

## Studies on Preprocessing of Reservoir Oil Sludges for Further Hydroconversion

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Results of the investigations demonstrate that for preprocessing and dewatering of reservoir oil sludge by solvent method on laboratory facility the most suitable solvent is toluene. Using calculations and experiments, the optimum parameters of solvent processing of oil sludge with extraction of organic components have been determined, which correspond to the temperature of 100°C, mixer rotation speed of 500 rpm, inert gas flow rate (N<sub>2</sub>) of 30 nl/h, water to toluene ratio of at least 1:5 by weight. Conditions of separation of mineral components of oil sludge by filtration have been determined: 45-50°C, slightly excessive pressure. A complex layout of oil sludge preprocessing has been proposed, including additional distillation of organic components of oil sludge at 300°C, facilitating obtaining of heavy fraction suitable for the use as initial raw material for hydroconversion.

**Key words:** Oil sludge, Waste products, Reservoir sludge, Solvent extraction, Toluene, Hydroconversion.

Production, transportation and processing of crude oil is always accompanied with the problem of oil wastes of various type and leads to significant environmental pollutions with huge amount of heavy residues, such as oil sludges of various types, vacuum residue, heavy residual fractions, bottom products, residues in oil storage reservoirs and so on<sup>1-5</sup>. Generation of such residues occurs both in industrial controlled processes, such as oil refining from water, treatment of oil containing effluents in treating facilities, during oil storage and transportation in various tanks as well as in emergencies with oil spillage.

Accumulation of oil residues leads to significant environmental pollution and involves

concentration of significant environmental damage (it is considered that about 1 t of sludge is generated per 500 t of oil, this value is given for developed countries). Disposal in sludge collectors, which are opened earth sites for sludge storage occupying vast surface areas, leads to alienation of agricultural lands and environmental pollution as a consequence of evaporation of petroleum products and their penetration into ground waters. Heavy aromatic hydrocarbons in the sludges are characterized with marked carcinogenic and mutagenic properties. The sludges and the wastes are highly resistant against decomposition in environment, their components can be distributed for significant distances, being accumulated in animals, plants, ground and water, thus destroying equilibrium of environmental systems, leading to death of animals and plants, making environment unfit for vital activity. Penetrating into human organism these compounds are accumulated in fat tissues, causing

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genetic mutations and teratosis of newborns. As a consequence, neutralization and disposal of oil sludges is a burning issue<sup>5,6</sup>.

The proposed in literature<sup>7-14</sup> methods of disposal of oil sludges in many cases do not provide the required level of environmental protection against secondary pollutions and do not permit to use efficiently these resources. Up-to-date approaches to reprocessing of oil wastes should envisage refusal not only of disposal (which cannot be applied from the point of view of environmental protection), but of combustion as well. Oil wastes should be directed for extraction of oil components from the wastes for further use in oil reprocessing. This should be reliably supported by researches on preprocessing of oil wastes with removal of water, solids and light hydrocarbons.

Our researches are aimed at determination of optimum conditions of solvent preprocessing of reservoir oil sludges with obtaining of feedstock suitable for processing by hydroconversion<sup>15</sup>. It is assumed that the developed approach would permit to reduce the level of environmental burden with simultaneous increase in efficiency of the use of hydrocarbon resources due to development of engineering principles of production of marketable petrochemicals by means of catalytic hydroconversion of oil wastes. Among the most important requirements to preprocessing of feedstock we considered minimization of water content and mineral impurities.

## EXPERIMENTAL

The experiments on preprocessing of heavy oil wastes were carried out with reservoir oil sludge, obtained from oil trap of treating cascade facilities of a Russian oil terminal. Physicochemical properties of the oil sludge are summarized in Table 1.

Sulfur content in a sample was determined according to GOST Standard 1437-75, and in distillate fractions – using Spectroscan-S energy dispersive X-ray fluorescent analyzer in accordance with GOST Standard R 51947-2002, ASTM D 4294-98.

Density of the samples was determined by pycnometric method according to GOST Standard 3900. The method is based on comparison of the

mass of oil product in predetermined volume with the mass of distilled water in the same volume and at the same temperature.

Coking property of the samples was determined according to GOST Standard 19932 (the Conradson method), based on determination of the mass of coke residue obtained at high-temperature heating and decomposition of the tested oil product.

