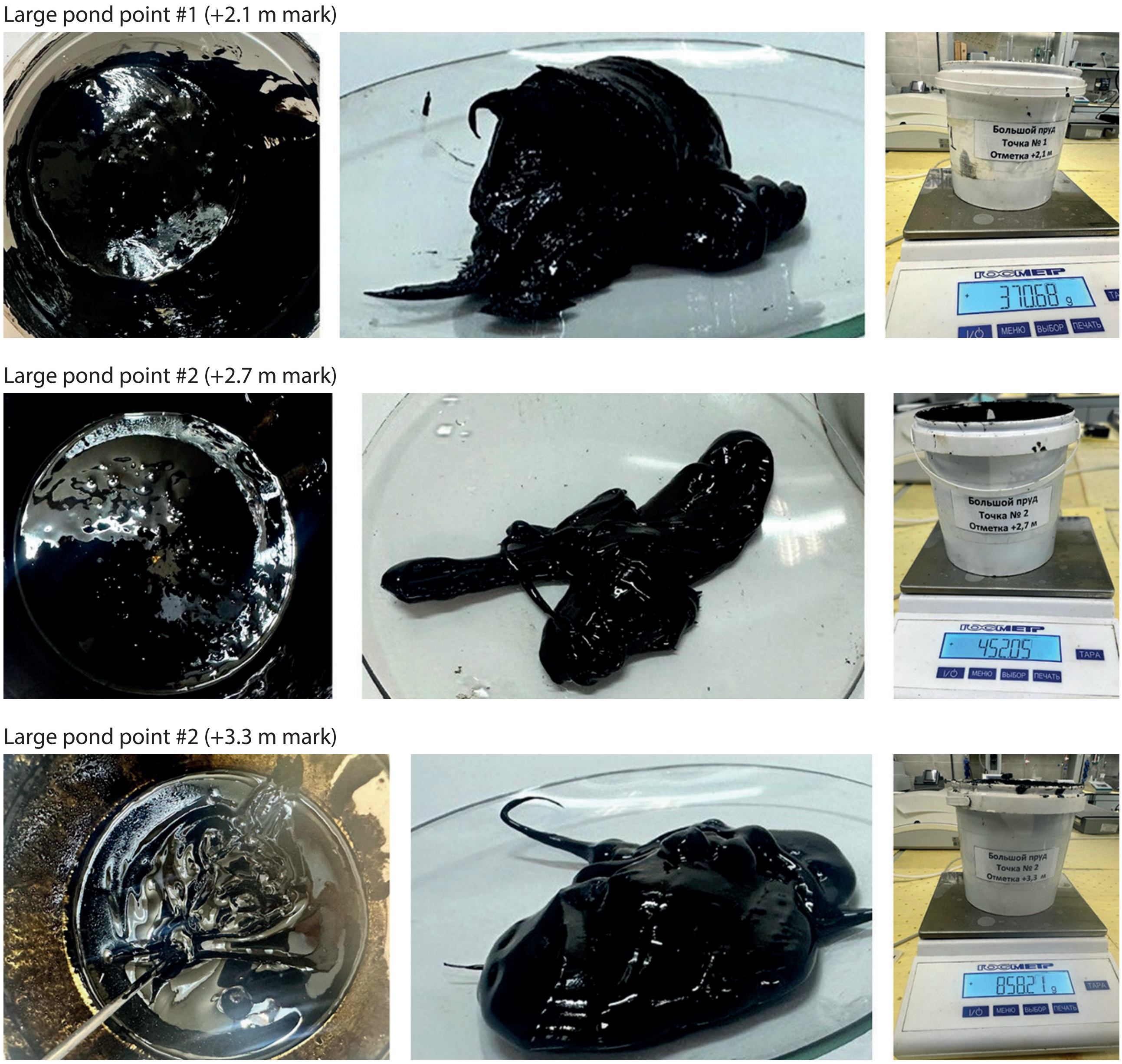

Fig. 1. Sample appearance

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Large pond point #3 (+2.8 m mark)

Small pond point #1 (+5.0 m mark)

Small pond point #3 (3.6-4.2 m mark)

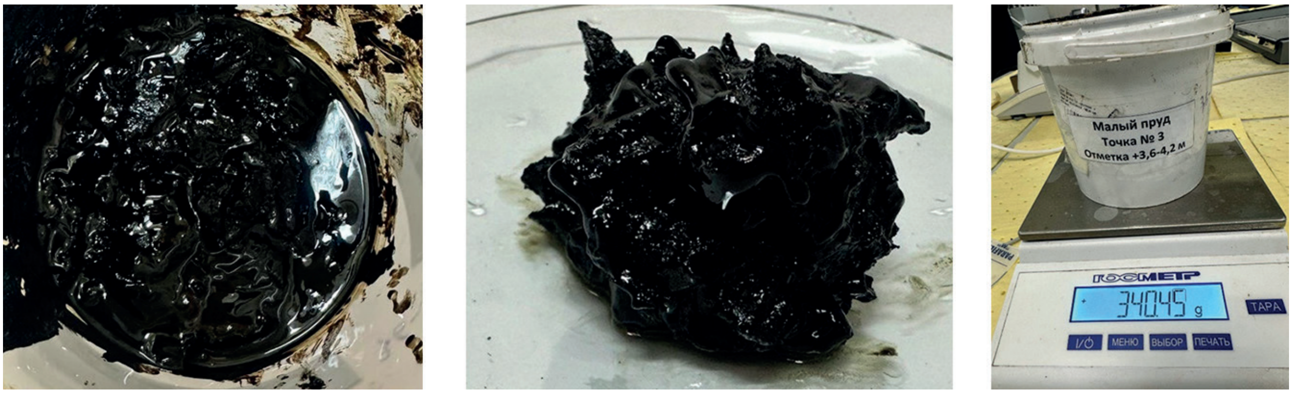

Fig. 1. The End

to achieve homogeneity, loading into centrifuge tubes and processing at 5000 rpm for 30 minutes. As a result of centrifugal action, phase separation occurs: heavy fractions (water and solid particles) settle to the bottom, while the light oil phase migrates to the upper layer (Fig. 2).

