



Reduction of Optical Density in Highly Viscous Oils through Ultrasonic Treatment within The Infrared Wavelength Range

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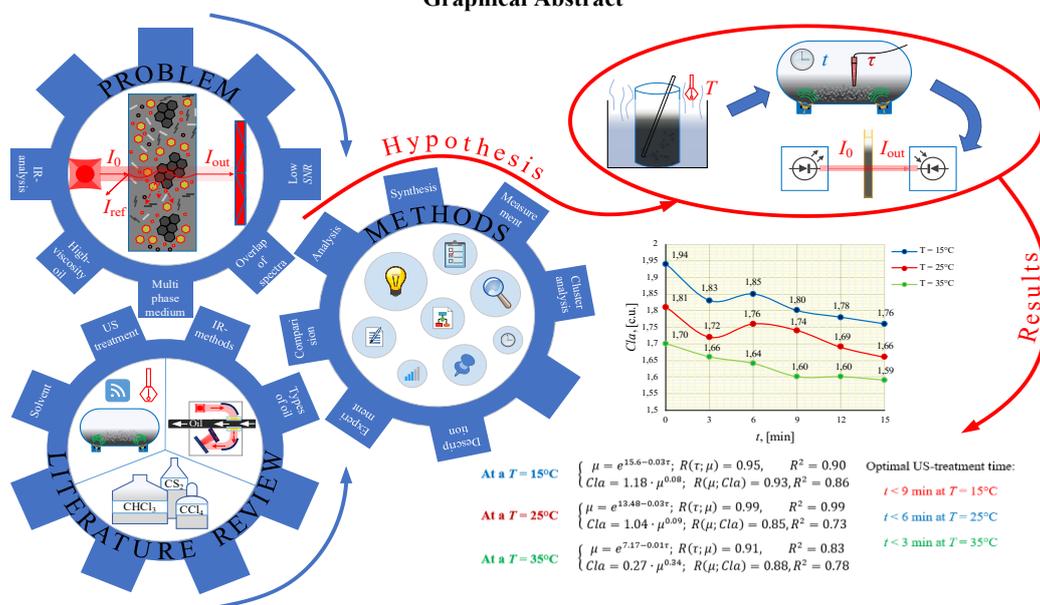
Sample Preparation

ABSTRACT

Today, Russia and the rest of the world are experiencing an increase in the production and reserves of heavy hydrocarbons (HC). This poses a problem in terms of both technical and financial needs, compared to conventional oil reserves. In this context, one of the key objectives for the advancement of the mineral resources sector is the enhancement of oil field diagnostic systems. This enables the "digitalization" of oil fields and processes related to extraction and transportation. The aim of the work is to analyze the current state of infrared (IR) oil diagnostic devices, existing problems and ways to optimize them, with special attention to the preliminary ultrasonic (US) treatment of highly viscous samples. This study was able to confirm the hypothesis that US treatment of resinous oil samples leads to an increase in the signal-to-noise ratio (SNR) of the IR diagnostic system. The highest efficiency of US treatment was recorded at a point (15°C; 15 min), where the increment of I_{out} was 51.36 % relative to untreated oil. The optimal time for US treatment based on effect on chemical composition of oil is: $t < 9$ min at $T = 15^\circ\text{C}$, $t < 6$ min at $T = 25^\circ\text{C}$, and $t < 3$ min at $T = 35^\circ\text{C}$. Despite positive results, authors emphasize problems with uneven distribution of US vibrations and need for further research on paraffinic oil.

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Graphical Abstract



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| NOMENCLATURE | | | |
|----------------|--|----------------------|---|
| C | Concentration (mol/l) | R^2 | Coefficient of determination |
| Cl_a | Optical density (c.u.) | SNR | Signal-to-noise ratio (dB) |
| E | Sensitivity of the photodetector (A/W) | T | Initial oil temperature (°C) |
| f | Frequency (Hz) | t | Ultrasonic treatment time (min) |
| I_0 | Initial IR-radiation intensity (W/cm ²) | V | Volume of oil (ml) |
| I_{abs} | Intensity of absorbed radiation (W/cm ²) | W_N | Photodetector's own noise power (W) |
| I_{out} | Intensity of the IR-radiation transmitted through the sample (W/cm ²) | W_{out} | The power of the incident IR radiation on the photodetector (W) |
| I_{ref} | Intensity of reflected radiation (W/cm ²) | Greek Symbols | |
| I_{scr} | Intensity of scattered radiation (W/cm ²) | ε | Molar extinction coefficient ($\frac{ml}{mol \cdot cm}$) |
| I_{t_0} | IR-radiation intensity of the sample without ultrasonic preparation (W/cm ²) | λ | Wavelength (nm) |
| l | Optical path length (mm) | μ | Dynamic viscosity (mPa·s) |
| m | Weight (g) | ρ | Density (g/cm ³) |
| P_{IR} | Radiation power of the laser diode (W) | σ | Measurement error according to device specification (%) |
| P_{US} | Ultrasound power (W) | τ | Oil temperature after ultrasonic exposure (K) |
| $R(\mu; Cl_a)$ | Correlation coefficient between μ and Cl_a | ΔI | IR-intensity increment (W/cm ²) |
| $R(\tau; \mu)$ | Correlation coefficient between τ and μ | | |

1. INTRODUCTION

Fossil fuels, despite significant investment by major companies in renewable energy sources, continue to be a significant source of energy demand. In light of the current global energy crisis and depletion of conventional oil reserves, mineral resources are increasingly dependent on alternative sources such as heavy oil and bitumen (1, 2). By 2030, production from these sources is expected to increase by nearly five times (3). The extraction and transport of heavy oil presents several challenges compared to conventional oil (4).

In the current climate, one of the key objectives is to enhance the profitability of extracting hard-to-access HC reserves through the use of advanced technologies. These new technologies involve creating "intelligent" and "digitalized" fields (5), which provide continuous and reliable monitoring of the physical and chemical properties of oil and the processes involved at various stages of the raw material's lifecycle. The main goal of these solutions is to optimize production Procedures that would enable: obtaining and comprehending data about reservoir performance; boosting oil extraction rates through the judicious selection of chemicals and stimulation techniques; selecting the most effective method for oil transportation, taking into consideration its characteristics; monitoring the environmental situation in oil-producing areas and creating predictive models for potential environmental incidents (6-8).