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

Content of solid (mineral) impurities was determined using modified procedure of determination of mechanical admixtures (GOST Standard 6370-83). A sample of tested product was placed into a beaker and dissolved in preset amount of toluene. Then the solution was filtrated via moisture-proof smooth pre-dried filter for slow filtration (grade MN 1640 de, thickness: 0.17 mm, filtration rate: 140 ml/cm<sup>2</sup>•s, paper density: 85 g/m<sup>2</sup>, complete retention of particles with the sizes of  $e''$  2  $\mu$ m). After drying the filter together with the sample was weighed, the content of insoluble in toluene was determined.

Group composition of the petrochemicals was determined using the procedure of Petrochemical Institute, Bashkiria and a Gradient-M laboratory liquid chromatograph, intended for determination of group chemical composition of heavy oil residues and petrochemicals with boiling point above 300°C (bitumen, vacuum residue, cracking residue, asphaltite and so on). The method is based on the principles of solid-liquid chromatography with gradient replacement and separation into hydrocarbon groups.

Experimental studies of preprocessing of feedstock were performed on laboratory facility of preprocessing of heavy oil sludges. The facility layout is illustrated in Fig. 1.

The laboratory facility is composed of the extraction unit (tanks E-1 and E-2), separation unit (tanks E-3, E-4, F-1) and collector of final products (E-7, E-8).

A sample of oil wastes is placed into the extractor (E-2), which is a tank of autoclave type with electric heater, magnetic mixer and gas supply system. Predetermined amount of solvent is fed from the solvent tank (E-1) under slight excessive

pressure into the extractor, then the extractor is purged with inert gas in order to remove residual air from the system.

In order to improve mixing of the solvent with oil residue the option of solution purging with inert gas via outlets in the extractor bottom is provided. The gas flow rate into the extractor is controlled by gas flow regulator at the extractor inlet and gas counter near the separator (E-3).

The gas pressure on the system is controlled by upstream pressure controller.

The emitted from the extractor liquid (water, solvent, distillate fractions) is collected in the separator (E-3). The liquid composition depends on the extraction conditions.

When the feedstock contains more than 5wt % of mineral impurities, the solution with organic components of oil residues and mineral components of oil wastes, which are suspended in the solution, are located to the gravity tank (E-5), which is a glass calibrated vessel with electric heating. In the gravity tank the solution is separated into liquid and solvent insoluble solid phases. The main portion of liquid phase is separated from the residue by decantation and fed to the collector (E-7), and the residue with organic liquid components is fed to the extractor for further extraction.

The solution obtained after the further extraction is fed to the filter of hot filtration (F-1), where organic and mineral components of oil residue are finally separated. For more efficient filtration of the mixture slight excessive pressure is provided in the filter and at the filter outlet negative pressure is provided by means of vacuum pump, if necessary. The obtained filtrate is collected in the tank E-6, from where it is fed further to the tank E-7, combining with previously obtained solution of the solvent and dissolved organic components of oil residue. Since the used in the experiments feedstock contained less than 5wt % of mineral impurities, the decantation stage was excluded. Then the solvent is removed from the organic phase of oil wastes by rectification and from mineral components - by boiling-off.

The experiments on removal of water from oil sludge were performed until complete termination of water unloading from the separator. Unloading of the distillation products was carried out once per 15 minutes. The experiment on removal of light distillate fractions from dewatered residue of oil sludge was performed until complete termination of unloading of distillation product from the separator.

## RESULTS

Phase state of oil sludge components and solvent (toluene) were preliminary calculated at various temperatures and pressure of inert gas (nitrogen) and under various process variables of extraction. The calculations were performed using universal modeling system HYSYS. The following initial flow rates of components were applied (kg/h): water - 0.097, toluene - 1.0, nitrogen - 0.023, oil sludge - 1.0. Fractional composition of the initial oil sludge, summarized in Table 1, was also taken into account in the calculations. The calculated results of component distribution under various extraction conditions are summarized in Table 2. The data on component distribution in gaseous and liquid phases under various conditions demonstrated the optimum parameters of evaporation of water and toluene from the system are as follows: temperature of 100- 150°C and ambient pressure. After reaching of 100°C according to the calculated data water is

**Table 1:** Physicochemical properties of oil sludge

Property	Value
Density, kg/m <sup>3</sup>	934.7
Coking property, wt %	7.81
Mineral impurities, wt %	0.49
Sulfur content, wt %	1.35
Water content, wt %	9.74
Fractional composition, wt %	
Fraction BP -180°C	0
Fraction 180-350°C	24.4
Fraction 350-520°C	24.5
Fraction >520°C	41.7
Fractional composition w/o water, wt %	
Fraction BP -180°C	0.00
Fraction 180-350°C	27.1
Fraction 350-520°C	26.8
Fraction >520°C	46.2
Resin content, wt %	7.3
Asphaltene content, wt %	2.4