When centrifuging the sample "Big Pond point #2 (+3.3 m mark)", the separation into layers did not occur. To improve separation, a demulgator was added to the sample, thoroughly mixed and centrifuged. Sample separation did not occur.

Determination of water content by the Dean-Stark method

Dean-Stark method (GOST 2477-2014 " Oil and petroleum products. Water determination method") is a standard method for determining the water content in oil and petroleum products, based on the distillation of water with an organic solvent. During the analysis, the sample weight is mixed with a water-removing solvent (toluene or nephras) in a special flask of the Dean-Stark

Table 1. Appearance and characteristics of samples

Selection point	Appearance	Smell	Weight, g
Large pond point #1 (+2.1 m mark)	Black viscous mass	The smell of petroleum products and bitumen breaks during mixing	190
Large pond point No. 2 (mark +2.7 m)	Black liquid mass, when mixed	The smell of petroleum products, bitumen stretches	270
Big pond point #2 (mark +3.3 m)	Sticky black solid mass, when mixed	The smell of petroleum products, bitumen stretches	680
Big pond point #3 (mark +2.8 m)	Sticky black solid mass, when mixed	The smell of petroleum products, bitumen, roofing material stretches	500
Small pond point No. 1 (mark +5.0 m)	Top with a transparent layer of liquid, possibly water. Black liquid paste	Smell of fresh paint, possibly sol-vents	520
Small pond point #3 (3.6–4.2 m mark)	A stationary black mass with solid mechanical impurities	Smell of a universal solvent	160

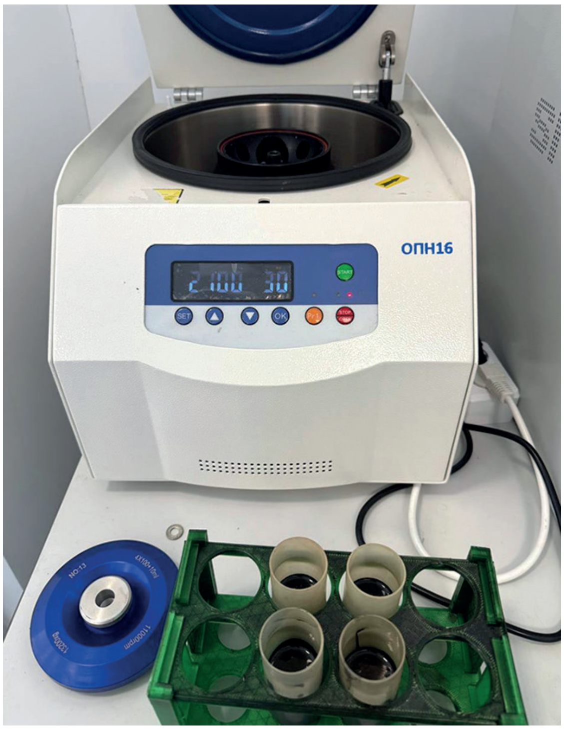

Fig. 2. The process of separation of the emulsion by centrifugation

Fig. 3. Determination of water content by the Dean-Stark method

apparatus. When the mixture is heated, solvent and water vapors are formed, which enter the refrigerator, where they condense and drain into a graduated receiver (Fig. 3).

The water content in the sample" Big Pond point No. 2 (mark +3.3 m) " was 33% (Table 2).

Content of mechanical impurities in petroleum products

The method for determining mechanical impurities in petroleum products is based on gravimetric analysis. The oil product sample is filtered through a pre-weighed paper filter with a hot solvent or a mixture of solvents (alcohol-benzene, alcohol-toluene, chloroform) to a transparent drop. After filtration, the filter is dried at 105 °C to a constant weight and re-weighed (Fig. 4).

The content of mechanical impurities (X,%) is calculated by the formula:

$$X = \frac{m_1 - m_2}{m} \cdot 100,$$

Table 2. Physical and chemical characteristics of the emulsion

Sampling point	Emulsion density, kg/m^3		Water content, %	Mechanical admixture content, %
	25 °C	50 °C		
Big pond point #2 (mark +3.3 m)	0.976	0.974	33	23.8

