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PROCESSING OF OILY WASTE. 1. LOW-TEMPERATURE PYROLYSIS OF THE ORGANIC PART OF OIL SLUDGE

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Abstract. The article presents the results of studies on the composition and properties of the organic part of oil sludge formed during oil production in the fields of western Ukraine. The thermal stability of the oil sludge organic part was studied. As a result of low-temperature pyrolysis of the organic (hydrocarbon) part of oil sludge, pyrolysis oil (pyrocondensate) was obtained. The fractional composition and properties of pyrolysis oil were studied. Pyrolysis oil was separated into gasoline fraction, diesel fraction, and the residue. The composition and properties of these fractions were determined. X-ray fluorescence analysis of pyrolysis oil and narrow fractions separated from it was performed. The composition and properties of the solid residue of the oil sludge hydrocarbon part after the pyrolysis process were investigated. It is proposed to use the narrow fractions separated from pyrolysis oil to produce motor fuels.

Keywords: oily waste, oil sludge, pyrolysis, pyrolysis oil, pyrocondensate, gasoline fraction, diesel fraction, residue.

1. Introduction

Oil sludge is a complex mixture of organic matter (petroleum products), mechanical impurities (clay, metal oxides, sand), and water.[1] Oil sludge is generated mainly during crude oil production, and less often during its refining and transportation.[2,3]

In most cases, oil sludge is accumulated in special pits or storage ponds and harms the environment, polluting the air and groundwater.[4,5] This problem is exacerbated by the large amount of such waste that has been accumulating for decades. On the other hand, oily waste contains a significant amount of hydrocarbons of various structures, which are valuable raw materials for the oil refining and petrochemical industries. That is why the problem of oil-containing waste processing is extremely urgent and needs to be addressed immediately.[6]

Oil sludge, which is the main oily waste by volume, consists of hydrocarbons, water, and mechanical impurities.[7] Oil sludge processing is complicated by its heterogeneity (its composition varies along the depth of the tank where it is stored), as well as significant contamination with mechanical impurities (mineral rock, sand, fallen leaves, and foreign objects). That is why only the surface layer of sludge ponds is often processed.

Most oil sludge utilization technologies involve the separation of the organic part of oil sludge using mobile three-phase centrifuges.[8,9] The separated organic part is transported for further use, while water with mechanical impurities remains in the pond. The organic part of oil sludge is used as fuel,[10] and less often it is combined with crude oil for processing at refineries.[11] Incineration of the oil sludge organic part causes air pollution with ash and is not the best method of its utilization.[12]

The methods of thermal decomposition and coking of the organic part of oil sludge are well-known[13-16] but they are not sufficiently studied and used. These methods require the installation of special equipment, and significant capital and operating costs. Another important aspect is the need to study the thermal decomposition of oil sludge of various origins.

Given that the composition of not only oil sludge, but also the organic part separated from it, depends on the characteristics of crude oil, and oil sludge generated from Ukrainian oils has not been studied sufficiently, it is necessary to study oil sludge generated at Ukrainian oil fields and develop methods for its rational utilization.

The purpose of the article is to study the composition and properties of oil sludge obtained during crude oil production in the fields of western Ukraine, to carry out the process of low-temperature pyrolysis of oil-containing waste, to study the properties of pyrolysis products, and to propose rational ways of their use.

2. Experimental

2.1. Materials

The starting materials for the research were oil sludge obtained during oil production. Four samples were

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taken at the Boryslavske oil field, which is located in western Ukraine. They were taken from different places of oil sludge storage after mixing them. In appearance, oil sludge is a viscous product containing water and mechanical impurities of different dispersions.

2.2. Methods

To separate the organic part from the oil sludge, we used settling at a temperature of 90ºC with the addition of water and stirring. Under these conditions, the bulk of the water and mechanical impurities were separated. The separated contaminated organic part of the oil sludge was dried with calcium chloride and filtered at a temperature of 80–90ºC to separate $CaCl_2$ particles and residual particles of mechanical impurities.

Comprehensive thermal analysis of the oil sludge was performed on a Paulik-Paulik-Erdey derivatograph Q-1500 (IOM, Hungary) connected to a personal computer. The samples of 100 mg by weight were analyzed in a dynamic mode at a heating rate of 10°C per minute in an air atmosphere. Aluminum oxide was the reference substance.