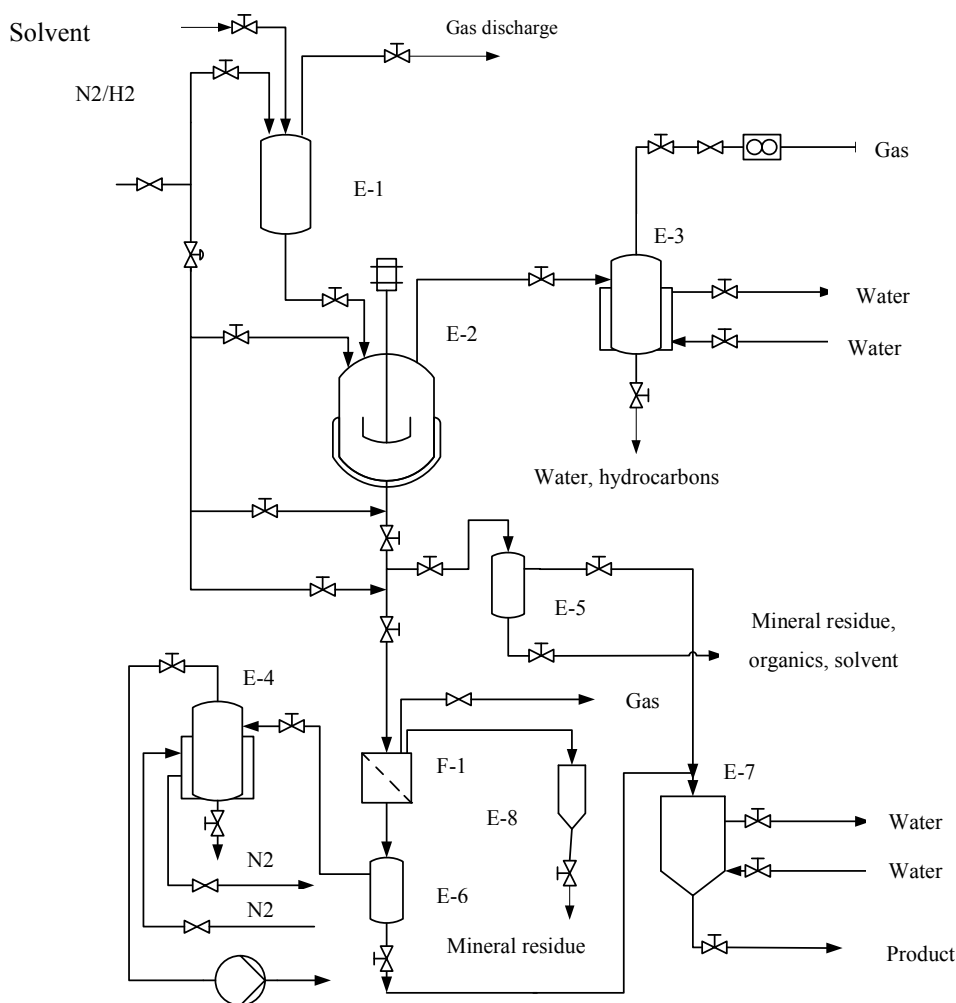
completely evaporated, and 150°C toluene is completely evaporated.

The aim of further experiments was to determine optimum parameters of preprocessing. In the experiments the initial toluene amount was based on its ratio to oil sludge of 1:1. Selection of such ratio was based on the information about water to toluene ratio during their boiling-off (azeotropic mixture) amounting to 1:5. Taking into consideration that the water content in oil sludge was ~9.7wt %, the minimum amount of toluene with respect to sludge should be 1:2. The experiments were performed with excess amount

of toluene. The conditions of the performed experiments are summarized in Table 3.

Initially the influence of temperature on the distillation of oil sludge was studied (experiments #1-3, Table 3). Using preliminary calculated data the following conditions were selected: temperature of 100 and 150°C, ambient pressure and pressure above the ambient value. Material balance of the experiments is summarized in Table 4.

After experiments at temperatures of 100°C, 150°C and ambient pressure, in order to decrease a chance of entrapment of 180-350°C



E-1 - solvent tank, E-2 - extractor, E-3 - separator, E-4 - vacuum pump trap, E-5 - gravity tank, F-1 - filter, E-6 - filtrate collector, E-7 - product collector, E-8 - collector of filtration residue

**Fig. 1.** Layout of laboratory unit for preprocessing of heavy oil sludges.

fraction into the distillation products, which is contained in organic components of oil sludge, the pressure in the system was increased to 0.4 MPa. Figs 2 and 3 illustrate the distillation diagrams of water and toluene at 150°C and 0.4 MPa.

