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Physico-Chemical Properties of Oil Sludges from Reservoirs

Natalia Vladimirovna Oknina, Khusain Magamedovich Kadiev, Anton Lvovich Maksimov, Alexander Evgenievich Batov, Malkan Khusainovna Kadieva and Askhab Umaltovich Dandaev

Joint-Stock Company «Elektrogorsky Institut Neftepererabotki», Budenova street, Elektrogorsk, Russia 142530.

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Decreasing the anthropogenic impact on the environment during oil production and refining is one of the most complex and diversified problem, requiring considerable resources and continuous monitoring. The resulting considerable amounts of oil sludge are the most dangerous source of pollution in terms of the load on natural environment. Studying physico-chemical properties of hydrocarbon sludge is very important for selecting an efficient method for its recovery. This paper presents the results of studying properties of the various sludge formed during refining, representing a complex system of organics, containing a wide range of hydrocarbons, water and mechanical impurities (sand, clay, silt, etc.), the ratio of which varies widely. As a result of oil sludge heavy residue hydroconversion in the presence of catalyst (MoS2), high degree of conversion of fraction boiling above 520°C (wt.%) and high yield of distillate fractions were achieved. The obtained data are of considerable interest for further research in order to develop technologies of continuous processing of oil sludge of different origin with the use of hydroconversion.

Key words: Oil sludge, Waste products, Reservoir sludge, Hydroconversion.

Production, transportation and processing of petroleum feedstock leads to formation of considerable amounts of oil sludge, increases technogeneous load on the environment that requires additional investment for restoring natural ecosystems[1, 2]. The existing methods of usage, deactivation, storage and disposal of technogeneous wastes do not meet fully the rational use of energy resources, causing economic damage and threatening health of future generations[1-4].

The issue of recycling and disposal of heavy oil residues (sludge, heavy products of oil refining, etc.) is one of the most important ones in the field of ecology, both in our country and abroad. Getting accumulated during storage, transportation and oil refining, oil wastes create considerable pollution of the environment. Absence of effective technologies for isolating and processing the organic part of heavy oil waste leads to wasteful use of oil resources, which may considerably affect not only the environment but the economy of the state as well. Various oil wastes are formed in certain conditions and environments, so their physico-chemical characteristics can change in a wide range. For the purpose of involving oil wastes into processing, research and accumulation of data about their physico-chemical properties are highly relevant. This creates prerequisites for predicting oil wastes transformation during pre-treatment and post-treatment.

The article shows the results of researching physico-chemical properties of oil reservoir sludge formed during petrochemicals

* To whom all correspondence should be addressed.

storage and transportation in a domestic oil depot. Among the indicators that are mandatory and the most important for raw materials of the refining process, the following were considered: content of heavy petrochemicals/oil fractions boiling over 520°C, water, solids and high molecular weight components (asphaltenes and resins). The results of the preliminary study of reservoir sludge heavy hydrocarbon fraction hydroconversion have been presented.

METHODS

Typical sludges formed during oil refining and storage of petrochemicals was taken as the study objects. These include reservoir sludge of the oil depot taken from oil traps of cascade scrubbing structures (No. 1, No. 2 and No. 3), a suction chamber of a drainage system control room (No. 4), oil sludge of presettler and oil trap of the oil refinery (No. 5, 6), as well as reservoir sludge of an oil refinery (No. 7).

Water content in the samples was defined using the method of azeotropic distillation of an oil sample or a petrochemical with solvents (GOST 2477-65). Toluene was used as solvent.

Group composition of petrochemicals was determined using the method of SUE Oil Refining Institute of the Republic of Bachkostostan and a laboratory liquid chromatograph "Gradient-M" intended for determining group chemical composition of heavy oil residues and petrochemicals boiling above 300°C (bitumens, tars, cracking residues, asphaltites, etc.). The method is based on the principles of liquid-adsorption chromatography with gradient displacement and separation into seven groups: paraffinic-naphthenic hydrocarbons, light, medium, heavy aromatic hydrocarbons, resins and asphaltenes.