Currently, innovative techniques such as steam injection, thermal treatment (9), the use of chemical additives (10), emulsification, and hydrotransport are being actively implemented to enhance the efficiency of producing and transporting high-viscosity oils (11).

These methods have the potential to significantly increase HC recovery rates and reduce the costs associated with transportation. Despite the progress made in this area, many existing techniques still fall short of their full potential and require further investigation, optimization, and intellectualization (12).

A crucial step in the process of intellectualizing deposits is the development of sensors to monitor the physico-chemical characteristics of HC. These sensors should allow for the assessment of the quality of raw materials, as well as the determination of parameters for oil production and transportation. For example, high concentrations of paraffins, sulfur-containing and organochloride compounds can lead to corrosion and potential accidents. To mitigate the negative effects of these impurities, various depressant additives and demulsifying agents are employed (13). These additives reduce the viscosity of oil, preventing the formation of paraffin deposits and cleaning pipes of tar-paraffin buildup, thereby increasing their throughput and reliability (14). The choice of additive composition and type depends on factors such as the concentration of resins, asphaltenes, paraffins and aromatic compounds, water content, initial viscosity, temperature and pH.

To determine the physical and chemical properties of oil, samples must be taken and transported to specialized laboratories. However, the results can be affected by the adsorption of surface-active substances and evaporation of lighter fractions, depending on the length of transportation. One of the modern and promising approaches to solving this issue is the use of oil diagnostic devices operating IR wavelength range. This range offers a harmonious balance between the ability to penetrate and sensitivity to oil composition. (15, 16).

Additionally, a significant benefit is the lack of need for signal processing systems in close proximity to IR sensor, as a trunking or fiber optic communication system can be used to connect them (17). Major developments in this area of technology include:

1. Raupov et al. (18) conducted investigations into the optical characteristics of oil, including optical density, absorption coefficient, and refractive index, across IR, visible (V), and ultraviolet (UV) spectrums. Their study presents a significant number of advanced designs for measuring oil optical properties directly at the wellhead. Additionally, Raupov et al. (18) experimentally established that oil optical properties correlate with density, viscosity, and surface tension. Based on their findings, the devices utilize tunneled lasers that provide a wide range of optical radiation but have several drawbacks, including a tendency to “jump” between wavelengths, and the need for high-intensity sources for efficient conversion. Many existing optoelectronic detectors either cannot operate in both UV and IR spectral regions simultaneously (19, 20), or exhibit partial overlap of these regions, such as in the case of PCF-SPR (elliptical plasmon) sensors (21). In addition, the study does not provide information on the sample holder thickness, which is crucial when analyzing high viscosity oil.

2. Santos et al. (22) developed a portable IR spectrometer for measuring oil density and chemical composition, including sulfur, nitrogen, asphaltenes, and various HC, in the wavenumber range of 11000–6000 cm^{-1} . The device was tested on 182 samples of oil, but the results revealed that the spectrometer’s coefficient of determination exhibited low efficiency ($R^2 < 0.7$) for determining resins, saturated, and aromatic HC. This suggests difficulties in analyzing viscous oils.

3. Xu et al. (23) proposed using algorithms with deep learning and an increased Mahalanobis distance to reduce the number of outliers and the influence of noise on measurement results. They also proposed the use of wavelet transformations combined with the T2-Hotelling test to achieve this. Yang et al. (24) achieved an improvement in the performance of mathematical apparatus for processing large data streams by combining the principal component analysis/autoencoder method with support vector regression. Despite a significant number of publications on improving approaches to data processing and spectrogram analysis (25, 26), it is also essential to pay attention to the physical aspects of the interaction between IR radiation and dispersed petroleum media. It is impossible to eliminate all random noise or remove overlapping peaks through mathematical transformations alone, so it is necessary to consider the physical properties of the medium. In this instance, a comprehensive approach is required, which encompasses not only data analysis, but also the appropriate preprocessing of the oil sample.

The task of determining the physicochemical properties, in particular, the composition, of high-viscosity oils is challenging due to their multiphase nature, heterogeneity, and saturation. IR devices used in the process of diagnosing heavy oils encounter a low signal output, which results in “blurring” of peaks on the spectrogram and darkening of other extrema (24, 27, 28). This issue has traditionally been addressed by utilizing various solvents and heat. Solvents effectively enhance the optical clarity of the oil (29), but their use precludes the automation of oil diagnostics processes at the wellhead, due to the need for empirical selection and disposal of chemical by-products. Heat likewise contributes to enhancing optical clarity in oil (30). However, when any issues regarding high-viscosity oils arise, it has been found that heating only destroys formed asphalt-resin-paraffin deposits when a specific melting point has been reached. A more modern alternative to use solvents and heat is the US treatment, which has three simultaneous effects: acoustic cavitation, mechanical heating, and degassing. The US action, due to the propagation of mechanical waves and the collapse of bubbles, contributes to the disruption of agglomerates and a more uniform distribution of the various components (31, 32). The effect of the disruption of US agglomerates can be observed prior to their melting point, which, in theory, would increase the optical clarity of the oil medium. Besides, US baths are more compact and versatile than solvent tanks and, therefore, can be installed directly at the wellhead. This would allow for the analysis of heavy oil in the field. Therefore, the investigation into the feasibility of using US treatment of heavy oil samples to address the issue of high absorption of IR frequency range optical signal in a dispersed petroleum medium should be considered a pressing and novel task.

2. LITERATURE REVIEW

One of the significant stages of the study is to identify a viable approach for performing IR diagnostics on oil and obtaining answers to several questions. Which technique will be more easily automated in the future? Which technique is more dependable and simpler to implement? What are the constraints of this technique?