Experimental results on searching for optimum content of solvent for water removal from the considered oil sludge (experiments #1, 4-6, Table 3) are summarized in Table 5. Distillation of water from extracted solution was studied for toluene content of 50%, 33.35% of total extracted

solution, as well as without solvent.

Further researches were focused on the study of influence of inert gas (nitrogen) flow rate and mixer rpm on distillation of water from oil sludge (experiments #1, 7-8, Table 3). The results are summarized in Table 6.

Heavy residues obtained after the experiments and containing organic and mineral components of oil sludge, as well as, in some cases, residues of toluene, were further separated into mineral and organic components. Since all obtained

**Table 2.** Distribution of water phases (gas/liquid) (in %) in extracting tank under various conditions

Pressure (abs), kPa	Temperature, °C					
	80	100	150	200	250	300
<b>Water</b>						
100	42.81/57.19	99.69/0.31	99.90/0.1	99.9/0.1	100/0	100/0
200	6.37/93.63	49.49/50.51	99.69/0.31	99.9/0.1	99.9/0.1	99.9/0.1
500	1.75/98.25	4.72/95.28	97.74/2.26	99.49/0.51	99.69/0.31	99.79/0.21
1000	0.72/99.28	1.75/98.25	27.52/72.48	97.33/2.67	99.08/0.92	99.49/0.51
1500	0.41/99.59	1.03/98.97	9.03/90.97	93.22/6.78	97.84/2.16	98.97/1.03
<b>Toluene</b>						
100	15.54/84.46	65.73/34.27	95.31/4.69	98.65/1.35	99.48/0.52	99.76/0.24
200	2.35/97.65	15.69/84.31	86.96/13.04	96.71/3.29	98.77/1.23	99.44/0.56
500	0.64/99.36	1.53/98.47	43.31/56.69	87.63/12.37	95.91/4.09	98.20/1.8
1000	0.028/99.72	0.58/99.42	6.91/93.09	60.55/39.45	88.75/11.25	95.42/4.58
1500	0.17/99.83	0.34/99.66	2.31/97.69	36.89/63.11	78.27/21.73	91.79/8.21
<b>Fraction 180-350°C</b>						
100	0.23/99.77	2.79/97.21	26.83/73.17	60.23/39.77	85.54/14.46	95.7/4.3
200	0.03/99.97	0.32/99.68	13.87/86.13	44.74/55.26	74.41/25.59	90.98/9.02
500	0.01/99.99	0.03/99.97	2.46/97.54	22.74/77.26	53.70/46.30	78.61/21.39
1000	0/100	0.01/99.99	0.28/99.72	8.0/92.0	34.78/65.22	63.62/36.38
1500	0/100	0/100	0.09/99.91	3.64/96.36	23.13/76.87	52.80/47.20
<b>Fraction 350-520°C</b>						
100	0/100	0/100	0.12/99.88	1.97/98.03	13.0/87.0	36.98/63.02
200	0/100	0/100	0.04/99.96	0.88/99.12	7.03/92.97	25.33/74.67
500	0/100	0/100	0.01/99.99	0.27/99.73	2.73/97.27	13.07/86.93
1000	0/100	0/100	0/100	0.08/99.92	1.24/98.76	7.21/92.79
1500	0/100	0/100	0/100	0.04/99.96	0.74/99.26	5.01/94.99
<b>Fraction &gt;520°C</b>						
100	0/100	0/100	0/100	0/100	0.02/99.98	0.31/99.69
200	0/100	0/100	0/100	0/100	0.01/99.99	0.15/99.85
500	0/100	0/100	0/100	0/100	0/100	0.06/99.94
1000	0/100	0/100	0/100	0/100	0/100	0.04/99.96
1500	0/100	0/100	0/100	0/100	0/100	0.03/99.97
<b>Nitrogen</b>						
100	99.58/0.42	100/0	100/0	100/0	100/0	100/0
200	97.89/2.11	99.58/0.42	100/0	100/0	100/0	100/0
500	92.83/7.17	94.51/5.49	99.58/0.42	100/0	100/0	100/0
1000	84.81/15.19	85.65/14.35	94.94/5.06	99.16/0.84	99.58/0.42	100/0
1500	76.37/23.63	76.79/23.21	85.23/14.77	97.89/2.11	99.16/0.84	99.58/0.42

residues contained not more than 3.5 wt % of insoluble in toluene compounds, mineral components were separated by filtration after preliminary dissolution of the residue in toluene, providing that toluene:(organic + mineral components) ratio equaled to 1:1. Filtration was carried out using hot filter of hot pressure with the pore size of filtering element of 1.5  $\mu\text{m}$  at various pressures and temperature of 45-50°C. As a consequence of the performed studies it has been established that filtration of the obtained samples is sufficient at slightly excessive pressure (0.4-0.6 MPa) in order to accelerate filtration. The obtained after filtration and separation of solvent samples contained mineral components (insoluble in toluene compounds) in amount of not higher than

0.024wt%.