Group composition of a mixture of light distillate fractions was determined using the Thermo Focus DSQ II mass spectrometer or equivalent (capillary column Varian VF-5ms, 30 m long, internal diameter - 0.25 mm, phase thickness - 0.25 µm, carrier gas is helium, mode of operation: injector temperature - 270 °C, initial chromatograph oven temperature - 30 ºC, followed by isotherm for 5 min, followed by heating at the rate of 10 ºC/min up to 300 ºC, followed by isotherm for 10 min; mode of mass spectrometer operation: ionization energy - 70 eV, source temperature - 230 ºC, scanning in the range between 10 and 400 Da at 2 scan/sec, and single resolution across the mass range). Components were identified with the use of reference mass spectra represented in the database of NIST/EPA/NIH 11. Components content was calculated on the basis of the area of chromatographic peaks on the chromatogram by full ion current, without correction by ionization efficiency. The structural-group composition was calculated on the basis of total mass spectrum of samples across the entire range of elution.

Fractional composition of petrochemicals was determined by the results of distillation at atmospheric pressure and in vacuum according to GOST 11011-85.

Sulfur content in the petrochemicals was determined according to GOST 1437-75 and in the distillate fractions–using an X-ray fluorescence energy dispersive analyzer "SPECTROSCAN-S" in accordance with GOST R 51947-2002, ASTM D 4294-98.

Samples density was determined using a pycnometric method according to GOST 3900. The method is based on comparing the mass of certain amount of petrochemical to the mass of the same amount of distilled water at the same temperature.

The value of coking property of the samples was determined according to GOST 19932 (Conradson's method of determining coking property), based on determining the mass of remaining coke obtained by high-temperature heating and decomposition of the test sample of a petrochemical.

The content of solid (mineral) impurities was determined by a modified method of determining mechanical impurities in oil (GOST 6370-83). The weighed quantity of the tested product was placed in a beaker and dissolved in a predefined amount of toluene. The solution was then filtered through a wet-strong, smooth pre-dried slow filtration filter (grade - MN 1640 de, thickness - 0.17 mm, filtration rate - 140 ml/ cm2•sec, paper density - 85 g/m2, full retention of particle size d ≥ 2 µm). After drying, the filter together with the weighed quantity of the product was weighed, and the content of mineral impurities insoluble in toluene was determined.

Elemental analysis of petrochemicals was performed using the method of atomic emission spectroscopy with inductively coupled plasma (AES-ICP) and mass spectrometry with inductively coupled plasma (MS-ICP). Previously, samples were prepared for placing them into a water-acid solution. For this purpose, the samples were decomposed in an autoclave, and subsequently dissolved in nitric acid.

For determining ash content in the samples, the modified method according to GOST 1461-75 was used. Preliminarily dehydrated oil sludge samples were distilled with separation of fractions boiling above 520°C. The obtained distillation residue was carbonized for 3 hours in an autoclave reactor in a stream of nitrogen with flow rate of 25 l/h at atmospheric pressure at 500-550°C. The resulting crushed distillation residue was calcined in a muffle furnace at 550 °C for 4 hours in a flow of air at the speed of 2.6 l/min, and the ash content was calculated by the yield of ash.

The hydrocarbonaceous oil sludge particles hydroconversion experiments were performed in an autoclave reactor described in [5]with loading raw material mixture up to 100 g, and hydrogen flowing at 18 to 20 nl/h. Hydroconversion was performed at the pressure of 7.0 MPa and the temperature of 430-450°C. The duration of the experiment was 2 hours of high pressure in the presence of synthesized in situ MoS2 particles.Ammonium paramolybdate $(NH_4)_6Mo_7O_{24}$*$4H_2O$ (APM) was used as a catalyst precursor. Before feeding into the reactor, the feed emulsion was prepared, with dispersion medium and dispersion phase presented by hydrocarbonaceous part of oil sludge, and a water solution of APM, respectively. APM loading with the water solution was 0.05 wt.% (calculated as Ìî) and 2 wt.% of water. Effectiveness of hydroconversion was assessed by the degree of conversion of raw material fraction boiling above 520°C (hereinafter 520°C+) and by deposition of coke on the walls of the reactor.

RESULTS

The results of studying main physico-chemical properties of oil reservoir sludge are presented in Table 1. The studied oil sludge contains considerable amounts of water (up to 36.5 wt.%). The highest amount of water was contained in the sludge from oil refinery No. 7. The density of the studied sludge is between 846 and 1,067 kg/m3.