2. 1. Methods of IR Diagnostics of Oil IR diagnostics of oil allows qualitative and quantitative study of the physicochemical characteristics of oil, including its composition, optical and rheological properties. There are four main methods for IR diagnostics (33-35). The implementation schemes and comparative analysis are presented in Figure 1 and Table 1, respectively.

TABLE 1. Comparison of IR diagnostic techniques for oil

| Technique name | Advantages | Disadvantages | A source |
|---|---|---|--------------|
| The method of disturbed total internal reflection | The ability to analyze oil with ultra-high viscosity; The capacity to analyze samples that contain water; Minimum sample preparation. | The need to determine the appropriate sample thickness; Significant energy loss; Fragile fiber optic construction. | (35-37) |
| The method of transmission spectroscopy | Ease of implementation; A comprehensive understanding of the functional components of oil; The ability to quantitatively analyze. | The complexity of analyzing samples with high levels of turbidity; Limited depth of penetration; Preparation of the sample is required. | (18, 38, 39) |
| The method of vapor phase spectroscopy | High signal-to-noise ratio; High sensitivity. | The need for stable operating conditions; Insufficient understanding of the oil composition (only light fractions are present). | (40) |
| The method of Raman spectroscopy | The ability to work with low oil concentrations; High spatial resolution; No need for sample preparation. | The need to minimize fluorescence; The challenges of working with viscous oils; The method is difficult to implement. | (41-43) |

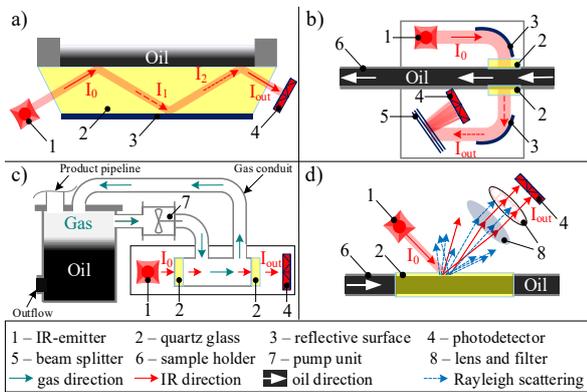


Figure 1. The scheme of implementation of IR diagnostics of oil: a) the method of disturbed total internal reflection; b) the method of transmission spectroscopy; c) the method of vapor phase spectroscopy; d) the method of Raman spectroscopy

Considering the potential use of IR spectroscopy in field applications with the possibility of automation, the transmission spectroscopy method is the most suitable approach. Table 1 illustrates that the transmission spectroscopic method is straightforward to implement and offers a comprehensive insight into functional groups. Nevertheless, the major obstacle is the requirement for pre-treatment of heavy oil samples. Next, we need to answer a number of questions: what prevents the transmission of IR beams? What is the mechanism for sample preparation? Will US treatment meet this requirement?

2. 2. The interaction of the IR beam with a highly viscous oil medium

It is known that only a part of the energy of the incident radiation passes through any substance (see Figure 2) (44).

The processes depicted in Figure 2 can then be described by Equation 1.

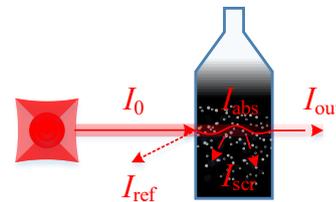


Figure 2. The processes that occur when IR radiation passes through a sample of oil

$$I_0 = I_{out} + I_{ref} + I_{scr} + I_{abs} \quad (1)$$

As can be seen in Figure 2 and Equation 1, the primary sources of loss are due to reflection, scattering, and absorption. A quantitative assessment of these losses is carried out in accordance with the Bouguer-Lambert-Beer Law (Equation 2) (18).

$$Cl\alpha = \lg \frac{I_0}{I_{out}} = \varepsilon \cdot C \cdot l. \quad (2)$$

In order to better understand the processes involved, it is necessary to examine the microscopic aspects of the formation of polymolecular layers and their interaction with IR radiation.

Microscopic examination of the polymolecular layers that form as a result of the interaction between asphaltenes, heavy aromatic HC, and paraffins has revealed that these layers possess a complex structure. The asphaltenes, which contain both aromatic and aliphatic groups, contribute to the formation of stable aggregates (45). Their polar groups are able to form hydrogen bonds and interact via London dispersion forces or Van der Waals bonding, leading to the creation of layers capable of retaining water molecules and other polar compounds (46-48). In addition, molecules that exhibit stronger Van der Waals interactions typically have higher boiling and melting points due to the additional energy required to overcome these attractive

forces (49). Heavy aromatic HC are non-polar and characterized by a high degree of conjugation. Resins are characterized by the presence of both polar and non-polar regions, and therefore act as a bonding agent for connecting non-polar HC molecules with highly polar asphaltenes (50). This interaction results in the formation of denser structures with increased mechanical strength (Figures 3a and 3b). The higher concentration of paraffins is evident in the increased opacity of the oil, as well as the accumulation of paraffin crystals (Figure 3c). Polymolecular layers may extend to tens of micrometers in thickness, which exceeds the wavelengths of the near- and mid-IR spectrum and results in significant energy losses during optical signal transmission (51).

High signal energy losses lead to low SNR values, blurred extremes, and spectrum distortion in spectrograms (24, 27). In this regard, the use of the Bouguer-Lambert-Baer law seems insufficient for the analysis of optical density, since it assumes a homogeneous medium with a linear relationship between absorption and concentration of matter. In practical scenarios, especially when IR radiation encounters highly viscous oil, the heterogeneous and multiphase nature of the oil environment leads to significant deviations from this principle.

Hence, the interaction between IR radiation and viscous oil is a sophisticated, multifaceted phenomenon. Various factors influencing the optical properties of oil are organized into a cluster (see Figure 4).

Based on Figure 4, it can be deduced that oil is an object with a composition of X at a temperature of Y and a pressure of Z, whose constituents exist in different physical states, but interact in an integrated manner. The correlation between chemical composition and rheological characteristics indicates that the composition of oil influences its density and viscosity, which in turn are linked to optical properties. To enhance the effectiveness of interaction between IR radiation and oil, particularly to increase its penetration depth, it is essential to explore options for reducing impurities and viscosity in the oil, as well as to strive for a uniform and single-phase medium.