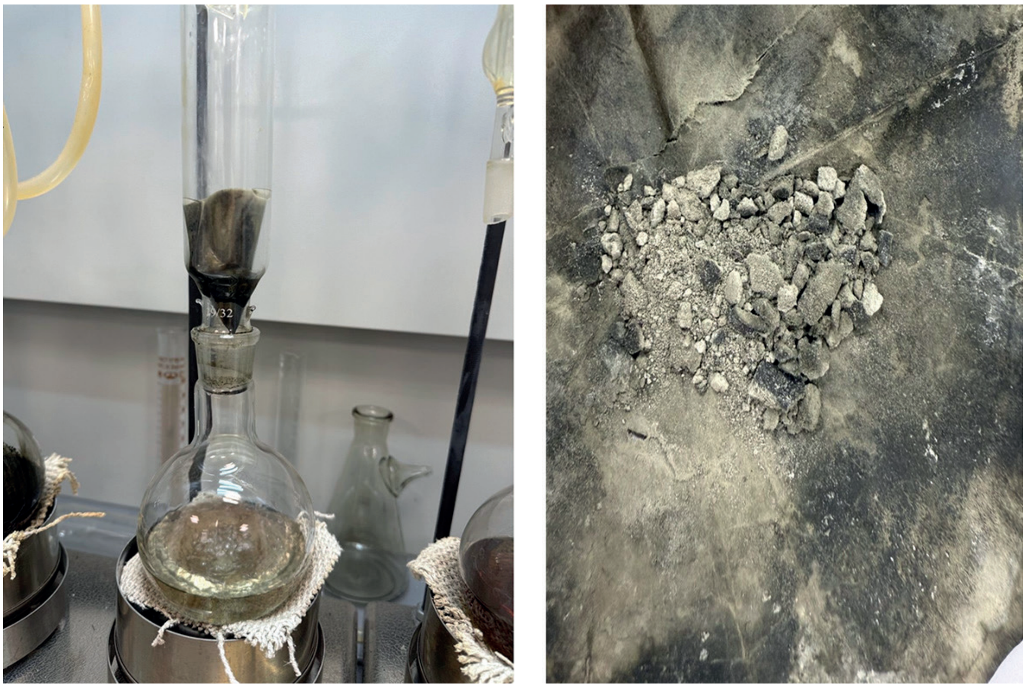

Fig. 4. Determination of the content of mechanical impurities

where m_1 – is the mass of a clean filter; m_2 – mass of the filter with impurities; m – is the mass of the sample.

The content of mechanical impurities in the sample "Big Pond point No. 2 (mark +3.3 m)" was 23.8% (Table 2).

These mechanical impurities were studied on a Fourier transform IR spectrometer and the main characteristic signals were isolated. Comparison with the database showed that the impurities are similar in composition to SiO_2 (sand, silica) (Fig. 5).

Emulsion density

The method is based on accurate measurement of the mass of a known volume of the emulsion using a calibrated pycnometer according to GOST 3900 (Fig. 6).

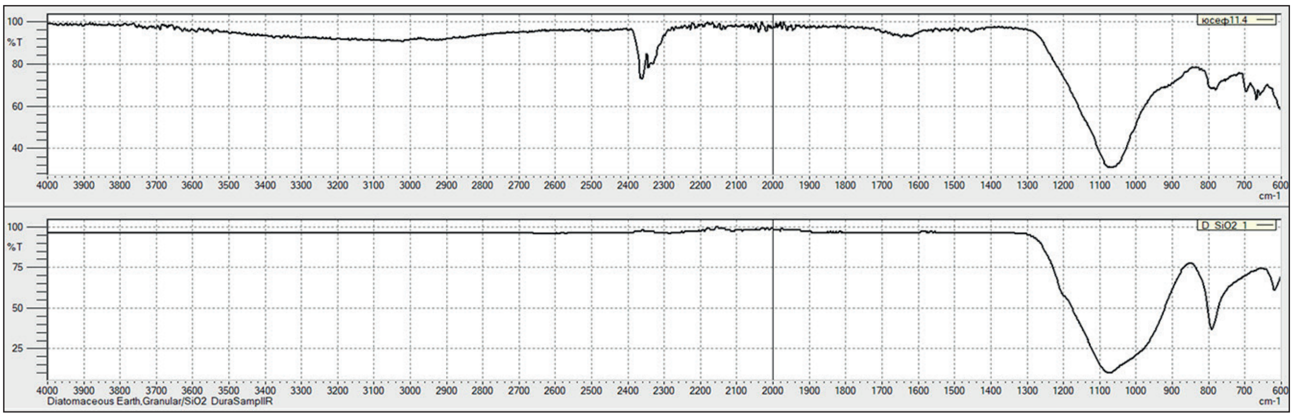

Fig. 5. IR spectrum of the mechanical impurities isolated from the sample "Big pond point #2 (+3.3 m mark)"

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Fig. 6. Density determination

The densities of the "Big Pond point # 2 (+3.3 m mark)" sample were determined at 25°C and 50°C and amounted to 0.976 and 0.974 kg/m^3, respectively (Table 2).

The density of the emulsion at ~0.976 g / cm^3 (at 25 °C), which is comparable to that of water, indicates a high degree of water cut and mineralization of the system. The proportion of water (33%) and mechanical impurities (23.8%) emphasizes the multicomponent composition, in which the sand fraction (SiO_2) functions as a stabilizer, forming a stable structure that prevents natural phase separation.

Dynamic viscosity of the emulsion

The viscosity parameters were determined on a Rheotest RN 5.1 rotary viscometer in the temperature range 25–90 °C (step 10 °C). Before each test, the sample was kept in the device's thermostat for 20–30 minutes to establish thermal equilibrium. The measurement mode provided for a fixed shift rate of 10 s^{-1}, guaranteeing reproducibility of the data (Fig. 7).

The change in dynamic viscosity with temperature is shown in Fig. 8 and Table 3.

The significant dynamic viscosity at 25 °C (105,379 MPa • s) makes it possible to classify the emulsion as a highly viscous system. A well-defined temperature dependence (a decrease in viscosity by more than two orders of magnitude to 90 °C) reflects the thermal sensitivity of its structure. At the same time, even at elevated temperatures (90 °C), the viscosity exceeds 1000 MPa • s, which justifies the combined approach to demulsification using thermal and chemical influences.