The process of low-temperature pyrolysis of oil sludge was carried out in a laboratory setup consisting of a metal-sealed reactor, a water cooler, and a receiving flask. The temperature in the reactor was measured using a thermocouple. A sample of oil sludge was loaded into the reactor, closed with a hermetic lid with bolted connections. The cooler was connected to the gas-removing tube. An electric heater was turned on, and water was circulated in the cooler. The temperature was gradually raised to the operating temperature required for pyrolysis. The pyrolysis oil that condensed in the refrigerator flowed into a receiving flask. The non-condensed part of the pyrolysis products (syngas) was discharged into the atmosphere. After the pyrolysis process was terminated (liquid flow into the receiving flask stopped), the heating was turned off. The amount of pyrolysis oil was determined by weighing the receiving flask before and after the experiment. After cooling the reactor, it was weighed, and the residue yield was determined. The pyrolysis process occurred with gradual heating. It began at a temperature of 380°C and continued at a temperature of 420°C for 70 min.

To conduct research and establish the possibility of using pyrolysis oil after the pyrolysis of oil sludge to produce motor fuels, it was separated into fractions of IBP–200ºC, 200–350ºC, and a residue >350ºC. The pyrolysis oil separation process was carried out using a classical laboratory setup for the separation of light oil products. The setup consists of a flask, a flask heater, a water cooler, a "spider", and receivers for collecting narrow fractions.

X-ray fluorescence spectral analysis to determine the elemental composition of liquid and solid pyrolysis products was performed on an Elvax Light SDD precision analyzer.

All analyses of the pyrolysis oil, the fractions, and the residue separated from it were performed according to generally accepted standardized methods. The fractional composition of the light fractions of pyrolysis oil was determined using an ARNS apparatus. The iodine number of the liquid fractions was determined according to the Margoshes method. In addition, flash point in closed and open cups, pour point, cloud point, density, and refractive index of pyrolysis oil and fractions separated from it were determined.

3. Results and Discussion

3.1. Study of Oil Sludge Composition and Properties

The first stage of research was to study the content of individual phases in the oil sludge. For this purpose, the oil sludge samples were divided into three phases: water, mechanical impurities, and hydrocarbon part. The composition of oil sludge is shown in Table 1. It was found that the composition of oil sludge samples varies widely and depends on the conditions and terms of storage, the amount of precipitation, *etc*. In addition, the composition of oil sludge varies with the depth of the container where it is stored.

Table 1. Composition of oil sludge generated at an oil field

Content in oil sludge, wt%	Sample number			
	1	2	3	4
Water	34.8	11.5	33.8	24.4
Mechanical impurities	45.5	13.3	24.4	19.7
Hydrocarbon part	19.7	75.2	41.8	55.9

To establish the possibility of further processing of the organic part of oil sludge, it was necessary to study in detail its physical and chemical properties. For this purpose, the main indicators of the organic part of oil sludge samples were determined (Table 2). It was found that the organic (hydrocarbon) part of the oil sludge is characterized by a very low content of light fractions that boil to a temperature of 350ºC and, as a result, high density, pour point, and flash point. The sulfur content in the samples of the oil sludge organic part is 0.65–0.81 wt%, which is fully correlated with the sulfur content in Boryslavska oil (0.52 wt%). High values of oil sludge ash content may be due to the concentration of metals in the organic part of oil sludge, as well as the presence of microparticles of mechanical impurities.

Table 2. Characteristics of the organic part of oil sludge generated at an oil field

Index	Value			
	Sample 1	Sample 2	Sample 3	Sample 4
Density at 20°C, g/cm^3	927	941	935	952
Mechanical impurities content, wt%	0.4	0.2	0.3	0.3
Water content, wt%	0.4	0.5	0.3	0.4
Sulfur content, wt%	0.77	0.81	0.67	0.65
Pour point, °C	–3	11	6	21
Cloud point, °C	93	131	113	186
Cokeability, wt%	4.1	6.9	6.0	8.5
Ash content, wt%	0.23	0.24	0.22	0.29
Fractional composition: to 200 °C boiled, wt% to 350 °C boiled, wt%	 2.5 19.2	 1.5 12.8	 1.8 16.7	 0.9 9.3