According to the data about atmospheric and vacuum distillation of the studied sludge, it was found that reservoir sludge contained small amount of fraction IBP-180°C (up to 3.9 wt%. for oil sludge No. 6). With that, the content of the 180-350°C fraction ranges between 19.3 and 77.3 wt.%, and the content of the 350-520°C faction ranges between 9.9 and 50.5 wt.%. The content of heavy residues boiling above 520°C that the distilled mineral impurities and highmolecular oil components are reduced to is not more than 41.7 wt.% calculated as original oil sludge. Coking property of the studied samples does not exceed 7.8 wt.% and changes symbatically to the content

Table 1. Physico-chemical properties of oil sludge

Indicator name	Designation of oil sludge sample						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Density, kg/m3	934.7	907.1	930.0	914.8	846.2	896.0	1,067
Coking property, wt.%.	7.81	4.46	6.04	-	-	-	-
Mineral impurities, wt.%.	0.49	0.68	0.63	0.36	0.46	0.84	3.4
Ash content, wt.%	0.22	0.67	0.27	0.18	0.16	0.57	24.03
Sulfur content, wt.%	1.35	0.95	1.16	1.15	1.03	1.23	0.66
Water content, wt.%	9.7	9.9	17.4	4.8	traces	3.8	37.5
Fractional composition, wt.%							
Water	7.7	8.5	15.9	5.1	0	1.8	36.5
IBP-180°C fraction	0	0.7	0	0	1.1	3.9	3.5
180-350°C fraction	26.5	29.4	19.3	30.1	77.3	34.3	21.5
350-520°C fraction	24.2	41.4	34.5	40.6	9.9	50.5	18.7
520°C+ fraction	41.7	19.9	30.3	24.2	11.7	10.2	19.9

of 520°C+ heavy residues. The results of the structural-group analysis (Table 1-5) of 520°C+ residues shows that almost all resins and asphaltenes that act as coke precursors are concentrated in them.

The content of solid (mineral) impurities in the studied samples is in the range between 0.4 and 3.4 wt.%. Ash content reaches the highest value (24 wt.%) for the sample with the highest content of mineral impurities (Table 1), due to the concentration of the mineral part of the raw material in the ash residual.

Fractional composition of reservoir oil sludge after dehydration is shown in Table 2. The highest content of 520°C+ fractions is characteristic forthe oil sludge from oil traps No. 1, 3 of the cascade treating facilities of the oil refinery and the sludge from oil refinery No. 7. Thus, the organic part of the studied sludge samples after pretreatment with dehydration contains 10.3 to 46.1 wt.%of the 520°C+ heavy residue. By this indicator, the studied products can be classified as traditional petroleum feedstock that is suitable for processing using the existing oil refinery schemes.

The content of sulfur in the studied oil sludge samples varies between 1.0 and 1.4 wt%. Moreover, the prevailing part of sulfur-containing compounds is concentrated in the 520°C+ fractions.

Table 2. Fractional composition of organic part of oil sludge after dehydration

Fractional composition, wt.%	Designation of oil sludge sample						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
IBP-180°C fraction	0.0	0.8	0.0	0.0	1.1	3.9	5.6
180-350°C fraction	27.1	32.5	22.9	31.8	77.3	34.7	33.8
350-520°C fraction	26.8	45.6	41.1	42.8	9.8	51.1	29.4
520°C+ fraction	46.1	21.9	36.0	25.4	11.7	10.3	31.2
Total:	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Table 3. Group hydrocarbonaceous composition of oil sludge 180-350°C fractions

Contents of the hydrocarbons group	Designation of oil sludge sample						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Paraffinic hydrocarbons, wt.%.	26.5	27.4	27.5	20.7	35.4	31.7	30.6
Naphthenic hydrocarbons, wt.%.	43.2	42.1	41.3	39.7	34.4	33.5	43.3
including mono-naphtenic	10.4	11.6	11.4	7.6	16.8	10.4	14.0
bi-naphtenic	18.2	17.2	17.0	17.6	11.1	13.1	19.0
tri-naphtenic	10.1	11.0	11.3	12.0	6.6	8.6	8.3
tetra-naphtenic	4.5	2.4	1.6	2.5	0.0	1.5	2.1
Aromatic hydrocarbons, wt.%.	30.4	30.5	31.2	39.6	30.1	34.8	26.1