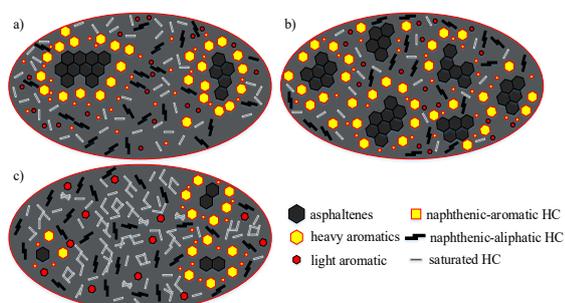


Figure 3. The distribution of components in heavy, dehydrated, and degassed oils can be classified into the following categories: a) sol type; b) gel type; c) paraffinic

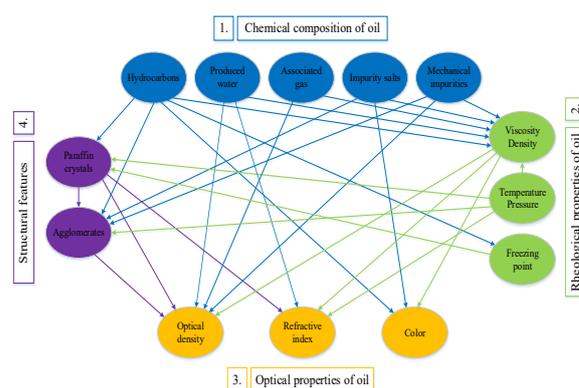


Figure 4. Factors affecting the optical properties of oil

2. 3. Preparation of Oil Samples for IR Analysis

In order to obtain accurate results in IR spectroscopy, it is necessary to carry out preliminary sample preparation to ensure the required uniformity of the oil (30). This preparation process involves removing unnecessary phases and different states of components, reducing the number of objects that absorb and scatter radiation. The process may include filtration to remove mechanical impurities, separation of water, degassing, and methods to reduce viscosity.

Filtration is performed using either mesh or advanced composite coalescing filters (52), while dewatering is achieved through membrane separation (53) or the use of demulsifiers (54). The main sample preparation process involves reducing optical density and turbidity, traditionally accomplished by the addition of solvents, or more rarely by thermal methods such as microwaving and heating (30). Table 2 lists some of the most commonly used solvents along with their relevant parameters.

The solvents, in their pure form, do not interact with the metals employed in the construction of the equipment, such as steel. The outcomes of chemical reactions between solvents and oil constituents, particularly alkanes, can be hazardous. These reactions result in the formation of chlorinated HC, hydrochloric acid and thiol compounds with corrosive properties (55, 56). While single measurements may not cause harm, repeated or systematic use can lead to decreased reliability and durability of the diagnostic system. Additionally, there are several other disadvantages associated with this process: the use of solvents requires

TABLE 1. Oil solvents

| Solvent, [Formula] | Proper bands, [cm ⁻¹] | Hazard class, GOST 12.1.007-76 | A source |
|--------------------|-----------------------------------|--------------------------------|----------|
| CCl ₄ | 730-825 | II | (57) |
| CHCl ₃ | 720-810 | II | (57, 58) |
| CS ₂ | 2178.99-2180.79 | II | (59) |

an additional storage tank for the reagent; the optimal solvent proportion must be determined experimentally; disposal of reaction products must be addressed (29, 30).

A more environmentally sustainable alternative to solvents for oil sample preparation is US technology. This method does not generate any chemical waste that needs to be disposed. Additionally, issues with high noise levels and energy inefficiency commonly associated with US equipment can be addressed by reducing the operating time, lowering the generator power, or increasing the frequency. Despite extensive research on this method, the US treatment has not yet implemented it widely in the oil production and transportation stages due to the lack of powerful generators that can meet the specific needs of these processes. However, in conditions with small volumes of oil, the US treatment is a promising method for preparing samples for IR analysis.

When high-viscosity oil is subjected to US treatment, several physical processes occur. Firstly, US induces the phenomenon of acoustic cavitation, resulting in the formation of microbubbles. As these microbubbles collapse, localized high temperatures and pressures are generated, which contribute to the disruption of Van der Waals forces between molecules (60). Secondly, US treatment has a mechanical and thermal effect on the liquid, reducing its viscosity and breaking down aggregates and interactions between components such as

asphaltenes, paraffins, aromatics and resins. This makes the oil more uniform and evenly distributes its constituents (31, 32). This property minimizes deviations from the Bouguer–Lambert–Baer law. Thirdly, US treatment helps to degas oil (61). Despite the benefits, there are also several drawbacks, which are manifested in the individual physical response of oil to US, which depends on its chemical composition, viscosity, and temperature. Table 3 provides a summary of scientific studies on the impact of US on oil viscosity, as this parameter is correlated with optical density (18). The review articles described the composition of the oil, its physical properties, and a detailed methodology for the experiment.

The main conclusions from Table 3 are as follows:

1. The most studied frequency range for US effects on oil viscosity is from 18 kHz to 28 kHz. Frequencies above this range are less studied. Using a 42 kHz frequency has demonstrated positive results in reducing the viscosity of low-sulfur resinous oils.

2. The results of experiments using paraffinic ($\geq 1.5\%$) and sulfurous oil (62) were extremely contradictory.

3. The review indicates the absence of a unified metrological approach to US treatment, requiring further clarification during experimental studies.

