

# Paraffinic oil residues after heating as a basis for fuel

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**Abstract.** The possibility of reuse of heavy residues (oily sludge) of low-sulfur, highly paraffinic Mangyshlak oils as a basis for fuel with improved environmental characteristics for engines to marine, river and railway transport, gas turbine and boiler plants, steam boilers and industrial furnaces was shown. A variant of the joint application of the methods of nuclear magnetic resonance, laser confocal microscopy and rheology for study of micro- and mesoscopic structure and some physicochemical properties of crude oil products has been developed. It has been shown that preliminary heating to 90 ° C leads to disruption of the structure and hydrogen bonds between oil associates and complexes, increasing sample homogeneity due to precipitation of solid impurities, partial removal of water content, air bubbles and dissolved gases. The addition of medium distillate diesel fractions to preliminary heated oil residues will make it possible to obtain a higher quality version of the fuel with a reduced resinous and asphaltene substances, lower pour point, good energy content and low sulfur content. Thus, valuable energy-containing raw materials can be reused. It should also be noted that the reuse of accumulated oil waste will reduce the harmful effects of landfills, will preserve the natural landscape, and will cut the cost of extracting natural resources. Keywords: fuel, oil residues, NMR, viscosity, structure.

## 1 Introduction

Fuel oil is widely used not only as a raw material for high-tech secondary processing processes, but also serves as a source of energy for marine engines, gas turbine and boiler plants, steam boilers and industrial furnaces. The main consumers of fuel oil are river and marine fleet, sphere of housing and communal services, power plants. The physical and chemical properties of fuel oil depend on chemical composition of original oil and degree of its processing. Marine fuel oil and M-40 fuel oil are similar in their characteristics, but marine fuel oil is still of a higher quality. In the process of producing these brands of fuel

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oil, residual oil products, such as fuel oil, tar, heavy gas oils of secondary processes are mixed with 8 - 12% (up to 30% for marine fuel oil) of middle distillate diesel fractions.

Oil residues are a waste generated at all stages of oil production, transportation, and refining. The main reason for the formation of oil residues is the interaction of oil products with moisture, air oxygen and mechanical impurities. The properties and composition of oil residues depend on chemical compound of the feedstock, technological processes used at manufacturing of oil products, the duration and conditions of its storage. Superficial oil refining, previously practiced in republics of the ex-USSR, led to accumulation of large quantities of energy-intensive raw materials suitable for re-processing. Fuel oil and some types of oil residues can be very similar in composition and their physical and chemical properties. In particular, they can have similar viscosity values, which can be reduced by using different fuel additives. (Zvereva et. al., 2018; 2019; Zvereva et al., 2016a, 2016b, 2016c). Also, both products are close in assessment of their energy intensity. But the oil residues from Mangyshlak oils has one remarkable feature - a small amount of sulfur in its composition. Considering Mangyshlak Peninsula location on the Caspian Sea and near the water area of large rivers, where there is a need for marine fuel, we were interested by using oil residues as a basis for fuel, primarily for low-sulfur marine fuel. It should be noted that the development of environmentally friendly methods for disposal of oil residues is an urgent problem (Hu et. al., 2013; Hu et. al., 2020; Chen et. al., 2019). The presence of a large amount of oil residues stored at open landfills increases the risk of groundwater and air pollution. Pollution of soils with oil products, in addition to its direct impact, can lead to an excess accumulation of heavy metals such as zinc, copper, lead in them. Reuse of accumulated oil waste will reduce the harmful impact of landfills, preserve the natural landscape and the cost of extracting natural resources (da Silva et al., 2012; Johnson and Affam, 2019).

The relevance of search for new sources of low-sulfur hydrocarbon raw materials is beyond doubt. The possibility of reducing oil residues dumps along with extracting a useful energy-intensive product is important for improving the ecology of the peninsula and the economy of the region. The purpose of this work was to study the possibilities of using low-sulfur oil residues from Mangyshlak oil as a basis for fuel with improved environmental characteristics.

## 2 Materials

### 2.1 Characterization of oil residues

As oil residues, we used a viscous hydrocarbon liquid with a low odor taken from an open-type landfill near Zhetybai field in Kazakhstan. The landfill contains oil residues recovered during the cleaning of tanks for the transportation of crude oil from the fields of the Mangyshlak Peninsula. A study of similar landfills carried out by the authors of (Vdovenko et. al., 2015) showed that oil residues contained in landfills is subject to layer-by-layer phase separation. The upper layer, which can be separated by a layer of water from the rest of the layers, contains the highest quality hydrocarbons, similar in their physical and chemical properties to the original crude oil. The content of light fractions in this layer reaches 30 wt. % at low values of coking capacity and specific viscosity (Vdovenko et. al., 2015). Therefore, this product can be added to crude oil for recycling or used in a similar way to fuel oil.