Fig. 7. Determination of the dynamic viscosity of the sample

Extraction of light fraction (oil) from the emulsion sample

Upon completion of the Dean-Stark water content analysis, the solvent was completely removed from the flask by distillation. Mechanical impurities and an oil phase remained in the residue. The liquid oil fraction was separated by decanting into a clean laboratory container for further physico-chemical studies, while solid impurities with residual oil remained in the initial flask (Fig. 9).

Next, the contents of the flask were dissolved in an alcohol-toluene mixture, followed by filtration through a blue ribbon. Mechanical impurities were retained on the filter, and the solvent was removed on a rotary evaporator.

Characteristics of the purified sample

In the purified sample "Large Pond, point # 2 (+3.3 m mark)" without mechanical impurities and water, dynamic viscosity was measured in various temperature conditions, as well as density at 25 °C and 50 °C (Table 4, Fig. 10).

After removing water and mechanical impurities, a significant drop in dynamic viscosity was recorded – from 105,379 to 7,771 MPa • s (measurements were carried out at 25 °C). At the same time, the density also decreased: from 0.976 to 0.918 g/cm^3. The obtained data clearly confirm that these components make the greatest contribution to the stability of the emulsion disperse system. At the

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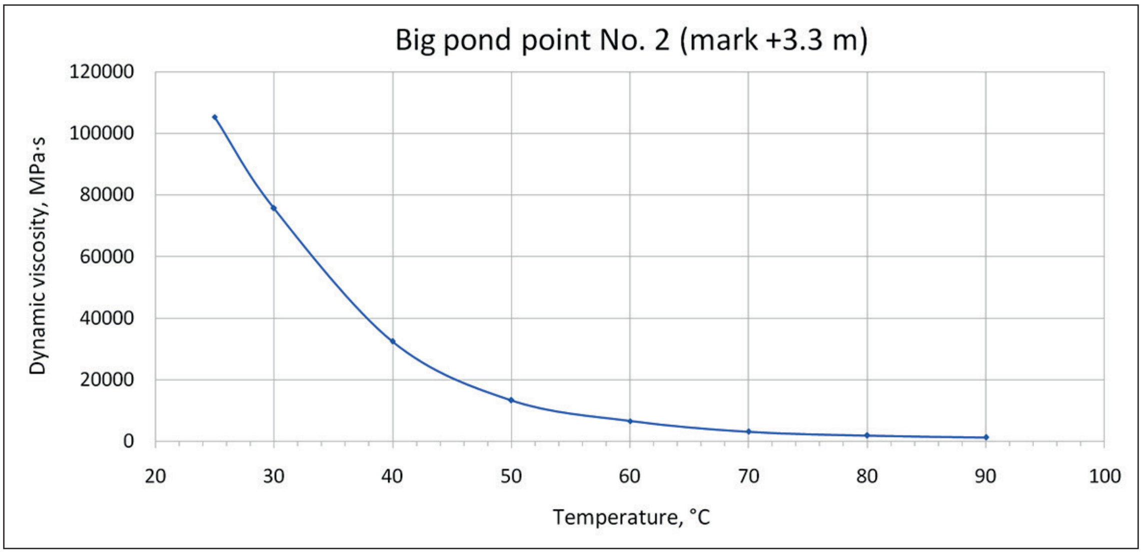

Fig. 8. Dynamic viscosity of the sample as a function of temperature

Table 3. Dynamic viscosity of a sample collected from a large pond point #2 (+3.3 m mark)

Sample location	Dynamic viscosity, MPa • s							
	25 °C	30 °C	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C
BP No. 2 (+3.3 m)	105 379	75 721	32 446	13 300	6527	3046	1820	1181

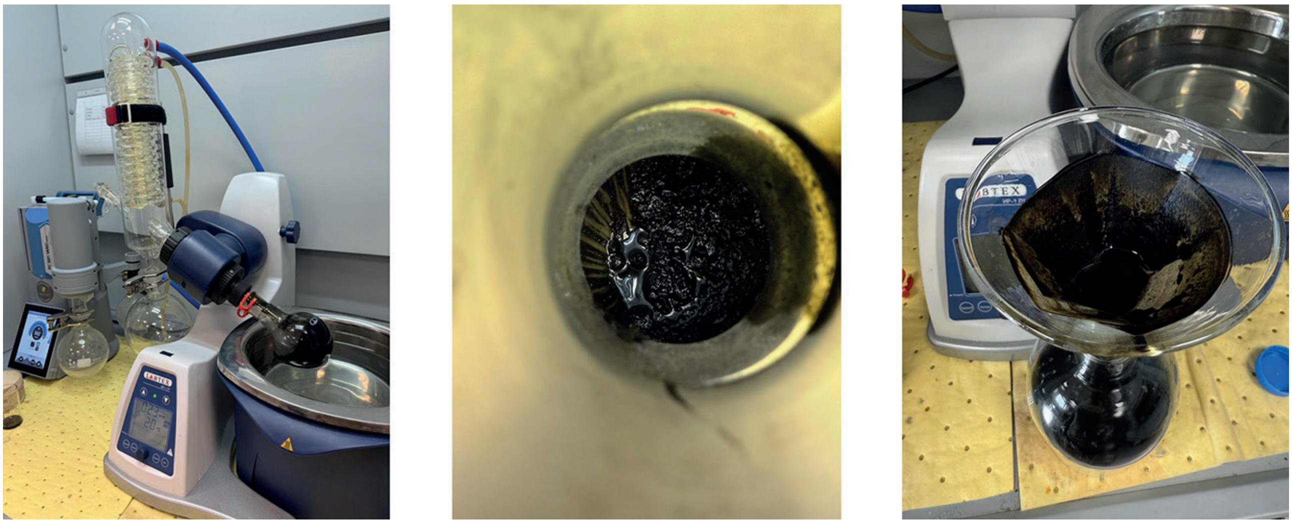

Fig. 9. The process of extracting the oil component from the "Large Pond point # 2 (+3.3 m mark)" sample

same time, the dependence of viscosity on temperature does not disappear, but the current viscosity values are already comparable with the characteristics typical for heavy oil products.