TABLE 2. Review of existing approaches to US treatment of high-viscosity oil

| Oil composition/ The ratio of the elements | Ultrasound/Oil parameters | Results | A source |
|---|---|---|-------------|
| Resins – 32.1%, Asphaltenes – 8.2%, Paraffins (par) – 2.2%. | $f = 22 \text{ kHz}, t = 20 \text{ mins}, P_{US} = 2 \text{ kW}/$ $T = 20^\circ\text{C}, V = 200\text{-}300 \text{ ml}, \mu_0 = 2400 \text{ mPa}\cdot\text{s}$ | $\mu_{20\text{mins}} = 0.75\mu_0$ | (63) |
| C:H:O:N:S - 205:24:1:1:5. | $f = 18 \text{ kHz}, t = 6; 12 \text{ mins}, P_{US} = 0.25 \text{ kW}/$ $T = 65^\circ\text{C}, V = 60 \text{ ml}, \mu_0 = 625 \text{ mPa}\cdot\text{s}$ | $\mu_{6\text{mins}} = 0.8\mu_0, \mu_{12\text{mins}} > \mu_0$ | (60) |
| C:H:O:N:S - 210:28:3:8:1, Resins – 32.5%, Aromatic – 24.3%. | $f = 42.1 \text{ kHz}, t = 3 \text{ mins}, P_{US} = 0.15; 0.4; 0.8 \text{ W/ml}/$ $T = 41^\circ\text{C}, V = 100 \text{ ml}, \mu_0 = 661.2 \text{ mPa}\cdot\text{s}$ | $\mu_{0.15\text{W}} = 0.798\mu_0, \mu_{0.4\text{W}} = 0.47\mu_0,$ $\mu_{0.8\text{W}} = 0.18\mu_0$ | (31) |
| Resins – 47.82%, Asphaltenes – 7.39%, Paraffins – 0; 0.5; 1; 1.5%. | $f = 22 \text{ kHz}, t = 2 \text{ mins}, P_{US} = 15 \text{ W/cm}^2/$ $T = 20^\circ\text{C}, m = 50 \text{ g}, \mu_0 = 246.9 \text{ mPa}\cdot\text{s}$ | $\mu_{0\%par} = 0.8\mu_0, \mu_{0.5\%par} = 1.1\mu_0,$ $\mu_{1\%par} = 1.4\mu_0, \mu_{1.5\%par} = 1.6\mu_0$ | (64) |
| Resins – 22.65%, Asphaltenes – 8.15%, Alkanes – 39.75%, Aromatic – 28.55%. | $f = 28 \text{ kHz}, t = 30 \text{ mins}, P_{US} = 105 \text{ W}/$ $T = 50^\circ\text{C}, m = 30 \text{ g}, \mu_0 = 1015 \text{ mPa}\cdot\text{s}$ | $\mu_{30\text{mins}} = 0.56\mu_0$ | (65) |
| Resins – 97.80%, of which silica gel – 2.2%, Alkanes – 9.80%. | $f = 22 \text{ kHz}, t = 10 \text{ mins}, P_{US} = 8 \text{ W/cm}^2/$ $T = 22^\circ\text{C}, m = 50 \text{ g}, \mu_0 = 253 \text{ mPa}\cdot\text{s}$ | $\mu_{10\text{mins}} = 0.763\mu_0$ | (66) |
| Resins – 6.48%, Asphaltenes – 2.85%, Paraffins – 1.67%, Water – 58%. | $f = 22 \text{ kHz}, t = 5 \text{ mins}, P_{US} = 1.5 \text{ kW}/$ $T = 22^\circ\text{C}, V = 600 \text{ ml}, \mu_0 = 465 \text{ mPa}\cdot\text{s}$ | $\mu_{5\text{mins}} = 3.3\mu_0$ | (67) |
| Sulfur – 4.68%, Asphaltenes – 6.379%. | $f = 25 \text{ kHz}, t = 2 \text{ mins}, P_{US} = 360 \text{ W}/$ $T = 25^\circ\text{C}, V = 250 \text{ ml}, \mu_0 = 426 \text{ mPa}\cdot\text{s}$ | $\mu_{2\text{mins}} = 0.857\mu_0$ | (68) |

4. US treatment may affect not only the physical properties of oil but also its chemical composition (31, 32). Therefore, it is necessary to establish boundary conditions for US treatment in order to ensure accurate results.

3. METHODOLOGY AND MATERIALS

Experimental studies were carried out in the laboratory of the Department of Electronic Systems at Empress Catherine II Saint-Petersburg Mining University. The main techniques used in the course of the study include US processing and transmission IR diagnostics. Prior to conducting the experiments, the oil was subjected to initial preparation to remove large mechanical particles using a mesh filter. No degassing of the oil was performed.

The selection of 35-45 kHz US bath frequency range is based on the high efficacy of these frequencies for working with resinous oils (see Table 3). Additionally, according to our review, this frequency range has not been extensively studied, therefore, further investigation is required.

The selection of temperatures for testing heavy oil samples was determined by the following considerations. The lower temperature limit of 15°C was chosen due to the ability to draw oil using a syringe and fill a thin cuvette. The upper limit of 35°C is set slightly below the boiling point (40°C) of the petroleum fraction of the oil in order to ensure adequate conditions for analysis.

The key characteristics of the oil sample under analysis and the equipment used in the experiment are outlined in Table 4. In addition to the materials listed, a thermometer was used to monitor the heating temperature (τ), a syringe was used for filling the cuvette, and a water bath was employed to stabilize the temperature of the samples.

The research was conducted as a two-factor, four-stage experiment (see Figure 5). The measurements were conducted in accordance with the "three-point" method principle, that is, the experiment was replicated three times. It was carried out under the following conditions: ambient temperature (+20±1)°C, air humidity (60±0.2)%, and power supply voltage (220±5)V.