Table 4. Group hydrocarbonaceous composition of oil sludge 350-520°C fractions

Contents of the hydrocarbons group	Designation of oil sludge sample						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Paraffinic-naphthenic hydrocarbons, wt.%.	62.3	66.4	64.6	64.8	57.1	59.6	62.8
Aromatic hydrocarbons, wt.%.	30.7	28.0	30.6	28.8	34.7	35.9	32.0
including light aromatics	8.7	10.2	9.8	8.7	6.5	7.6	11.1
medium aromatics	7.3	6.3	7.4	7.0	7.1	5.6	6.3
heavy aromatics	14.7	11.5	13.4	13.0	21.1	22.7	14.6
Resins, wt.%.	7.0	5.6	4.8	6.5	8.2	4.5	5.3
including neutral resins	1.8	1.5	1.2	2.0	2.9	1.3	1.6
acid resins	5.2	4.1	3.6	4.5	5.3	3.2	3.7
Asphaltenes, % wt.	0	0	0	0.0	0.0	0.0	0.0

The study of hydrocarbon fractions included into the organic part of the sludge showed (Table 3–5) that the 180-350°C and 350-520°C fractions are characterized by high content of paraffinic-naphthenic hydrocarbons (over 60wt.%). The naphthenic hydrocarbons of the 180-350°C fraction are mainly represented by mono-, bi- and tri-naphtenic compounds. The 350-520°C fraction contains 4.5 to 8.2 wt.% of resins. It should be noted that processing these fractions is not a problem, since their hydrocarbonaceous composition is close to that of similar straight-run

Table 5. Group hydrocarbonaceous composition of oil sludge 520°C+ fractions

Contents of the hydrocarbons group	Designation of oil sludge sample						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Paraffinic-naphthenic hydrocarbons, %.	27.4	19.5	22.3	17.5	30.3	9.9	26.4
Aromatic hydrocarbons, %:	46.1	52.0	47.2	45.7	46.6	45.3	38.9
including:							
Light aromatics	9.6	9.5	7.3	8.4	9.1	6.2	8.2
Medium aromatics	6.9	8.3	6.3	5.4	5.4	4.4	4.9
Heavy aromatics	29.6	34.2	33.6	31.9	32.1	34.7	25.8
Resins, %	21.3	23.4	25.4	28.4	19.6	36.8	28.1
including:							
Neutral resins	8.2	9.6	11.3	10.3	6.5	11.9	9.7
Acid resins	13.1	13.8	14.1	18.1	13.1	24.9	18.4
Asphaltenes, %	5.3	5.1	5.1	8.4	3.5	8.0	6.6

Table 6. The elemental composition of the studied sludge samples (DL means detection limit)

Element	DL	Result, mg/kg Oil sludge						
		No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Al	0.380	66.0	90.0	84.0	46.0	75.0	463	15,168
As	0.135	0.840	0.560	0.780	0.700	<DL	0.240	5.40
Ba	0.211	38.0	619	35.0	30.0	11.0	27.0	1,777
Ca	0.125	83.0	199	81.0	84.0	331	377	1,843
Co	0.185	<DL	<DL	<DL	<DL	<DL	0.700	18.0
Cr	0.338	1.00	1.50	1.00	1.00	83.0	5.00	83.0
Cu	0.422	5.00	6.00	5.00	4.00	1.50	8.00	463
Fe	0.287	847	1,901	1,146	721	352	1,403	29,542
K	0.450	9.00	14.00	9.00	5.00	4.00	44.0	1,185
La	0.365	0.500	0.830	0.600	0.400	0.540	2.00	< DL
Mg	0.280	<DL	<DL	<DL	<DL	<DL	89.0	3,008
Mn	0.345	3.00	7.00	4.00	3.00	5.00	12.0	542
Mo	0.755	<DL	<DL	<DL	<DL	<DL	<DL	6.00
Na	0.350	3.00	6.00	3.00	<DL	3.00	7.00	200
Ni	0.445	29.0	13.0	25.0	22.0	3.00	15.0	100
P	0.407	46.0	365	48.0	43.0	40.0	35.0	922
Pb	0.460	9.00	10.0	9.00	8.00	<DL	3.00	62.0
Si	0.55	57.0	60.0	85.0	21.0	18.0	456	49,516
Sr	0.205	1.00	11.0	1.00	1.00	5.50	6.00	226
Ti	0.550	<DL	<DL	<DL	<DL	<DL	6.00	183
V	0.372	73.0	32.0	64.0	56.0	11.0	39.0	32.0
W	0.860	<DL	<DL	<DL	<DL	<DL	<DL	0.700
Zn	0.246	41.0	446	37.0	34.0	11.0	26.0	1,164
Zr	0.462	<DL	<DL	<DL	<DL	<DL	<DL	7.80

oil fractions, and can be processed in the existing hydroprocessing and cracking units.