Procedure for diluting the purified sample

The purified sample was diluted with diesel fuel. For this purpose, the initial material taken at point No. 2 "Big Pond" (mark +3.3 m) was mixed with fuel in equal volume fractions (1:1). The process was carried out using a top-driven agitator at a speed of 700 revolutions per minute until a homogeneous suspension was formed. After intensive mixing was completed, the upper diesel layer was selected. Visual observation showed that the viscosity of the resulting system decreased (the results are shown in Fig. 11).

The resulting sample was heated to 90 °C and held for 30 minutes, after which it was centrifuged at a speed of

Table 4. Dynamic viscosity value of a sample taken from a large pond, point #2 (+3.3 m mark) without mechanical impurities and bound water

Sample location	Dynamic viscosity, MPa • s								Emulsion density, kg/m³	
	25 °C	30 °C	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C	25 °C	50 °C
BP t. no. 2 (+3.3 m) without mechanical admixtures and water	7771	58.75	2954	1689	1009	646	380	249	0.918	0.915

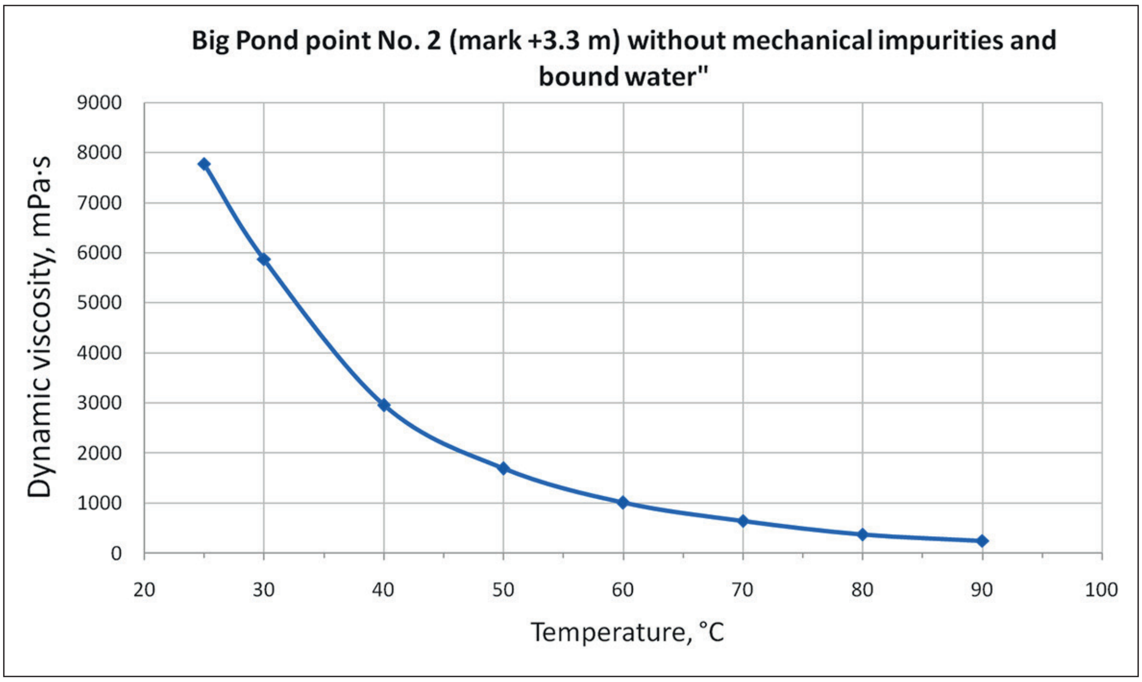

Fig. 10. Dependence of the dynamic viscosity of the sample on the temperature according to Table 4