Further experimental studies were performed in order to remove light distillate fractions and to obtain heavy fraction with its properties close to those of fuel oil and vacuum residue, processed by hydroconversion. The experimental conditions were selected on the basis of calculated data on boiling of oil sludge fractions from the reactor. Distillation was performed at 300°C (Table 3, experiment #9). The acquired results are summarized in Tables 7, 8. As can be seen, distillation at 300°C runs with complete removal of the fraction 180-350°C and accumulation of heavy residue of oil sludge (Table 8). It should be mentioned that in the distillation products slight increase in insoluble in toluene compounds (mineral

**Table 3.** Parameters of experiments on the influence of temperature and pressure of distillation of water and toluene from oil sludge

Experiment #	1	2	3	4	5	6	7	8	9
Temperature	100	150	150	100	100	100	100	100	100/300
Pressure, MPa	0.1	0.11	0.4	0.1	0.1	0.1	1.0	1.0	0.1
N <sub>2</sub> flow rate, nl/h	30	30	30	30	30	30	5	30	30
Mixer rpm	500	500	500	500	500	500	500	100	500
Experiment duration, h	0.75	0.75	2.0	1.0	1.75	0.75	1.75	2.5	3.0

**Table 4.** Material balance of experiments on the influence of temperature and pressure on distillation of water and toluene from oil sludge

Experiment #	1	2	3
Charged, wt %:			
Oil sludge	50	50	50
including: water	4.87	4.87	4.87
mineral components	0.25	0.25	0.25
organic components	44.88	44.88	44.88
Toluene	50	50	50
Total:	100	100	100
Obtained, wt %:			
Water	4.87	4.87	4.08
Toluene	41.3	50	17.1
Organic components of oil sludge	0	traces	0
Residue in reactor	53.83	45.13	78.82
including: organic components	44.88	44.88	44.88
mineral components	0.25	0.25	0.25
toluene	8.7	0	32.9
water	0	0	0.79
Total:	100	100	100

components) was observed, thus, it is undesirable to increase the distillation temperature above 300°C. The obtained residue was dissolved in toluene, filtered (in order to separate mineral components of oil sludge), and then the solvent was distilled. As a result, heavy residue was obtained suitable for experiments on hydroconversion.

## DISCUSSION

As can be seen in Table 1, the considered oil sludge is a complex disperse system composed of various petroleum hydrocarbons, water, solid mineral impurities, suspended in the form of fine and coarse particles. This agrees with the data in<sup>3, 16, 17</sup>.

It is known from the published data that in order to extract organic components from oil sludges various solvents are used, in particular, chloroform and toluene, as well as gasoline, diesel and gasoil fractions<sup>2-3, 18-20</sup>. Among them the most