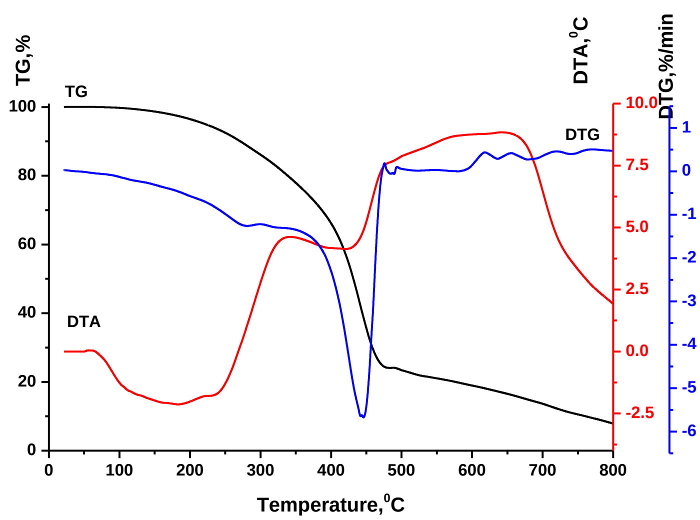

Fig. 1. Derivatogram of the organic part of oil sludge generated at oil fields

3.2. Study on the Process of Low-Temperature Pyrolysis of the Oil Sludge Organic Part

The second stage of the research was the low-temperature pyrolysis of four samples of the oil sludge. To establish the temperature interval of thermal decomposition of the components of the oil sludge organic part and study their behavior when heated, derivatographic studies were carried out (Fig. 1). To do this, we used an average sample of the organic part of oil sludge generated at oil fields.

As can be seen from Fig. 1, three characteristic temperature intervals can be distinguished. In the temperature range of 80-355 ºC, evaporation of light components is observed. With increasing temperature, the processes of thermal destruction of the components occur, which is confirmed by the loss of mass and the appearance of a deep endothermic effect in the temperature range of 335-480ºC. The compaction of the solid residue is confirmed by the appearance of an intense exothermic effect in the temperature range of 480-800ºC. Based on the results of derivatographic studies, the temperature of 420ºC was chosen for the low-temperature pyrolysis of the organic part of oil sludge.

As a result of low-temperature pyrolysis, pyrolysis oil (a mixture of hydrocarbons formed as a result of thermal decomposition), a small amount of solid residue, and pyrolysis gases were obtained. Further, we studied pyrolysis oil and the solid residue. We did not study the pyrolysis gases, since in industrial pyrolysis plants they are used as a gas for the own needs of the plant, *i.e.*, they are burned to generate heat. The material balance of the pyrolysis process of the organic part of oil sludge is shown in Table 3.

The pyrolysis oil is a transparent, light brown liquid with an odor typical of thermal process products. Table 4 shows the characteristics of the pyrolysis oil.

Table 3. Material balance of low-temperature pyrolysis of the organic part of oil sludge generated at an oil field

Raw material and products	Amount, wt%			
	Sample 1	Sample 2	Sample 3	Sample 4
Input: Oil sludge	100.0	100.0	100.0	100.0
Output: Pyrolysis oil Residue (char) Gases and losses	 89.8 5.2 5.0	 85.5 8.3 6.2	 87.9 6.4 5.7	 82.0 10.8 7.2
Total	100.0	100.0	100.0	100.0

Table 4. Characteristics of pyrolysis oil obtained after pyrolysis of the organic part of oil sludge generated at an oil field

Index	Value			
	Sample 1	Sample 2	Sample 3	Sample 4
Appearance	Light-brown liquid			
Density, kg/m^3	848.5	849.2	850.4	849.1
Refractive index	1.5159	1.5116	1.5134	1.5127
Sulfur content, wt%	0.72	0.77	0.59	0.64
Pour point, °C	–7	–5	–4	–6
Flash point open cup, °C closed cup, °C	 52 28	 54 29	 55 29	 54 28

The high iodine number of pyrolysis oil indicates a significant amount of unsaturated hydrocarbons in it. Judging by the refractive index, aromatic structures predominate in the pyrolysis oil. This is confirmed by the rather low value of its pour point. Pyrolysis oil is also characterized by a low flash point, which makes it impossible to use it as a heating or fuel oil. The boiling point of pyrolysis oil is 65ºC and is higher than the boiling point of average crude oils.

Despite the significant difference in the composition and properties of the studied oil sludge samples, the obtained pyrolysis oil samples are characterized by similar properties (Table 4). Therefore, pyrolysis oil obtained by mixing the four samples was used for further research.

The third stage of the research involved separating pyrolysis oil into narrow fractions (gasoline, diesel oil, and residue) and studying their properties to establish the direction of further practical application.

Table 5 shows the characteristics of the gasoline fraction of pyrolysis oil from the pyrolysis of the organic part of oil sludge. It was found that the fraction of IBP-200ºC is characterized by a slightly weighted fractional composition. The sulfur content in this fraction exceeds the requirements for commercial motor gasoline. In addition, this fraction is characterized by a fairly high content of unsaturated hydrocarbons, as indicated by the high iodine number. Such characteristics of this fraction make it impossible to use it as a component of commercial motor gasoline without additional processing.