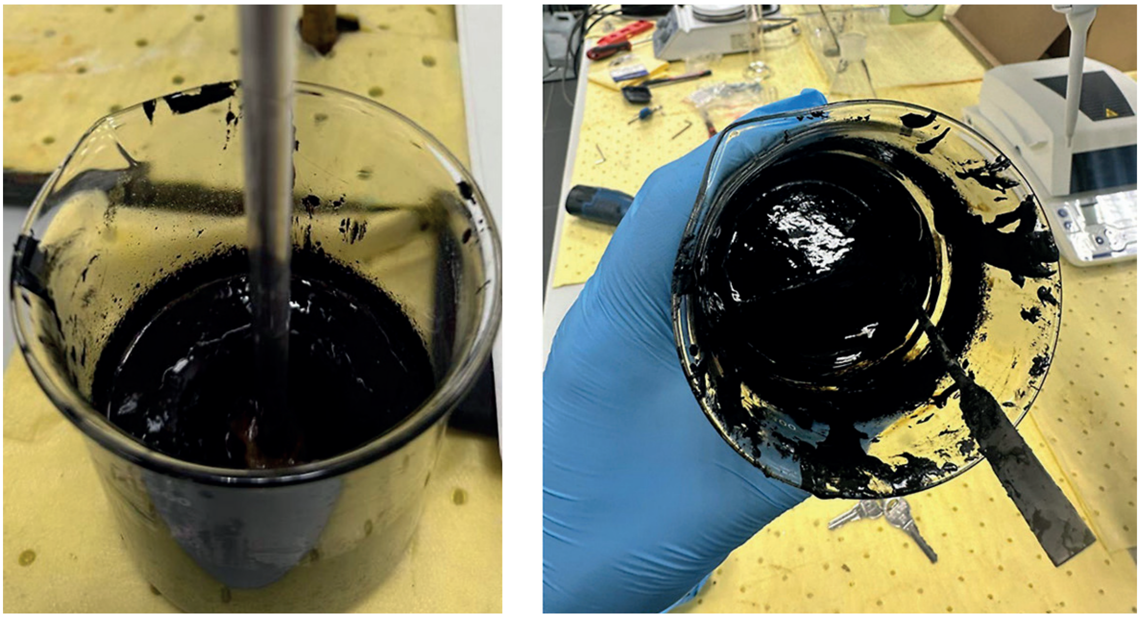

Fig. 11. Appearance of the mixture "sample + diesel fuel" in the ratio 1:1

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4000 rpm. As a result, the mixture was stratified into two components: the upper (mobile, which is an oil phase) and the lower, formed by mechanical impurities (Fig. 12). It should be noted that no water phase was detected in the sample.

Additionally, the temperature dependence of the viscosity was measured for a mixture consisting of a sample and diesel fuel in a ratio of 1:1. The measurement range was from 25 to 90 °C (the results are shown in Fig. 13 and Table 5).

When adding diesel fuel in a one-to-one ratio, a pronounced drop in viscosity was recorded. At a temperature of 25 °C, this indicator decreased to 836 MPa • s. Such a significant reduction in viscosity allows us to consider the resulting system technologically acceptable for subsequent operations — in particular, for processing by centrifugation. Low viscosity values over a wide temperature range confirm the effectiveness of this approach as a method of preliminary modification of the emulsion.

The effect of the Nefras-C280 " hydrocarbon solvent on sample separation and temperature-dependent changes in viscosity was studied (Fig. 14).

To do this, the sample "Large Pond point # 2 (mark +3.3 m)" was mixed with a solvent in a ratio of 1: 1 at

Fig. 12. Sample appearance after centrifugation of the "sample + diesel fuel" mixture

Table 5. Value of dynamic viscosity of the mixture "sample + diesel fuel" in the ratio 1:1

Place of sampling	Dynamic viscosity, MPa • s							
	25 °C	30 °C	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C
BP t. no. 2 (+3.3 m) + Diz. Fuel 1:1	836.0	728.7	616.2	431.9	242.7	167.1	151.4	125

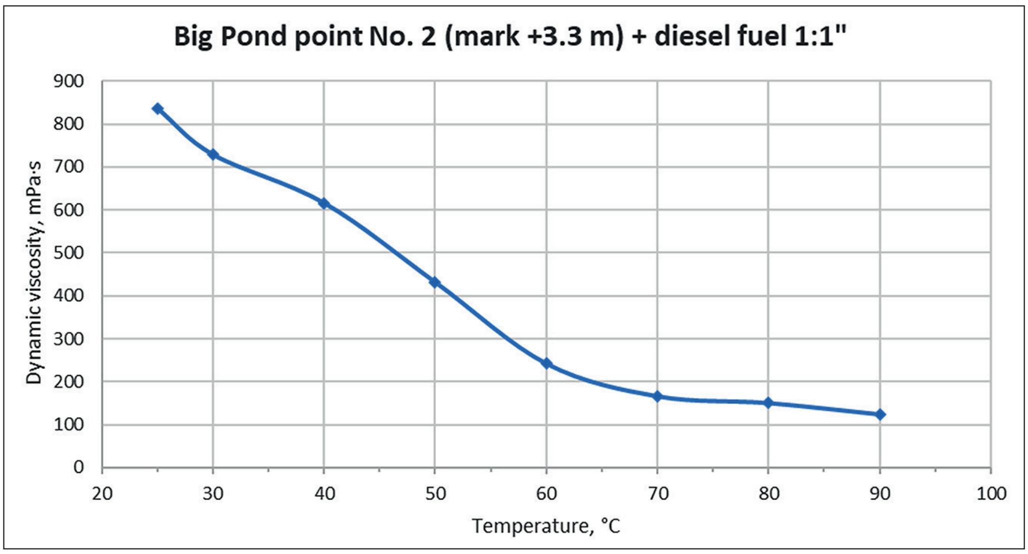

Fig. 13. Dependence of the dynamic viscosity of the sample on the temperature according to Table 5