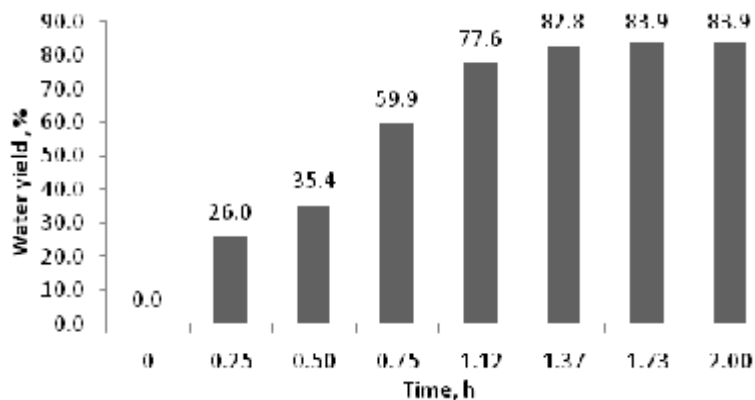


Fig. 2. Water yield under the conditions of Experiment #3

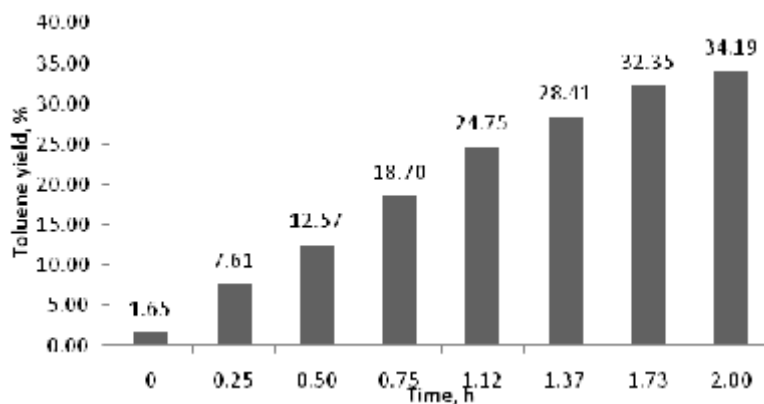


Fig. 3. Toluene yield under the conditions of Experiment #3

**Table 5.** Material balance of experiments on the influence solvent content on distillation of water from oil sludge

Experiment #	1	4	5	6
Charged, wt %:				
Oil sludge	50	66.65	100	33.35
including: Water	4.87	6.47	9.7	1.41
Mineral components	0.25	0.33	0.49	0.19
Organic components	44.88	59.85	89.81	33.75
Toluene	50	33.35	0	66.65
Total:	100	100	100	100
Obtained, wt %:				
Water	4.87	6.47	9.7	1.41
Toluene	41.3	29.08	0	49.2
Organic components of oil sludge	0	0	0.9	0
Residue in reactor	53.83	64.45	89.4	51.39
including: Organic components	44.88	59.85	88.91	33.75
Mineral components	0.25	0.33	0.49	0.19
Toluene	8.7	4.27	0	15.45
Water	0	0	0	0
Total:	100	100	100	100

efficient and available are chloroform and toluene. Taking into consideration the necessity of simultaneous extraction and removal of water from oil sludge on laboratory facility of preprocessing of heavy oil wastes, toluene was determined as the most optimum solvent in our works. In addition,

we considered the fact that toluene forms with water heterogeneous azeotropic mixture, the boiling point of which is about 83-85°C at the ratio of boiled water to toluene equaling to 1:5 according to calculations. Upon condensation of vapors of such mixture complete separation of its components

**Table 6.** Material balance of experiments on the influence of inert gas (nitrogen) flow rate and mixer rpm on distillation of water from oil sludge

Experiment #	1	7	8
Charged, wt %:			
Oil sludge	50	50	50
including:water	4.87	4.87	4.87
mineral components	0.25	0.25	0.25
organic components	44.88	44.88	44.88
Toluene	50	50	50
Total:	100	100	100
Obtained, wt %:			
Water	4.87	4.87	4.87
Toluene	41.3	23.85	34.5
Organic components of oil sludge	0	0	0
Residue in reactor	53.83	71.28	60.63
including:organic components	44.88	44.88	44.88
mineral components	0.25	0.25	0.25
toluene	8.7	26.15	15.5
water	0	0	0
Total:	100	100	100

**Table 7.** Material balance of experiments on removal of water and light distillate fractions from oil sludge

Experiment #	9
Charged, wt %:	
Oil sludge	50
including:Water	4.87
Mineral components	0.25
Organic components	44.88
Toluene	50
Total:	100
Obtained, wt %:	
Water	4.87
Toluene	50
Organic components of oil sludge	12.2
Residue in reactor	32.93
including:Organic components	30.91
Mineral components	0.72
Toluene	0
Water	0
Total:	100

occurs. The use of toluene would facilitate efficient extraction of organic components of oil sludge with rapid removal of water from the extracted solution of carried-over organic components of oil sludge in the separator as a consequence of acute boiling of water emulsified in oil sludge. In addition, liquid product, condensed in the separator and composed of emulsified water and toluene, will consist of two phases (water and toluene), which can be separated by simple settlement without the use of additional reagents and methods. It should be also mentioned

**Table 8.** Results of fractional distillation of heavy residue after removal of water and light distillate fractions from oil sludge

Fraction	Value, wt %
IBP - 180°C	0
180-350°C	0
350-520°C	37.2
> 520°C	62.8



that toluene, obtained from the separator after boiling and separation from water, can be used once again in subsequent experiments. Moreover, while selecting solvent for extraction we took into account that it should solute well hydrocarbon components of heavy oil wastes, forming solution capable to be separated readily from mineral components of the wastes by decantation and/or filtration. The solvent should be subsequently separated from hydrocarbon components of oil wastes or extracted into the product satisfying the requirements of further researches. The solvent to feedstock ratio should be optimum for the most efficient extraction of hydrocarbon components of the feedstock. Preliminary calculations (Table 2) demonstrated that the optimum conditions of boiling-off of water and toluene from the system are as follows: 100-150°C and ambient pressure. Herewith, after reaching 100°C, according to calculations, water is boiled-off completely, and at 150°C toluene is boiled off completely.