The 520°C+ fraction contains mainly aromatic hydrocarbons, besides, resins and asphaltenes are present in their composition. Resinous substances contained in the 350-520°C and 520°C+ fractions mainly consist of acid resins. Sample No. 1 is characterized by the highest content of 520°C+ residue; for this reason, the sample has been selected for testing as hydroconversion raw material.

The results of elemental analysis of the studied oil sludge are shown in Table 6. The samples contain a wide variety of elements, mainly Fe, Ni, V, Al, Ba, Ca, Si, Zn. Iron has the highest concentration in all samples. Apparently, the prevailing part of solids in oil sludge is particles of iron oxide, i.e. rust, which is drawn from reservoirs with oil sludge[6].

Table 7. Properties of the heavy residue extracted from oil sludge by extraction and distillation

Indicator	Value
Density at 15°C, kg/m3°	997.4
Fractional composition, wt.%:	
IBP - 180°C	0
180 - 350°C	0
350 - 520°C	20
520°C+	80
Asphalt-resinous components, wt.%	19.0
Sulfur content, wt.%	3.2
Metals content, mg/kg:	
- nickel	52
- vanadium	158
Content of elements, wt.%:	
- carbon	84.8
- hydrogen	10.9
Mechanical impurities, wt.%	0.01

The boiling above 520°C residue of oil sludge has been selected for testing as hydroconversion raw material (sample No. 1), the properties of which are shown in Table 7. As it can be seen from Table 7, the content of fractions boiling up to 520°C was 20%; hydroconversion raw materials is characterized by high (26,6%) content of asphalt-resinous substances, with the yield of the residueof 46.1%calculated on the initial oil sludge. The experiments were performed at the temperature (T) of 430–450 °C, pressure (P) of 7.0 MPa, loading of 60-70 g. into the reactor, and hydrogen blowdown flow 18 nl/h. The results of hydroconversion are shown in Table 8. The data show that with increasing temperature, the conversion of the 520°C+ fraction increases, and the yield of distillate fractions and gas increases as well. When the reactor temperature is 450 °C, coke yield is the highest.

In the test conditions, the optimum hydroconversion temperature limited by the yield of polycondensation products (coke) was not more than 430 °C, and in this case the conversion degree was about 51%. Further increasing conversion up to 90% or more is ensured by recycling unconverted residue to the hydroconversion reactor[7].

DISCUSSION

The results of our study show that the ratio of water, hydrocarbonaceous and solid phases

Table 8. Results of hydrocarbon part of oil sludge hydroconversion (P=7.0 MPa, hydrogen flow - 18 nl/h, catalyst precursor–APM, content of Mo in the reaction medium - 0.05 wt.%, that of water - 2 wt.%).

Parameter	Value		
The reactor temperature, °C	430	440	450
Yield of hydroconversion products, wt.%			
Gaseous products	1.4	2.8	6.8
Hydrogenate	98.5	96.8	90.0
including factions: IBP-180°C	5.4	12.2	14.7
180-350°C	19.8	18.6	22.3
350-520°C	34.5	32.3	33.7
520°C+	38.9	33.7	19.3
Polycondensation products (coke)	0.1	0.4	3.2
Conversion of 520°C+ fraction, wt.%	51.3	57.9	75.9

in reservoir sludge varies within a wide range, which is consistent with the data[6, 8, 9]. The studied samples of reservoir sludge are complex dispersions consisting of various petroleum hydrocarbons, water, compounds of heavy metals and other mineral impurities that are suspended in the form of fine and coarse particles[10-12].

Selecting an efficient method of dehydration is a considerable aspect of the developed technologies for processing oil sludge. In laboratory conditions, the prevailing part of the water from the "crude" oil sludge can be removed by simple sedimentation and decantation of the organic phase. However, the product obtained this way still contains water emulsion stabilized by highmolecular components of oil and solids of mineral impurities [13].

On the industrial scale, installations for efficient dehydration of oil sludge require the use of more costly and energy-intensive methods, such as centrifugation, vacuum filtering or solvent extraction[8, 14, 15].

The content of elements in oil sludge of various origins varies in a wide range, and is, apparently, largely determined by the nature of the initial petroleum feedstock. According to[16, 17], metals in oil sludge are present in the form of thermally stable oil-soluble complex compounds (such as porphyrins and their derivatives). Refining the residual hydrocarbon part of oil sludge containing a lot of heavy metals, with the use of the existing catalytic processes of oil refining can be impossible, since the impurities introduced with the raw materials may act as catalyst poisons and may cause considerable corrosion of reaction equipment at oil refineries[18, 19].