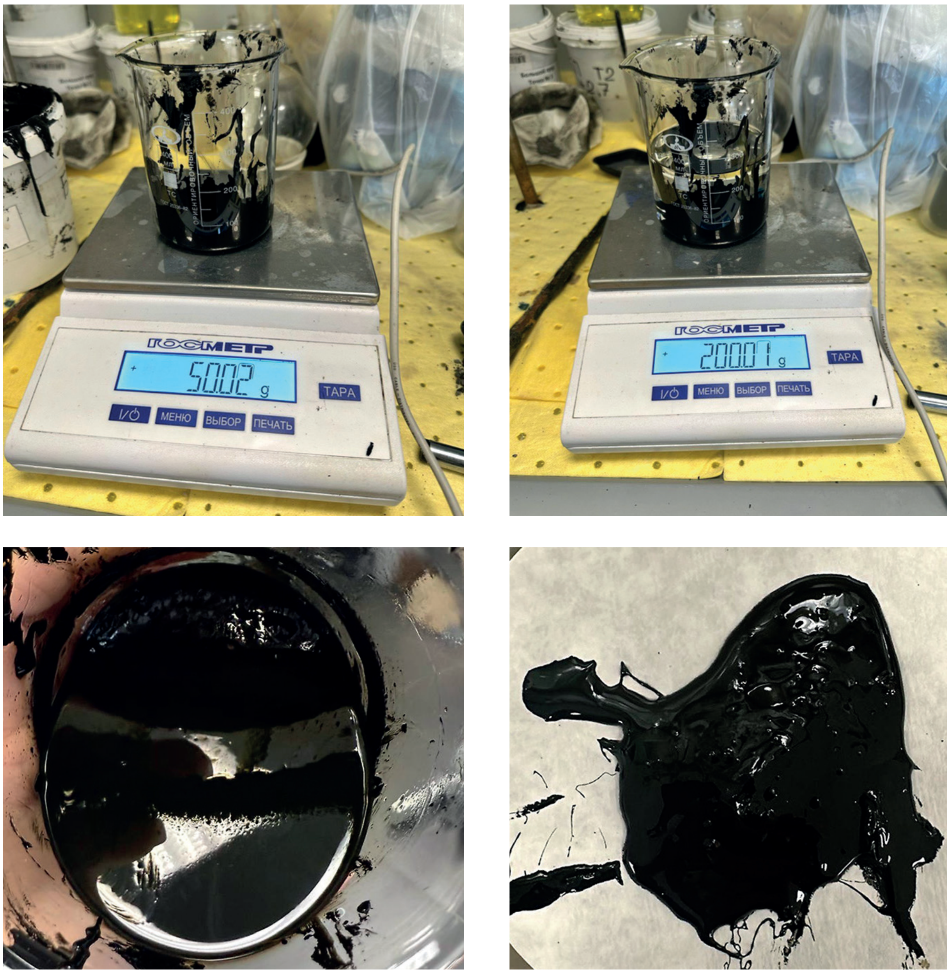

Fig. 14. Sample separation after centrifugation of the sample + Nefras C2 80 1:2 mixture

700 rpm for 30 minutes. Then the upper part of the solvent was drained and further studies were continued on the sample-solvent mixture.

After centrifugation, the sample was divided into two phases: mechanical admixtures and the oil component. Water separation was also not observed.

The obtained dependence of the dynamic viscosity of the mixture "sample + Nefras C2 80 1:1" of the temperature is shown in Fig. 15 and Table 6.

Although Nefras-C2-80 significantly reduces the viscosity compared to the initial sample, **diesel fuel is a more effective agent for pre-preparation of the emulsion for mechanical separation (centrifugation)**, as it provides much lower viscosity values over the entire temperature range. "Nefras" can be useful for other tasks, for example, for extraction of certain fractions, but not for optimal viscosity reduction.

A sample mixed with "Nefras C280" was also examined, followed by the removal of bound water and mechanical impurities. Dynamic viscosity at various temperatures (Fig. 16, Table 7) and densities at 25 °C and 50 °C. were determined.

The use of the solvent "Nefras-C2 80" also led to a significant decrease in viscosity, although less pronounced compared to diesel fuel. The density of the resulting oil phase decreased to 0.908 g/cm^3, which is typical for light oil products. This indicates that the solvent not only reduced the viscosity, but also changed the composition of the continuous oil phase, contributing to more efficient separation from mechanical impurities.

DISCUSSION OF THE RESULTS

The study revealed that the initial sample of contaminated soil is a highly stable emulsion of the "water in oil" type, stabilized by mechanical impurities of sand (SiO_2). High dynamic viscosity and resistance to standard centrifugation, even with the use of a demulgator, are due to the formation of strong interfacial nanolayers, where sand particles act as natural emulsifiers, preventing coalescence of water droplets [11–13].

The key problem is precisely the nanoscale character of stabilization, which is consistent with modern ideas about the role of solid particles in the stabilization of

Table 6. Dynamic viscosity of the mixture "sample + Nefras C280 1:1"

Sample collection point	Dynamic viscosity, MPa • s							
	25 °C	30 °C	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C
BP T. No. 2 (+3.3 m) + Nefras C2 80 1:1	5711.2	4680.4	3741.8	2121.4	1329.1	891.4	848.8	787.4

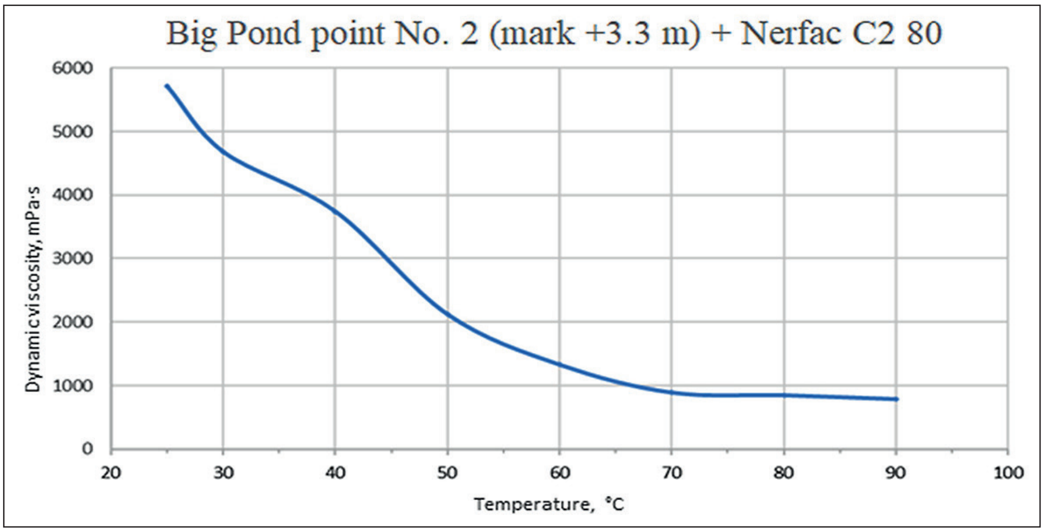

Fig. 15. Dependence of the dynamic viscosity of the mixture " sample + Nefras C2 80 1:1" from the temperature