A total of 3 samples of high-viscosity oil, each with a volume of 250 ml, were used in each cycle of the experiment. At the first stage, the oil sample was stabilized to temperature T in a water bath. In the second stage, heated oil was placed in an US bath, where it was subjected to US. After the treatment time t_n ($n = 0, 1, \dots, 5$) had elapsed, a 1 ml sample was removed from the bath and placed in a quartz cuvette. The maximum value of t was selected based on parameters and limitations of the US bath. Oil placed in the cuvette was

TABLE 3. Materials and equipment for the experiment

| Material/equipment | Composition/main element | Specifications |
|------------------------|---|--|
| Oil (resinous) | Resins – 22.21%; alkanes – 33.91%; aromatics – 32.62%; asphaltenes – 0.58% | $T = 15^\circ\text{C}$; $\mu_0 = 712.4 \text{ mPa}\cdot\text{s}$; $\rho = 0.961 \text{ g/cm}^3$; $V = 2.25 \text{ l}$ |
| US bath | – | $f = 35 \dots 45 \text{ kHz}$; $P_{\text{US}} = 30 \text{ W}$; $V_{\text{max}} = 500 \text{ ml}$ |
| Cuvette | Quartz | $\lambda_{\text{min}} \dots \lambda_{\text{max}} = 200 \dots 2500 \text{ nm}$; $l = 0.5 \text{ mm}$ |
| Viscometer | – | $\mu_{\text{min}} \dots \mu_{\text{max}} = 0.1 \dots 10 \cdot 10^6 \text{ mPa}\cdot\text{s}$; $\sigma = \pm 1.0 \%$; $T_{\text{min}} \dots T_{\text{max}} = -60 \dots +360^\circ\text{C}$ |
| Source of IR radiation | Laser diode | $\lambda = 1540 \text{ nm}$; $I_0 = 5.1 \text{ W/cm}^2$; $P_{\text{IR}} = 40 \text{ mW}$ |
| IR sensor | Photodiode | $\lambda_{\text{min}} \dots \lambda_{\text{max}} = 900 \dots 1700 \text{ nm}$; $E_{\text{min}} = 0.95 \text{ A/W}$ |

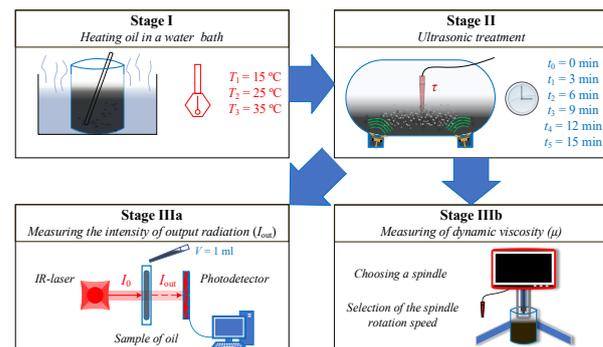


Figure 5. The scheme of the experiment (one cycle)

positioned between an IR radiation source and photodetector that recorded I_{out} . Photodetector were coaxial with emitting IR laser at a distance no more than 3 cm apart. Measuring unit was protected from sunlight by wooden board covered in black cloth.

The temperature τ was measured at time t_n .

The dynamic viscosity was determined using a rotational viscometer equipped with a thermometer. The principle of operation of a rotary viscosimeter is based on measuring the force required to maintain a given rotational speed of the rotor element (10). The higher the viscosity of the fluid, the greater the force needed to maintain the same rotational speed. The spindle and torque are selected empirically.

Cl_a of the oil sample was determined using the Bouguer-Lambert-Beer law (Equation 1). The assessment of the effect of US treatment of high-viscosity oil on SNR was carried out according to Equation 3 (69).

$$SNR = 10 \lg \frac{W_{out}}{W_N} \quad (3)$$

Considering that in Equation 3: $W_{out} = I_{out} \cdot S$ (70), where W_N and S are constant values, since the equipment and experimental conditions have not changed, it is most advisable to evaluate the effectiveness of the effect of US treatment on SNR by calculating ΔI (Equation 4).

$$\Delta I = \frac{I_{out} - I_{t_0}}{I_{t_0}} \cdot 100\% \quad (4)$$

4. RESULTS AND DISCUSSION

The measurement results, μ , I_{out} , I_{t_0} , τ , represent the average of three measurements. No emissions were observed during these measurements.

Figure 6a illustrates the downward dynamics of changes in μ with increasing processing time. The data obtained are consistent with available studies (31, 63), which confirms the reproducibility of results on reducing viscosity of resinous oils.

Analysis of Figure 6b revealed a clear trend of Cl_a decreasing with increasing t in all three experiments, conducted at different initial oil sample temperatures. Let us consider each curve individually:

Blue curve: In the time interval of $t = 0 \dots 3$ mins, an initial decrease in Cl_a was observed, which could be interpreted as the result of cavitation processes that lead to the destruction of asphalt-resin-paraffin aggregates. This corresponds with the active effect of US on the oil

sample. During the time period of $t = 3 \dots 6$ mins, Cl_a increased, indicating a probable redistribution of colloidal particles and their aggregation into larger structures, as well as the separation of the emulsion (resins and other components) into distinct phases (differences are visible in Figures 7a and 7b). This extreme can also be attributed to an uneven distribution of US vibrations within the bath, leading to incomplete sample treatment and the partial accumulation of heterogeneities at specific points (71-73). This hypothesis seems more plausible, given that μ continues to decline within the range of $t = 3 \dots 6$ mins (Figure 6a). During the time period $t = 6 \dots 15$ mins, there is a decrease in Cl_a , which can be interpreted as the active process of disintegration of newly formed aggregates. This suggests that cavitation effects remain active, and the process of particle dispersal and disintegration continues.

Red curve: In the time interval of $t = 0 \dots 3$ mins, similar to the blue curve, there is an initial decrease in Cl_a , indicating active cavitation processes. During the period of $t = 3 \dots 6$ mins, a sharp increase in Cl_a is observed, although this phenomenon is more pronounced compared to the blue curve. In addition to the aforementioned explanation of this phenomenon, it is important to note Figure 7c, which clearly shows significantly more "traces" of collapsed microbubbles caused by acoustic cavitation on the right side of the US bath compared to the left side, visually confirming the assumption of uneven distribution of vibrations. During the interval of $t = 6 \dots 15$ mins, the particle destruction process remains active, with Cl_a decreasing.