In whole, the processes of transforming hydrocarbonaceous raw materials containing so many metals can be associated with considerable adverse effects on the environment. The problem of qualified sludge refining should be resolved taking into account the need of disposing of the transformation products containing considerable amounts of heavy metals.

The results of oil sludge pretreatment and testing its hydroconversion showed the possibility of effective utilization of oil sludge with the use of hydroconversion for processing high-boiling residues.

With regard to the properties of the studied sludge that is high density liquid petrochemicals with high content of organic compounds, water and low content of mechanical impurities[20, 21], and the results of hydroconversion, the scheme of processing oil sludge that includes the following stages seems to be the most promising one (Fig.1):

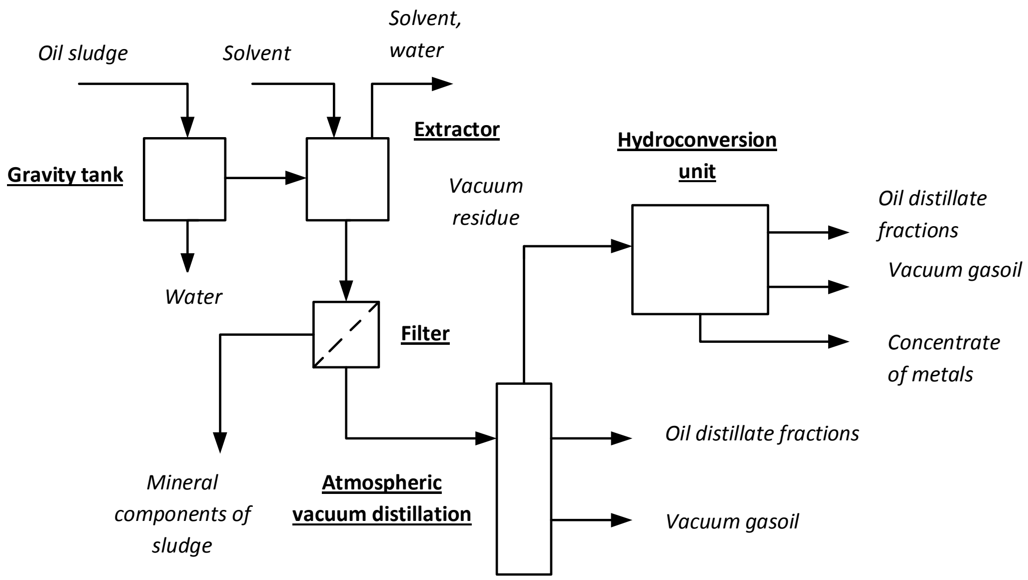

Fig. 1. Oil sludge processing scheme

1) oil sludge pretreatment for removing water and mechanical impurities;
2) atmospheric and vacuum oil sludge fractionation with obtaining heavy vacuum residue;
3) processing heavy vacuum residue using the technology of hydroconversion, which will make it possible to obtain additional light products and a concentrate of precious metals.

CONCLUSIONS

The disposing of the oil sludge produced during oil refining is a serious environmental and economic problem. Knowledge of physico-chemical properties of hydrocarbon sludge is very important for selecting an efficient method for its recovery. This article shows the results of researching physico-chemical properties of reservoir sludge of various origin (from the cascade treatment plants and the drainage system, oil depots), showing that the samples have characteristics that are typical for reservoir sludge, and are high density fluids with high content of hydrocarbons and water. Individual properties of sludge depend on properties of the original petroleum feedstock, conditions of oil sludge formation and storage. Content of heavy fractions boiling above 520 °C in the studied samples amounted to 41.7 wt.%, that of water–up to 37.5 wt.%, of solid (mineral) impurities–up to 3.4 wt%, the content of high-molecular components, in particular, asphaltenes and resins, was 8.4 and 36.8 wt.% calculated as the fraction boiling above 520 °C, respectively.

The paper proposes a scheme for processing reservoir sludge with known physico-chemical properties. As a result of hydroconversion of the selected oil sludge vacuum residue in the presence of catalyst (MoS2), high degree of conversion of fraction boiling above 520°C (wt.%) and high yield of distillate fractions were achieved. The obtained data are of considerable interest for further research for developing technologies of continuous processing of oil sludge of different origin with the use of hydroconversion.

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