Table 7. Dynamic viscosity of the mixture "sample + Nefras C280 1:1", which does not contain bound water and mechanical admixtures

Sample location	Dynamic viscosity, MPa • s								Emulsion density, kg/m³	
	25 °C	30 °C	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C	25 °C	50 °C
BP t. no. 2 (+3.3 m) without mechanical admixtures and water	5197	4028	3459	1892	1269	884	836	762	0.908	0.907

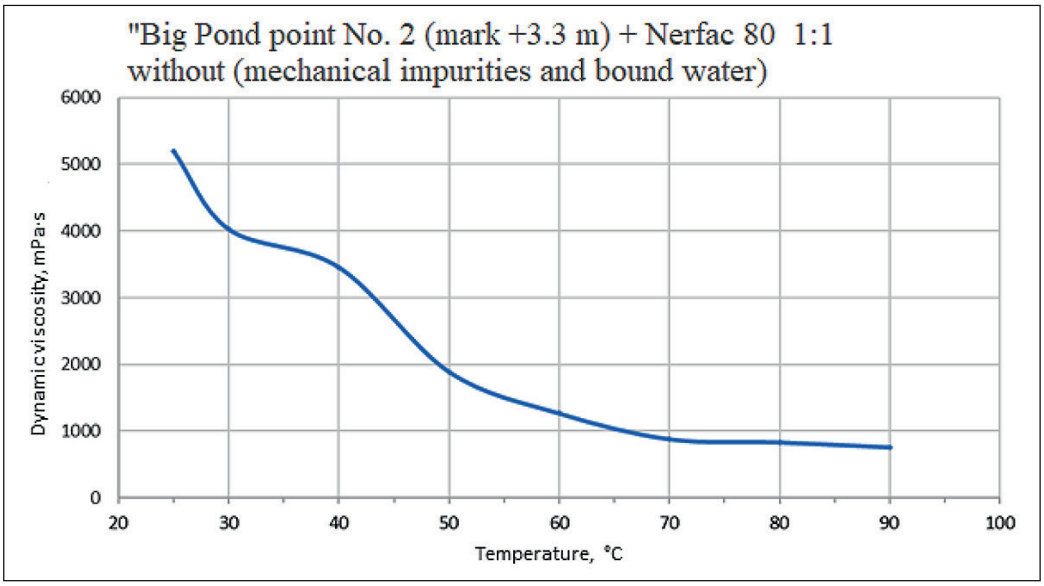

Fig. 16. Dependence of the dynamic viscosity of the mixture "sample + Nefras C2 80 1:1 without mechanical impurities and bound water" on the temperature

emulsions [14, 15]. Traditional demulsification methods, which are mainly aimed at destroying surface-stabilized films, were ineffective in this case without first destroying the entire structure of the system.

The most effective strategy was chemical dilution, which leads to two main effects:

1. Viscosity reduction: a diluent (diesel fuel or solvent) reduces the viscosity of the continuous oil phase, which facilitates sedimentation and kinetics of separation processes under the action of centrifugal forces [16, 17].

2. Violation of interphase stabilization: the introduction of a large volume of hydrocarbon liquid changes the phase ratio, the thickness of interphase layers, and the wettability of solid particles, which leads to destabilization of the emulsion and aggregation of sand particles [18, 19].

Subsequent thermomechanical treatment (heating and centrifugation) completes the separation process, providing sedimentation of coagulated mechanical impurities. The absence of visible separation of the aqueous phase after dilution and centrifugation can be explained by its dispersion in excess of the hydrocarbon phase or the formation of microemulsions.

A comparative analysis of diluents showed that diesel fuel is a more effective agent for reducing viscosity, while Nefras-C280, being a lighter solvent, probably copes better with solubilization of asphaltene-resinous components that contribute to the stability of the emulsion [20, 21].

CONCLUSION

1. It was found that the contaminated soil is a highly viscous (105379 MPa • s at 25 °C) and highly stable emulsion containing 33% water and 23.8% mechanical impurities (sand, SiO_2).

2. The ineffectiveness of standard demulsification methods (centrifugation, including with a demulger) for separating the initial emulsion is shown, which is associated with nanoscale stabilization of interfacial layers by solid particles.

3. An efficient two-stage emulsion separation technique has been developed, including chemical dilution with diesel fuel or Nefras-C280 solvent in a 1:1 ratio, followed by heating and centrifugation.

4. It has been proven that dilution with diesel fuel is the most effective way to reduce the viscosity (by a factor of 126 at 25 °C), which makes the system suitable for successful mechanical separation.

5. The obtained data are of practical significance for the development of formulations and technologies for the elimination of complex oil and sand pollution in coastal zones.

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ADDITIONAL INFORMATION

The authors state that generative artificial intelligence technologies and technologies based on artificial intelligence were not used in the preparation of the article.

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A. Y. Abusal Yusef – writing the original text; analysis and elaboration of the research methodology.

A. R. Yakhin – scientific text editing; research concept.

D. V. Silnov – scientific guidance, formulation of final conclusions.

D. I. Araslanova – analysis of literary sources and previous research; design of text material in accordance with the requirements of the journal.

V. A. Gorshkov – analysis of literary sources and scientific research of Russian and international scientists; text design.

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