Features of oil residues are determined by the composition and properties of the original crude oil. Studies of the physicochemical properties of Mangyshlak oils show that they are low-sulfur (sulfur content - 0.06-0.18 wt. %), with low acidity, large amounts of saturated

hydrocarbons and resins and with low content of asphaltenes (Zvereva et al, 2022). The characteristics of Zhetybai oil residues were investigated in paper (Zueva et al, 2022). Measurement of its density at 20 °C gave a value of 939 kg/m<sup>3</sup>. Calculated API gravity equal to 18.8° corresponds to heavy crude oil. Investigated sample of Zhetybai oil residues contains 68.5 wt. % saturated hydrocarbons, 16.3 wt. % aromatic hydrocarbons, 14.6 wt. % resins, and only 0.6 wt. % C<sub>6</sub> asphaltenes. The sulfur content in oil residues is 0.23 wt. %. Such values of sulfur concentration are quite consistent with those of low-sulfur fuels.

Oil residues from other Mangyshlak's deposits are close to them in properties.

## 3 Method

### 3.1 Studies of conditional viscosity of oil residues

Determination of the conditional viscosity of oil residues (in Engler degrees, °E) was carried out using an Engler viscometer VU-M-PCP. At room temperature, a sample of the investigated oil residues was an unformed mass. After mixing and heating, this mass became liquid, which made it possible to measure its conditional viscosity. It turned out that the conditional viscosity of oil residues at a temperature of T = 50 °C is 4.2 °E.

### 3.2 Nuclear magnetic resonance method

To analyze the experimental results, data obtained by the method of nuclear magnetic resonance were used. Proton NMR spectra were recorded on an AVANCE III 600 MHz NMR spectrometer (Bruker) operating at a proton resonance frequency of 600.13 MHz and equipped with a high-resolution inverted probe (TXI, 5 mm). Details of the measurement process were given in (Zueva et al, 2022).

### 3.3 Study of the structure of oil residues

Images of the microstructure of oily sludge were obtained using a scanning laser confocal microscope LSM-510 Meta (Carl Zeiss, Germany) operating in an optical mode. To obtain images, a small amount of the sample was slightly warmed up, dropped onto a glass slide, smeared with the end of the cover glass, and then covered with the same cover glass.

### 3.4 Study of the lower heating value of oil combustion

The study of the lower heating value of combustion of oily sludge Q, carried out using an ABK-1B adiabatic bomb calorimeter.

## 4 Experimental Section

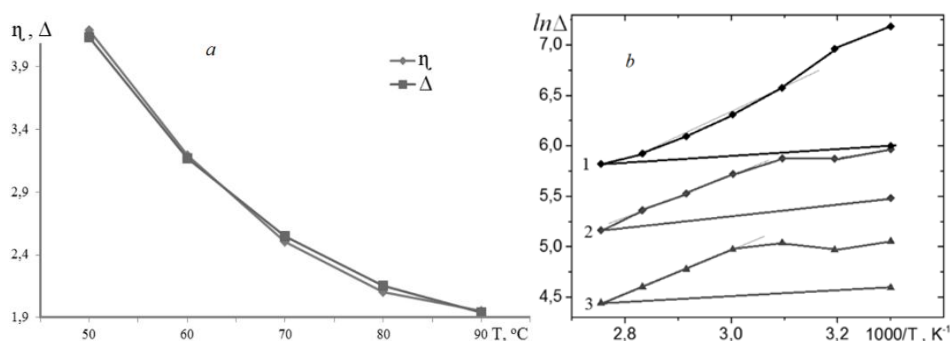
### 4.1 Studies of conditional viscosity of oil residues

Viscosity is one of the most important physical properties of oil, largely determining the processes of its production, transportation, and processing. The values of the viscosity of a petroleum product along with its density allow an approximate estimate of its composition. When measuring the conditional viscosity of the initial oil product at 50 °C, an average value of 4.187 °E was obtained. It should be noted that this value turned out to be even lower than that of various samples of M100 fuel oil with which we worked. The viscosity

increases with growth of molecular weight of the fractions. Therefore, it can be concluded that this oil residues contains, along with resin-asphaltene substances, a sufficient amount of oil fractions suitable for use as fuel or for recycling in a mixture with crude oil.