The article discusses the results of investigation into the influence of process variables, performed with the aim of selection of optimum conditions corresponding to the most efficient solvent aided extraction of hydrocarbon components and water. The experimental results (Table 4) demonstrate that at ambient pressure and temperature water is removed from the extraction area in 0.75 h. Toluene yield in this interval was 82.6 wt %. Since the experiment time interval was determined from the instant of achievement of preset process variables, and distillation can initiate before this instant, then at 0 h of experiment significant distillate yield is already observed. Increase in the distillation temperature to 150°C leads to increase in initial rate of boiling-off of water and toluene, however, total time of water removal from oil sludge remained the same (Figs. 2, 3). Toluene under these conditions is also boiled-off completely. It should be noted that by means of chromatographic analysis it has been established that toluene contains traces of the fraction 180-350°C of organic components of oil sludge, which is unacceptable from the point of view of these experiments. As can be seen, pressure increase significantly hinders water boiling-off from oil sludge and even double increase in distillation time does not lead to complete water removal from the

considered feedstock. Thus, from the performed investigations it follows that optimum extraction conditions are as follows: 100°C and ambient pressure.

At the same time, decrease in amount of the applied solvent (Table 5) leads to increase in time of water removal from extracted solution. Without toluene (experiment #4) the time of water boiling-off was comparable with the experimental results with 50 wt % of toluene, however, water, removed from the reaction area, contained minor amount of organic components of oil sludge. Occurrence of organic components of oil sludge in stripped water can be attributed to carry-over of a portion of extracted solution due to severe boiling of water in the reaction area. Increase in toluene amount did not lead to significant variations in water distillation.

It appeared that the influence of inert gas flow rate, studied in experiments #7, 8, has reverse influence on the duration of water removal. Decrease in the values of considered parameter lead to sharp increase in the time of water removal from oil sludge, herewith, increase in nitrogen flow rate via liquid phase leads to carry-over of organic components of oil sludge together with gaseous products and accumulation in separator. Increase in the mixer rpm under the experimental conditions was impossible, since 500 rpm was the maximum value for the applied equipment.

Therefore, on the basis of performed investigations the optimum conditions were determined for preprocessing of the used reservoir oil sludge with extraction of organic components, suitable for further reprocessing. Solvent: toluene, solvent to oil sludge ratio: 1:1 wt. Solvent, removed from the distillation area, can be used for extraction once again.

The investigations into filtration of preprocessed oil sludge samples demonstrated that it would be reasonable to perform filtration at slight excessive pressure (0.4-0.6 MPa). At the same time, the content of solid mineral components (insoluble in toluene compounds) did not exceed 0.024 wt %.

The results of distillation of organic components of oil sludge at 300°C demonstrated that under these conditions the fraction 180-350°C is removed completely from heavy components of oil sludge. As a consequence, it is possible to

obtain heavy residue containing more than 60 wt % of fraction with boiling point of  $> 520^{\circ}\text{C}$ , which is important for subsequent processing of this feedstock using hydroconversion<sup>15</sup>. Increase in the distillation temperature above  $300^{\circ}\text{C}$  is undesirable, since it can lead to generation of additional amount of high molecular components in the feedstock, which are coke precursors.

Therefore, as an alternative of preprocessing of heavy oil wastes we consider the following variant: 1) solvent preprocessing with removal of water and toluene from oil sludge; 2) distillation of organic components of oil sludge with separation of light distillate fractions; 3) filtration of heavy residue, preliminary dissolved in toluene, with extraction of mineral components and subsequent removal of solvent.

### CONCLUSIONS

The presented in the work results demonstrate that for preprocessing of reservoir oil sludge and dewatering by solvent method the most suitable solvent is toluene. Using calculations and experiments, the optimum parameters of solvent processing of oil sludge with extraction of organic components have been determined, which correspond to the temperature of  $100^{\circ}\text{C}$ , mixer rotation speed of 500 rpm, inert gas flow rate ( $\text{N}_2$ ) of 30 nl/h, water to toluene ratio of at least 1:5 by weight. Separation of mineral components of oil sludge by filtration is performed at  $45\text{-}50^{\circ}\text{C}$ , at slight excessive pressure. A complex layout of oil sludge preprocessing have been proposed, including additional distillation of organic components of oil sludge at  $300^{\circ}\text{C}$ , facilitating obtaining of heavy fraction suitable for the use as initial raw material for hydroconversion.