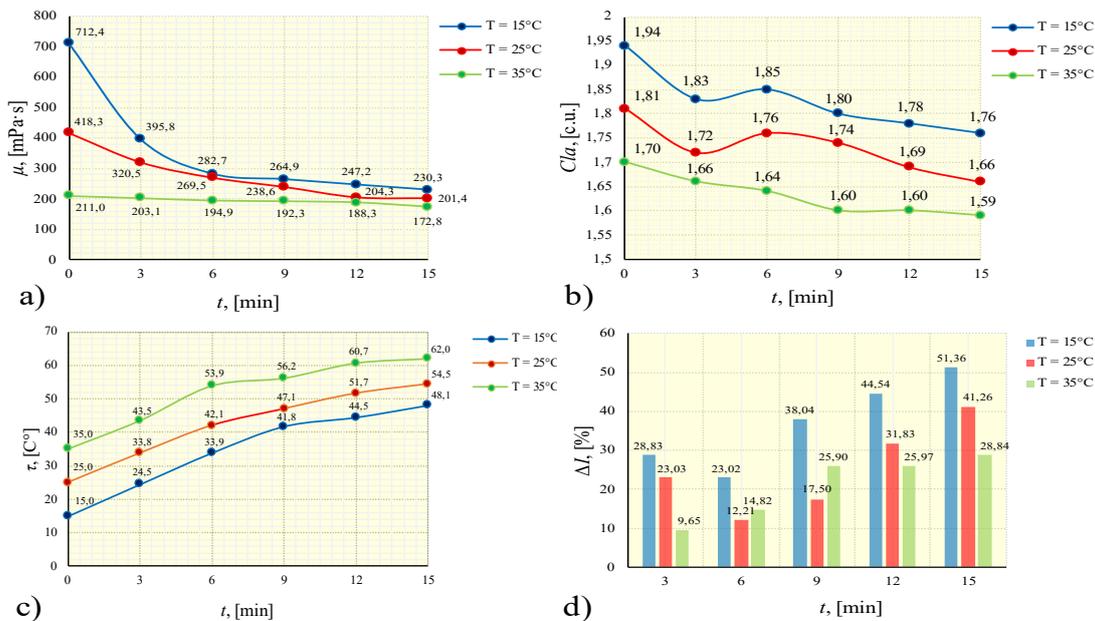


Figure 1. a) Curves of changes in μ from the time of US treatment; b) Curves of changes in Cl_a from the time of US treatment; c) Curves of change in τ from the time of US treatment; d) Dependence of ΔI on the US treatment time

The green curve's low volatility and lack of extreme values at time $t = 0 \dots 9$ mins can be explained by the adequate energy level required to disrupt interactions between particles, which is linked to a higher temperature T . The higher temperature of the sample weakens intermolecular bonds (van der Waals forces), preventing particle recombination and the formation of stable aggregates. The minimal change in the Cl_a value on the green curve between $t = 9 \dots 15$ mins indicates an almost completed process of destabilization of inhomogeneities. At the same time, the blue and red curves in the interval $t = 9 \dots 15$ mins show the active phase of this process.

The overall decrease in the concentration of Cl_a relative to the initial measurement ($T = 15^\circ\text{C}$; $t = 0$ mins) and the final measurement ($T = 35^\circ\text{C}$; $t = 15$ mins) was 0.35 c.u. or -18.04%.

Figure 6c shows that the increment of τ decreases with increasing t . This phenomenon can be explained by the fact that, as the oil's temperature increases, its thermal capacity also increases, and consequently, the amount of energy needed to raise the temperature varies. It is crucial to note that heating can affect the chemical properties of oil. In this instance, it is essential to take into account the onset of evaporation of the petroleum (light) fraction, which occurs at approximately 40°C and above. Figure

7b shows the formation of bubbles, indicating the beginning of light oil fraction evaporation. The oil's temperature at this point was 42.1°C . Therefore, the limit of US treatment at $f = 35 \dots 45$ kHz and $P_{\text{US}} = 30$ W should be < 9 mins at $T = 15^\circ\text{C}$, < 6 mins at $T = 25^\circ\text{C}$, and < 3 mins at $T = 35^\circ\text{C}$.

In Figure 6d, the histogram shows an increase in the optical transparency of the oil medium as a result of US treatment, which indicates a decrease in energy loss of detected IR radiation and an increase of SNR . This conclusion is supported by observed increase in I_{out} . The highest ΔI was recorded at point ($T = 15^\circ\text{C}$; $t = 15$ min) and amounted to 51.36%.

Figure 8 shows the main dependencies obtained during experimental studies. From the results, it can be seen that Cl_a and μ have a direct correlation in a generalized form up to a certain point in processing time. Therefore, the reduction of Cl_a through US processing has a limited and finite importance.

The research results are presented in the form of a system of equations describing the viscosity-optical properties of oil samples under US exposure (Table 5). System of equations for oil sample at $T = 15^\circ\text{C}$ has the highest correlation coefficient.



Figure 7. a) Oil before processing ($T = 25^\circ\text{C}$; $t = 0$ min); b) Oil at the point ($T = 25^\circ\text{C}$; $t = 6$ min); c) Oil at the point ($T = 25^\circ\text{C}$; $t = 12$ min)

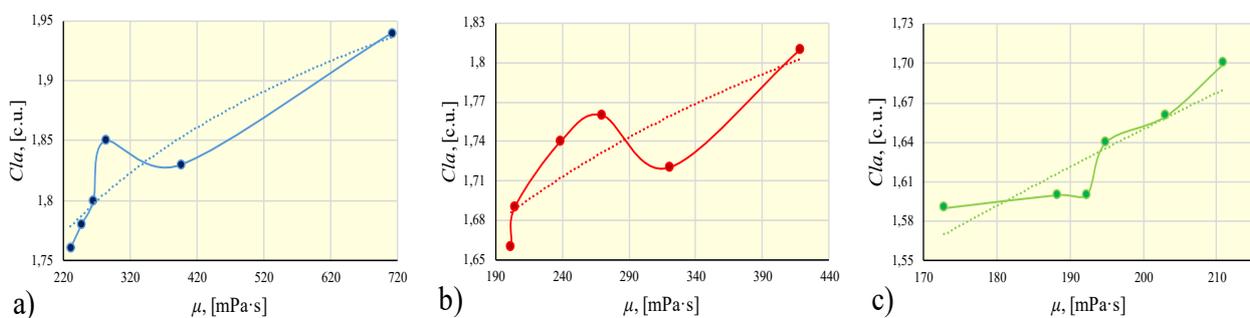


Figure 8. Viscosity-optical curves of oil samples obtained during US treatment: a) $T = 15^\circ\text{C}$; b) $T = 25^\circ\text{C}$; c) $T = 35^\circ\text{C}$