Table 5. Characteristics of the gasoline fraction of pyrolysis oil obtained after pyrolysis of the organic part of oil sludge generated at an oil field

Index	Value
Appearance	Transparent light-yellow liquid
Yield relative to pyrolysis oil, wt%	22.9
Density, kg/m^3	751
Refractive index	1.5087
Fractional composition, °C: IBP 10 % 50 % 90 % EBP	 66 77 135 182 203
Sulfur content, wt%	0.384
Iodine number, g I_2/100 g	68.7

For the possible use of the gasoline fraction of pyrolysis oil obtained after oil sludge pyrolysis as a component of commercial motor gasoline, it is necessary to carry out its hydroforming, including hydrogenation of unsaturated hydrocarbons and hydrofining to reduce sulfur content. Then, it is advisable to process this fraction at a catalytic reforming unit to increase its octane number. It should be noted that such additional processing of the gasoline fraction of oil sludge pyrolysis oil can be carried out in a mixture with gasoline obtained during oil refining at oil refineries.

It has been established (Table 6) that the pyrolysis oil fraction of 200-350°C is characterized by a high flash point that meets the requirements for diesel fuels. The low-temperature properties, characterized by cloud point and pour point, are sufficient to use this fraction for the production of summer diesel fuels. However, high content of sulfur and unsaturated hydrocarbons needs to be reduced, and for this purpose, additional processing stages of this fraction must be used.

Table 6. Characteristics of the diesel fraction of pyrolysis oil after pyrolysis of the organic part of oil sludge generated at an oil field

Index	Value
Appearance	Transparent dark-yellow liquid
Yield relative to pyrolysis oil, wt%	58.7
Density, kg/m^3	843
Refractive index	1.5167
Iodine number, g I_2/100 g	61.3
Fractional composition, °C:	
IBP	193
10 %	225
50 %	278
90 %	336
98 %	352
Sulfur content, wt%	0.520
Cloud point, °C	–8
Pour point, °C	–15
Flash point in closed cup, °C	73

Further, the diesel fraction of oil sludge pyrolysis oil should also be sent for hydroforming, which includes hydrogenation of unsaturated hydrocarbons and hydrofining to reduce sulfur content. After such processing, this fraction can be used as a component of commercial diesel fuels.

The residue >350°C after pyrolysis oil distillation is a highly viscous product that resembles a grease in appearance when cooled (Table 7). It also contains a significant amount of unsaturated hydrocarbons, as evidenced by the high iodine number, and a large amount of sulfur. Low-temperature properties, characterized by the pour point, as well as an excessively high flash point, make it difficult to use the residue as a component of fuel oil. However, it can be added in small quantities to fuel oils after additional research. Another use of the residue is for the production of various types of greases (including preservation greases). This method of using the residue also requires additional research.

Table 7. Characteristics of the residue >350°C of pyrolysis oil obtained after pyrolysis of the organic part of oil sludge generated at the oil field

Index	Value
Appearance	Viscous brown liquid
Yield relative to pyrolysis oil, wt%	18.4
Density, kg/m^3	917
Refractive index	1.5312
Sulfur content, wt%	1.473
Iodine number, g I_2/100 g	49.7
Pour point, °C	+14
Flash point open cup, °C closed cup, °C	 128 102

The method of X-ray fluorescence spectral analysis was used to determine the content of individual chemical elements in pyrolysis oil, its fractions and residue (Table 8).

Table 8. Content of individual chemical elements in pyrolysis oil after pyrolysis of the organic part of oil sludge and in fractions distilled from pyrolysis oil

Element	Element content, ppm			
	pyrolysis oil	gasoline fraction	diesel fraction	residue
Ca	18.5	9.8	17.8	32.3
Cu	10.4	9.7	10.4	12.3
Zn	0.7	0.2	0.7	1.4
Cr	< 1.8	< 1.7	< 1.8	< 1.8
Fe	< 3.0	< 0.8	< 0.8	10.2
Ni	< 0.3	< 0.3	< 0.3	< 0.3
V	< 0.3	< 0.3	< 0.3	< 0.3
Mn	< 0.1	< 0.1	< 0.1	< 0.1
Ba	< 0.1	< 0.1	< 0.1	< 0.1
Mo	3.8	< 0.3	2.6	12.5
Pb	< 1.0	< 1.0	< 1.0	< 1.0

The content of metals in pyrolysis oil, its fractions, and the residue was found to be insignificant. Among the identified metals, so-called accidental impurities were found, in particular Ca. Another positive aspect is the absence of heavy metals (V, Ni) in pyrolysis oil and its fractions, which are typical for heavy oil fractions and residues. Contrary to the classical ideas about the non-uniform distribution of metals in oil fractions, metals are distributed more or less uniformly in the fractions of the studied pyrolysis oil, although their largest amount is still in the pyrolysis oil residue. The low content of metals in pyrolysis oil and its fractions is a positive fact, since it characterizes the further possibility of their processing in catalytic processes.