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### REFERENCES

1. E. R. Shperber. Some types of wastes of oil refineries and their classification // *Environmental protection in petroleum industry*, 2011; **2**: PP.27-32.
2. Hu, G., Li, J., & Zeng, G. Recent development in the treatment of oily sludge from petroleum industry: a review. *Journal of hazardous materials*, 2013; **261**: 470-490.
3. M. Elektorowicz, S. Habibi, Sustainable waste management: recovery of fuels from petroleum sludge, *Can. J. Civil. Eng.*, 2005; **32**: 164-169.
4. K. Shailubhai. Treatment of petroleum industry oil sludge in soil // *Trends in biotechnology*, 1986.
5. Conversion of oil refinery wastes (oil sludges) / *Oil and Gas Journal*, 1991; **89**, # 1: 73-77.
6. Gron' V. A., Korostovenko V. V., Shakhrai S. G., et al. Problem of generation, reprocessing and disposal of oil sludges. *Advances of modern natural science*. 2013; **9**: 159-162.
7. Shlepkin Yu. Analysis of disposal methods of oil sludges. Advantages and disadvantages // *Environmental protection in gas and petroleum industry*, 2009; **12**: 32-34.
8. Velghe, I.; Carleer, R.; Yperman, J.; Schreurs, S. Study of the pyrolysis of sludge and sludge/disposal filter cake mix for the production of value added products. *Bioresource Technology*, 2013; **134**, 1.
9. Shie, J.; Lin, J.; Chang, C.; Wu, C.; Lee, D.; Chang, C.; Chen, Y. Oxidative Thermal Treatment of Oil Sludge at Low Heating Rates. *Energy & Fuels*, 2004; **18**: 1272
10. Zheng, C.; Wang, M.; Wang, Y.; Huang, Z. Optimization of biosurfactant-mediated oil extraction from oil sludge. *Bioresource Technology*, 2012; **110**: 338.
11. Je-Lueng Shie, Jyh-Ping Lin, Ching-Yuan Chang, Shin-Min Shih, Duu-Jong Lee, Chao-Hsiung Wu. Pyrolysis of oil sludge with additives of catalytic solid wastes. *Journal of Analytical and Applied Pyrolysis*, 2004; **71**(2): 695-70.
12. Rocha, O., Dantas, R., Duarte, M., Duarte, M., & Silva, V. Oil sludge treatment by photocatalysis applying black and white light. *Chemical Engineering Journal*, 2010; **157**: 80-85.
13. Xu, N., Wang, W., Han, P., & Lu, X. Effects of ultrasound on oily sludge deoiling. *Journal of Hazardous Materials*, 2009; **171**: 914-917.
14. Roldán-Carrillo, T., Castorena-Cortés, G., Zapata-Peñasco, I., Reyes-Avila, J., & Olguín-Lora, P. Aerobic biodegradation of sludge with

- high hydrocarbon content generated by a Mexican natural gas processing facility. *Journal of Environmental Management*, 2012; **95**: 93-98.
15. Kadiev, Kh., Dandaev, A., Gyl' Maliev, A., Batov, A., & Khadzhiev, S. Hydroconversion of polyethylene and tire rubber in a mixture with heavy oil residues. *Solid Fuel Chem. Solid Fuel Chemistry*, 2013; **47**(2): 132-138.
  16. M.J. Ayotamuno, R.N. Okparanma, E.K. Nweneka, S.O.T. Ogaji, S.D. Probert, Bio-remediation of a sludge containing hydrocarbons, *Appl. Energ.*, 2007; **84**: 936-943;
  17. M.H. Greg, A.H. Robert, D. Zdenek, Paraffinic sludge reduction in crude oil storage tanks through the use of shearing and resuspension, *Acta Montan. Slovaca*, 2004; **9**: 184-188
  18. Taiwo, E. A. and Otolorin, J. A. Oil Recovery from Petroleum Sludge by Solvent Extraction', *Petroleum Science and Technology*, 2009; **27**: 8,836 — 844;
  19. Zubaidy, E., & Abouelnasr, D. Fuel recovery from waste oily sludge using solvent extraction. *Process Safety and Environmental Protection*, 2010; **88**: 318-326.;
  20. Ahmed, C. M. Sinnathambi, U. Eldmerdash, "N-Hexane, Methyl Ethyl Ketone and Chloroform Solvents for Oil Recovery from Refinery Waste", *Applied Mechanics and Materials*, 2014; **699**: 666-671.