In the study of the temperature dependence of the viscosity of investigated oil product, a curve  $\eta(T)$  was obtained (Fig. 1, a), showing a decrease in the viscosity with increasing temperature. When the temperature of oil residues raises to 80–90 °C, its viscosity decreases to about 2 °E which indicates a good degree of sprayability of this oil product under engine performance.

Various empirical correlations were proposed to describe the viscosity of crude oil and petroleum products (Balestrin and Loh, 2020; Zadymova et. al., 2016; Malkin, 2019; Imamverdiyev et. al., 2019; Aralov et. al., 2019). They can give the most accurate coincidence of the calculated and experimental curves, which is convenient for engineering calculations. However, they do not always describe the essence of ongoing processes at the level of intermolecular interactions.

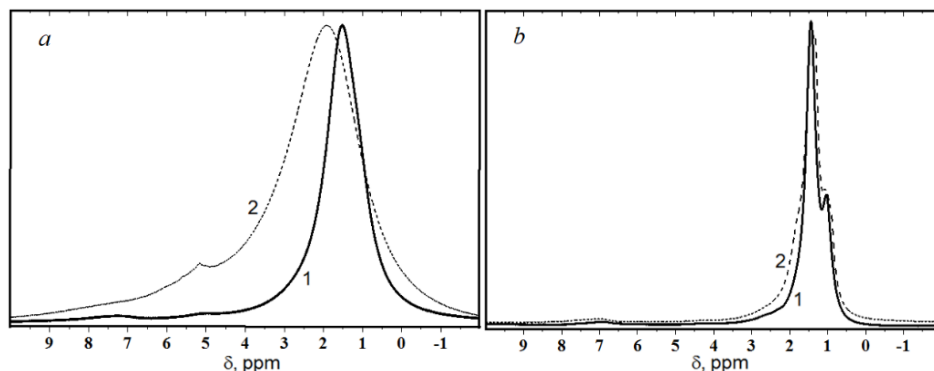


**Fig. 1.** The plots of the temperature dependences of (a) conditional viscosity  $\eta(T)$  in °E and NMR line width  $\Delta(T)$ , expressed in nominal units (Hz/174); (b)  $\ln\Delta$  versus  $1/T$ . Line 1 corresponds to the first heating (signal from the protons of  $\text{CH}_2$  and  $\text{CH}_3$  groups, common line), and then cooling. Signal from protons of  $\text{CH}_2$  groups (line 2) and  $\text{CH}_3$  groups (line 3) correspond to the second heating after 4 days and subsequent cooling.

## 4.2 Nuclear magnetic resonance measurements

To clarify the ongoing processes, we studied the NMR signals of the oil residues samples taken at different temperatures. The NMR method makes it possible to predict the composition of crude oil and oil products (Sadykov et. al., 2012; Sanchez-Minero et. al., 2013) and to study the relationship between the physicochemical properties of oil and the processes of structural aggregation of components in its volume (Zielinski et. al., 2010; Gao et. al., 2020; Korb et. al., 2015).

NMR spectra of two samples of oil residues at 30 °C (a) and 90 °C (b) are shown in Fig. 2. The lines in the spectra are characterized by a significant width, of the order of 500 Hz, since the sample is viscous. The available peaks can be attributed to the following groups: 0-3 ppm - signals from  $\text{CH}_3$  and  $\text{CH}_2$  groups; 4-5 ppm - signals from OCH and  $\text{OCH}_2$  groups, as well as from  $\text{H}_2\text{O}$ ; 6-9 ppm - signals from aromatic groups. A more detailed correlation of signals from different groups of the spectrum is described in the work (Sadykov et. al., 2012).



**Fig. 2.**  $^1\text{H}$  NMR spectra of two samples of oily sludge at a temperature of  $30\text{ }^\circ\text{C}$  (a) and  $90\text{ }^\circ\text{C}$  (b). The solid line is sample 1, the dashed line is sample 2.