To study the general properties of the solid residue obtained after pyrolysis of oil sludge, we determined the ash content, the content of volatile matters, and the moisture content. The elemental composition was studied using X-ray fluorescence analysis (Table 9). The ash content of the residue was found to be 16.8%. The residue of oil sludge pyrolysis contains many different chemical elements (16 elements were identified in total), among which the highest content belongs to Ca, Zn, and Si. There is no clear explanation for the presence of these chemical elements in the residue ash. Obviously, they get into the oil sludge together with oil or the mineral part of mechanical impurities. Analyzing the material balance of pyrolysis (Table 1), as well as the content of chemical elements in pyrolysis oil (Table 8) and its solid residue (Table 9), it was found that the bulk of the metals that are part of the hydrocarbon part of the oil sludge are transferred to the residue as a result of pyrolysis.

Table 9. Characteristics of the solid residue (char) obtained after pyrolysis of the organic part of oil sludge

Index	Value
Ash content, wt%	16.8
Water content, wt%	0.27
Volatile content, wt%	12.9
Individual element content, ppm	
Mg	4918.7
Al	3524.3
Si	134257.7
P	64850.9
S	72548.3
K	2712.5
Ca	228147.4
Ti	1546.2
Cr	132.7
Mn	216.8
Fe	19311.2
Ni	245.7
Cu	17523.2
Zn	218311.4
Sr	225.6
Pb	38.4

Based on the composition and properties of the solid residue obtained after low-temperature pyrolysis of the organic part of oil sludge, it is proposed to use it in road construction for the arrangement of the lower layer of the road surface. Another promising method of using the residue is the production of solid fuel briquettes, but this method requires additional research.

4. Conclusions

We have established the fundamental possibility of processing the hydrocarbon part of oil sludge generated during oil production. The organic part of oil sludge may be processed by low-temperature pyrolysis to obtain liquid and solid products that can be used in the production of motor fuels and other products.

The composition of oil sludge and the properties of its organic part were studied. It was found that the composition of oil sludge varies widely: the content of the organic part is 19.7−75.2 wt%, the content of mechanical impurities is 13.3−45.5 wt%, and the water content is 11.5−34.8 wt%.

The low-temperature pyrolysis of the organic part of oil sludge was carried out at a temperature of 420°C, which resulted in 82.0−89.8 wt% of pyrolysis oil, 5.2−10.8 wt% of solid residue, and 5.0−7.2 wt% of pyrolysis gas.

It has been established that the light fractions obtained during the pyrolysis oil distillation can be used as raw materials for the production of commercial motor fuels. To this end, it is necessary to additionally hydro-process them to reduce the content of unsaturated hydrocarbons and sulfur, as well as to increase the octane number of the gasoline fraction. The residue after pyrolysis oil distillation can be used as a fuel oil component or as an additive to greases. The solid residue formed in the process of low-temperature pyrolysis of oil sludge is recommended for use in road construction for the arrangement of the lower layers of the road surface.

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ПЕРЕРОБКА НАФТОВМІСНИХ ВІДХОДІВ. ПОВІДОМЛЕННЯ 1. НИЗЬКОТЕМПЕРАТУРНИЙ ПІРОЛІЗ ОРГАНІЧНОЇ ЧАСТИНИ НАФТОВИХ ШЛАМІВ

Анотація. *Наведено результати досліджень складу та властивостей органічної частини нафтошламів, утворених під час видобутку нафти на родовищах заходу України. Вивчено термічну стабільність органічної частини таких нафтошламів. У результаті низькотемпературного піролізу органічної (вуглеводневої) частини нафтошламів одержано піроконденсат. Вивчено фракційний склад і властивості піроконденсату. Проведено розділення піроконденсату на бензинову та дизельну фракцію і залишок. Встановлено склад і властивості цих фракцій. Проведено рентгенофлуоресцентний аналіз піроконденсату та вузьких фракцій, виділених з нього. Досліджено склад і властивості твердого залишку процесу піролізу вуглеводневої частини нафтошламу. Запропоновано використовувати вузькі фракції, виділені з піроконденсату, для виробництва моторних палив.*

Ключові слова: *нафтовмісні відходи, нафтові шлами, піроліз, піроконденсат, бензинова фракція, дизельна фракція, залишок.*