TABLE 5. Systems of equations describing the viscosity-optical properties of oil samples under US exposure

| Oil sample | The system of equations |
|-------------------------------|--|
| At a $T = 15^{\circ}\text{C}$ | $\begin{cases} \mu = e^{15,6-0,03\tau}; R(\tau; \mu) = 0,95, R^2 = 0,90 \\ Cla = 1,18 \cdot \mu^{0,08}; R(\mu; Cla) = 0,93, R^2 = 0,86 \end{cases}$ |
| At a $T = 25^{\circ}\text{C}$ | $\begin{cases} \mu = e^{13,48-0,03\tau}; R(\tau; \mu) = 0,99, R^2 = 0,99 \\ Cla = 1,04 \cdot \mu^{0,09}; R(\mu; Cla) = 0,85, R^2 = 0,73 \end{cases}$ |
| At a $T = 35^{\circ}\text{C}$ | $\begin{cases} \mu = e^{7,17-0,01\tau}; R(\tau; \mu) = 0,91, R^2 = 0,83 \\ Cla = 0,27 \cdot \mu^{0,34}; R(\mu; Cla) = 0,88, R^2 = 0,78 \end{cases}$ |

5. CONCLUSION

Based on the objectives of the study and the results of the experiment, the following conclusions can be drawn:

US treatment reduces the optical density of highly viscous oil. The total decrease was 0.35 c.u., which is -18.04% of the initial value.

Pre-US treatment of oil samples helps to increase the SNR of the IR diagnostic system. The effectiveness of this process was assessed through calculations of ΔI . The highest efficiency of US treatment was achieved at a point (15°C; 15 min) and was 51.34%.

The greatest volatility of the Cl_a reduction curve under US exposure is observed in an oil sample at $T = 25^{\circ}\text{C}$.

Of particular interest is the extremum observed in the US treatment interval, $t = 6..9$ mins, which may be associated with the redistribution of asphalt-resin-paraffin structures in the sample or measurement errors caused by uneven propagation of US vibrations in the bath. This extremum significantly affected the accuracy of the system of equations describing the viscosity-optical properties of an oil sample at $T = 25^{\circ}\text{C}$. Future research should explore the use of US baths with vibration modulation, as this may theoretically reduce the irregularity of cavitation by minimizing interference effects. Additionally, analysis of the intensity distribution of US vibrations within the oil sample could enhance the outcomes of these investigations.

An important consideration in US treatment is mechanical heating, which can affect the chemical composition of oil. This requires the establishment of additional boundary conditions in order to optimize the IR diagnosis of oil based on the "signal-to-noise" criterion to the maximum extent possible. During the study, time limits for US treatment were determined based on the evaporation of petroleum fractions from oil: less than 9 mins at 15°C, less than 6 mins at 25°C and less than 3 mins at 35°C. Additionally, the optimal time for US treatment should be determined taking into account the acceleration or deceleration of heating for oil samples with different thermal capacities.

US oil treatment is a more environmentally friendly

alternative to the use of solvents, as it eliminates the need for the disposal of hazardous chemical reaction byproducts. Existing issues with noise levels and energy consumption may be addressed through the use of higher frequencies, shorter durations, and correctly selected US power.

Before implementing this technique, it is essential to conduct further research, including testing with other types of oils, particularly paraffinic ones. Furthermore, future research should concentrate on the analysis of IR spectrograms acquired at different oil temperatures and under different US treatment times, as well as comparing and contrasting the findings of IR diagnostics of various oil types with the potential for generalization.

6. AUTHORS CONTRIBUTION

Rastvorova I. – Conceptualization, scientific supervision and guidance and interpretation of results;

Filatov V. – Literature review, development of methodology, conducting experiments, processing and presentation of results;

Vilkov S. – Conducting experiments and interpretation of results.

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Persian Abstract

چکیده

امروزه، روسیه و سایر نقاط جهان شاهد افزایش تولید و ذخایر هیدروکربن‌های سنگین (HC) هستند. این امر در مقایسه با ذخایر نفتی متعارف، از نظر نیازهای فنی و مالی، مشکلاتی را ایجاد می‌کند. در این زمینه، یکی از اهداف کلیدی برای پیشرفت بخش منابع معدنی، بهبود سیستم‌های تشخیص میدان نفتی است. این امر "دیجیتالی‌سازی" میدان‌های نفتی و فرآیندهای مربوط به استخراج و حمل و نقل را امکان‌پذیر می‌سازد. هدف از این کار، تجزیه و تحلیل وضعیت فعلی دستگاه‌های تشخیص نفت مادون قرمز (IR)، مشکلات موجود و راه‌های بهینه‌سازی آنها، با توجه ویژه به تصفیه اولیه اولتراسونیک (US) نمونه‌های بسیار چسبناک است. این مطالعه توانست این فرضیه را تأیید کند که تصفیه نمونه‌های نفت رزینی با US منجر به افزایش نسبت سیگنال به نویز (SNR) سیستم تشخیص IR می‌شود. بالاترین راندمان تصفیه US در نقطه‌ای (۱۵ درجه سانتیگراد؛ ۱۵ دقیقه) ثبت شد، جایی که افزایش I_{out} نسبت به نفت تصفیه نشده ۵۱.۳۶ درصد بود. زمان بهینه برای عملیات اولتراسوند بر اساس تأثیر بر ترکیب شیمیایی روغن عبارت است از: $t > 9$ دقیقه در دمای ۱۵ درجه سانتیگراد، $t > 6$ دقیقه در دمای ۲۵ درجه سانتیگراد و $t > 3$ دقیقه در دمای ۳۵ درجه سانتیگراد. علیرغم نتایج مثبت، نویسندگان بر مشکلات مربوط به توزیع ناهموار ارتعاشات اولتراسوند و نیاز به تحقیقات بیشتر در مورد روغن پارافینی تأکید می‌کنند.