It can be seen that the initial NMR spectra of two various samples recorded at  $30\text{ }^\circ\text{C}$  are quite different (Fig. 2, a). This distinction, first of all, may be due to the different supramolecular structure, which is formed by paraffins inclined to association, which are present in large quantities in the Mangyshlak oils. In addition, this behavior may be due to the presence of impurities and inhomogeneities in the samples. After heating to  $90\text{ }^\circ\text{C}$  the lines narrowed and separated, the chemical shift slightly changed (Fig. 2, b). The spectra obtained from different samples became almost identical. We want to emphasize this fact, since for processing oil is taken from different parts of the landfill with initially different characteristics, and then, as experience shows, almost the same result is obtained. This may be due to the destruction of the features of supramolecular structures formed by paraffins and ceresins. This behavior can be caused by other factors, such as disruption of the structure of hydrogen bonds between associates and complexes of oil structures, increasing sample homogeneity due to dissolution and/or precipitation of solid structures (for example, particles of impurities or asphaltenes), removal of air bubbles and dissolved gases. A certain contribution can be made by different water content in the sample structure. During heating, water can be separated or evaporated, additional hydrogen bonds are destroyed. At  $90\text{ }^\circ\text{C}$  separation of signals from  $\text{CH}_3$  and  $\text{CH}_2$  groups and from protons of aromatic groups appeared in the spectra. The spectra of the samples of the investigated oil product became very similar to the signal from the crude oil.

### 4.3 Study of the structure of oil residues

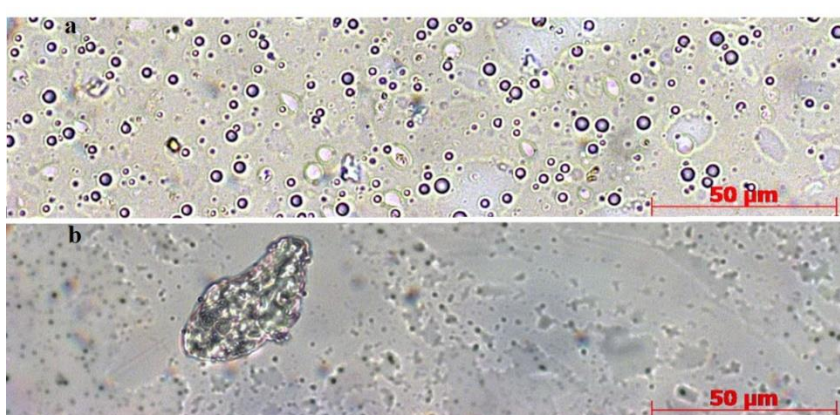
The study of the structure of oil residues using confocal microscope showed that oil residues, like oil, has a colloidal nature. In Fig. 3a shows images of a thin layer of oil residues between the glasses. It can be seen that the studied oil product is a medium in which particles of spatial supramolecular structures are located. They consist of a variety of macromolecular compounds formed by saturated and aromatic hydrocarbons, as well as resins and asphaltenes which are the highest molecular weight polar components of oil (Akbarzadeh et. al., 2007; Ganeeva et. al., 2011). Resinous and asphaltene substances form the cores of many spherical formations. It is known that asphaltenes tend to self-aggregate and can form not only nanoaggregates of asphaltenes (Mullins, 2010; Mullins et. al., 2011), but also clusters of asphaltene nanoaggregates (Eyssautier et. al., 2012; Mullins et. al., 2013). In accordance with size of the asphaltene cores (Fig. 3a), asphaltenes can form not only clusters, but also conglomerates consisting of a large number of clusters, possibly connected, or surrounded by resins (Gray et. al., 2011; Balestrin and Loh, 2020). In fig. 3a, resinous and asphaltene substances are represented by spherical formations ranging in size

from several hundred nanometers (resolution limit of a confocal microscope) to about 3  $\mu\text{m}$ .

In addition to them, the image clearly shows amoeba-like oval formations with an average size of 5-20 microns. In our opinion, these are gel-like regions formed by molecules of paraffins and ceresins, which tend to form spatial structures. It is known that all types of crude oil products contain water-in-oil emulsions (Zadymova et. al., 2016). We assume that such emulsions can be an integral part of the internal structures of gel-like regions. Appearance of such areas leads to a loss of mobility of inner layers of oil products.

The viscosity of spherical structures in a liquid medium is largely determined by the viscosity of interlayer. However, the presence of gel-like regions of various sizes and shapes, interacting with each other, actually leads to gelation in the entire volume. That is why below the melting points of paraffins, which fall in the region of 40-50  $^{\circ}\text{C}$ , heavy oil products are typical samples of soft matter having a viscoelastic (gelatinous) structure.

It should also be noted that all supramolecular structures of oil residues are covered with fairly uniform shells (light edging) with a size of 500-800 nm. Since these samples belong to highly paraffinic oil products, we assume that they appear as a result of the deposition of high molecular weight paraffins at the boundaries of formations, which is typical for such oil products. (Malkin, 2019).



**Fig. 3.** Micrographs of a thin layer of oil sludge, taken using a laser confocal microscope LSM-510 Meta at 25  $^{\circ}\text{C}$  (a) and 77.5  $^{\circ}\text{C}$  (b).

#### 4.4 Study of the lower heating value of combustion of a petroleum product

The study of the lower heating value of combustion of oil residues Q, carried out using an ABK-1B adiabatic bomb calorimeter, made it possible to determine the value  $Q = 41.272$  MJ/kg. Note that our earlier measurements of the lower heating value of the M100 brand high-sulfur fuel oil produced by the Nizhnekamsk Oil Refinery gave 41.867 MJ/kg. So, in terms of energy intensity, as well as in rheological properties, these two petroleum products are close to each other.

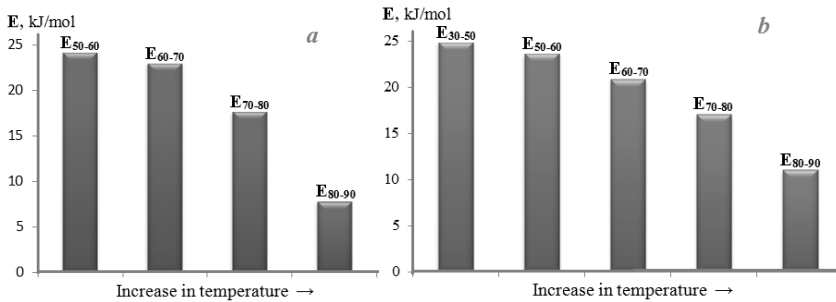
## 5 Results and discussion

Viscosity is the result of the interaction of molecules in the studied dispersed systems and therefore reflects the structural processes occurring in these systems. To a greater extent, the presence of structural rearrangements makes it possible to determine the form of the viscosity dependence plotted in Arrhenius coordinates, when  $\ln \eta$  is considered versus  $1/T$ .

This dependence makes it possible to analyze the temperature dependence of the activation energy of a viscous flow,  $E$ , associated with the microstructural ordering of liquid disperse systems and their stability, and to calculate its values in specific areas (Loskutova et. al., 2014; Nelyubov et. al., 2017). In cases characterized by a linear dependence of  $\ln \eta$  versus  $1/T$ , the activation energy is constant; the structure of the system does not change. Changes signal the presence of structural transformations in the system.

Modern concepts of oil as a dispersed system, in which associates and complexes of paraffin molecules, polycyclic hydrocarbons, resins, and asphaltenes are nanoparticles of a dispersed phase, have increased interest in the study of viscosity. In the case of oil residues, the dependence of  $\ln \eta$  versus  $1/T$  does not change very much when the temperature changes from 50 °C to 80 °C. Visually, in this region the dependence is almost straightforward, although the calculation shows that  $E_{50-60} = 24.2$  kJ/mol,  $E_{60-70} = 23.0$  kJ/mol,  $E_{70-80} = 17.7$  kJ/mol. This means that structural changes actually taking place in the system.

These changes can be associated both with the rearrangement of structures created by paraffinic hydrocarbons and with a decrease in the degree of binding of resinous-asphaltene associates into large spatial structures. In the last studied temperature region, the activation energy calculated from the data of viscosity measurements drops sharply, amounting to  $E_{80-90} = 7.8$  kJ/mol. Such data indicate further changes in the supramolecular structure of the substance and, possibly, signal about irreversible rearrangements characterized by the disintegration of existing formations, the association of part of the structures into large complexes and the sedimentation of large complexes.



**Fig. 4.** Change in the activation energy of oily sludge with increasing temperature: a - according to the viscosity data, b - according to the data on the width of the main line of the NMR spectrum.

Narrowing of the NMR line (Fig. 2b), the width of which  $\Delta$  is related to the viscosity  $\eta \sim \Delta$  of the investigated liquid (Korb et. al., 2015; Hirasaki et. al., 2003; Mutina et. al., 2008; Benamsili et. al., 2014), univocal indicates a decrease in viscosity due to heating of the oil product and the stabilization of this effect, at least for several days. The correspondence of the NMR line width  $\Delta(T)$  during its first heating (Fig. 1a), the previously obtained values of the conditional viscosity  $\eta(T)$  (Fig. 1a) is shown for temperature region 50-90 °C. The dependences of the width of the NMR lines  $\ln \Delta$  (Fig. 1b) versus  $1/T$  made it possible to calculate the activation energy from the NMR data for each temperature region (Fig. 4b) and were demonstrate clearly that the activation energy, proportional to the slope tangents (depicted by thin gray lines in Fig. 1b) for sections 60-80 °C are almost the same. The calculations show that, the values of the activation energy in the range 60-80 °C are  $E_{60-80} = 18.9$  kJ/mol for curve 1,  $E_{60-80} = 17.6$  kJ/mol for curve 2,  $E_{60-80} = 18.1$  kJ/mol for curve 3. These data from different areas indicate that the same type of processes occur in the system in the specified temperature range.

Only the region 80-90 °C of the first heating is different, where the activation energy, calculated both from the measurements of viscosity and from measurements of the NMR

line width, sharply decreased. This means that as a result of the first heating to 80 ° C, some structural rearrangements could occur in the oil product. To clarify this circumstance, we obtained new images (Fig. 3b) of the structure of the heated oil residues using an LSM-510 Meta scanning laser confocal microscope and a thermostat, which allowed the samples to be heated to 77.5 ° C.

All images obtained at the temperature of 77.5 ° C are characterized by dramatic changes:

- large resinous-asphaltene spherical particles were disintegrated into many small ones, up to 1 micron in size;
- the outer shell of the structural formations has dissolved;
- the number of amoeba-like gel areas has significantly decreased, and their boundaries have become indistinct;
- large composite particles capable to sedimentation appeared in all images;
- the content of large composite particles includes convoluted cord-like structures of a dark color, possibly consisting of associates of asphaltene particles interconnected.

Comparison of images at different temperatures allowed us to draw the following conclusions. In the process of heating, paraffins are dissolved first, and then ceresins with higher molecular weight dissolved. Accordingly, resinous and asphaltene formations lose their paraffin shells. At the next stage, part of the resinous and aromatic compounds is dissolved. As a result, associates of asphaltene particles decompose. Asphaltene particles have the tendency to self-aggregation. Therefore, it is possible that some of them were combined into mesoscopic structures. These structures roll up into coils, envelope with resinous and aromatic compounds and then precipitate. The result of such changes is elimination of a certain excess of resinous-asphaltene substances and a decrease in the viscosity of crude oil products. Thus, after such sedimentation an improvement in the fuel properties of oil residues occur.

Our studies have shown that the samples of the studied oil residues at elevated temperatures are very similar in their properties to M100 fuel oil, which suggests the possibility of its use for marine engines, gas turbine and boiler plants, steam boilers and industrial furnaces. Its additional mixing with 8-12% (for naval fuel oil up to 30%) of medium-distillate diesel fractions will make it possible to obtain a higher quality version of the fuel with lower pour points.

## 6 Conclusions

The work done has shown the possibility of using of highly paraffinic low-sulfur Mangyshlak oil residues as a basis for fuel with improved environmental characteristics for marine engines, gas turbine and boiler plants, steam boilers and industrial furnaces. The joint application of methods of nuclear magnetic resonance, laser confocal microscopy and rheology for the study of the micro- and mesoscopic structure and some physicochemical properties of crude oil products have been developed. It has been established by microscopy that when oil residues are heated, large resinous-asphaltene spherical particles disintegrate into many small ones with sizes up to 1 micron, the outer paraffin shell of structural formations partially dissolves, and large composite particles capable of sedimentation appear. It has been shown by NMR that preliminary heating to 90 ° C leads to disruption of the structure of hydrogen bonds between associates and complexes of oil structures, increasing sample homogeneity due to precipitation of solid impurities, partial removal of water content, air bubbles and dissolved gases. It was assumed that a decrease in viscosity and an improvement in fuel properties are a consequence of such changes. The addition of medium distillate diesel fractions to heated oil residues will make it possible to obtain a quite higher quality version of the fuel with a reduced resinous and asphaltene



substances, lower pour point, good energy content and low sulfur content. Thus, valuable energy-containing raw materials can be reused. It should also be noted that the reuse of accumulated oil waste will reduce the harmful effects of landfills, will preserve the natural landscape, and will cut the cost of extracting natural resources.

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