

SELECTION OF COLLECTOR COMPOSITION AND TEMPERATURE CONDITIONS FOR DIAMOND FOAM SEPARATION

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Abstract

Quantitative and qualitative regularities of a collector low and high molecular weight fractions distribution between solid and liquid phases in foam separation process were determined with the use of extraction-spectrophotometrical technique. The feasibility of producing a collector with optimal ratio of fractions of light distillates, hydrocarbon oils, resin, and asphaltenes by mixing M-40 fuel oil with diesel fuel oil in specified ratios was substantiated. It was shown that applying compound collectors with M-40 fuel oil weight percentage of 60-70 enabled increasing diamond recovery by 2.7-3.5%. Similar increase was also achieved when using F-5 bunker fuel oil diluted by 10-18% with diesel oil fraction. The optimal temperature of 24 °C for initial ore feed conditioning with flotation agents and foam separation providing maximum diamond recovery into concentrate and high selectivity was determined. The proposed collectors and temperature conditions enabled increasing diamond recovery.

Key words: Diamonds; Foam separation; Extraction; UV spectroscopy; IR spectroscopy; Compound collectors; Fuel oil; Diesel fuel oil; Medium temperature.

1. Introduction

In the process of foam separation, being the main method for extracting small diamonds, various oil products are used as collectors, including oil, F-5 bunker fuel oil, and water-oil emulsions [1, 2]. Changing the range of oil products used as collector and fluctuations of flotation pulp temperature often lead to deterioration of foam separation performance [3]. This is due to insufficiently stable fixation of an apolar collector on a diamond surface. Increasing the stability of collector fixation on a diamond surface is provided by applying collectors of optimal fractional composition and selection of optimal temperature conditions for conditioning and foam separation [4]. To increase efficiency of diamond-bearing kimberlite foam separation, properties and optimal component composition of collectors were studied in this research, and the temperature conditions of foam separation feed conditioning with flotation agents were selected.

2. Testing approach

The share of collector fixed on a diamond surface was selected as a criterion of the apolar collector fixation stability. Extraction-spectrophotometric technique [5] was used to measure the collector distribution between products and phases in flotation tests.

In accordance with the extraction-spectrophotometric technique, after flotation testing, the obtained diamond concentrate was dried and treated for 4 minutes in a closed flask with the extractant, carbon tetrachloride (CCl₄). The obtained extract was transferred to the PE-5400 UV spectrophotometer's cuvette, and its luminescence emission absorption was measured at wavelength (λ) of 315 nm. At this wavelength the signal value was directly proportional to the concentration of adhesive-active fractions of an oil product.

The technique included extraction of the reagent from the surface of solid phase and from aqueous phase of the flotation pulp by organic solvents and measuring

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the extracted substance amount. To determine the fractional composition of the collector fixed on the diamonds, the IR spectrophotometry was used both directly for the diamond crystals and for the samples of organic compounds, components of the collector under study, extracted from the extractants [6]. In both cases the FT-IR spectroscopy method was used.

To study the oil products structure, the optical microscopy method was used [7]. Images of a thin layer of the oil products were produced using Micromed-3-LUM microscope at combined lighting conditions. A peculiarity of the technique used was the possibility to diagnose the presence of resins (tarry matter) and asphaltenes dissolved in oil products (in UV range) and occurring as solid matter (in visible range).

Verification of collecting properties of the studied oil products and their mixtures was carried out using a Hallimond tube, a non-frothing flotation unit. Semi-industrial tests were carried out at the foam separation unit of "Yakutniproalmaz" Institute. Specific conditions of the tests are given in the relevant sections of the paper.

3. Research Findings and Discussion

According to the selected testing scheme, the research of the collector distribution between the solid and liquid phases of the flotation system was carried out immediately after flotation (Fig. 1).

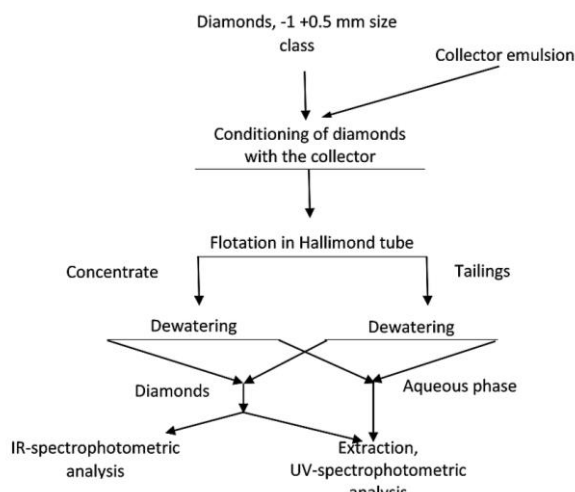


Figure 1 Arrangement of tests for studying diamond flotation and collector distribution

To determine the amount and fractional composition of the fixed compound collector, UV-spectra of the

extract from the diamond surface and IR-spectra of diamond crystals were measured. The spectral studies showed that when low-boiling oil products (kerosene, diesel fuel oil, etc.) were used as a collector, they were poorly fixed on a diamond surface. The percentage of the collector (diesel fuel) fixed on the surface of diamonds, as measured by UV-spectroscopy at 315 nm wavelength, was 34% (Table 1). After extraction with carbon tetrachloride or toluene no appreciable amounts of any organic substances were detected on the surface of diamonds by infrared spectroscopy.

Table 1 Amount of fixed collector and diamond recovery into concentrate at different hydrophobization conditions

No.	Diamond hydrophobization conditions	Share of fixed collector, %	Diamond recovery into concentrate, %
1	Diesel fraction treatment	34	46.5
2	F-5 bunker fuel oil treatment	61	78.6
3	F-5 bunker fuel oil treatment and extraction of the collector with toluene	5	26.2
4	Test 3 with subsequent diesel fuel oil treatment	60	72.2

When using oil products containing significant portions of medium- and high-molecular-weight hydrocarbons (e.g., fuel oil), 45-61% of the fed collector mass are fixed on the surface of diamonds. The IR spectrum of the treated diamonds contains characteristic absorption peaks of almost all fractions of the used oil product (Fig. 2a). After extraction with carbon tetrachloride, high-molecular-weight resinous substances and asphaltenes remain on the surface of diamonds. They were detected based on characteristic absorption peaks at wave numbers of 1.617, 1.462 and 1.377 cm^{-1} (Fig. 2b) [8]. Resinous substances and asphaltenes were not removed from the surface of diamonds even when strong organic solvents such as toluene were used as extractants.

To clarify the performance of high-molecular oil fractions, firmly fixed on the surface of diamonds, flotation tests with summer diesel fuel oil and F-5 bunker fuel oil were conducted. The flotation tests were carried out using a Hallimond tube, a non-frothing flotation unit. In the tests, a 200 mg weight of

diamonds of -1+0.5 mm fineness was used. The flotation test duration was 4 min, the air consumption was 50 ml. For diamond agitation, the collector

was used as an emulsion prepared by dispersion of 8 mg of the collector in 40 ml of aqueous phase (200 mg/l).

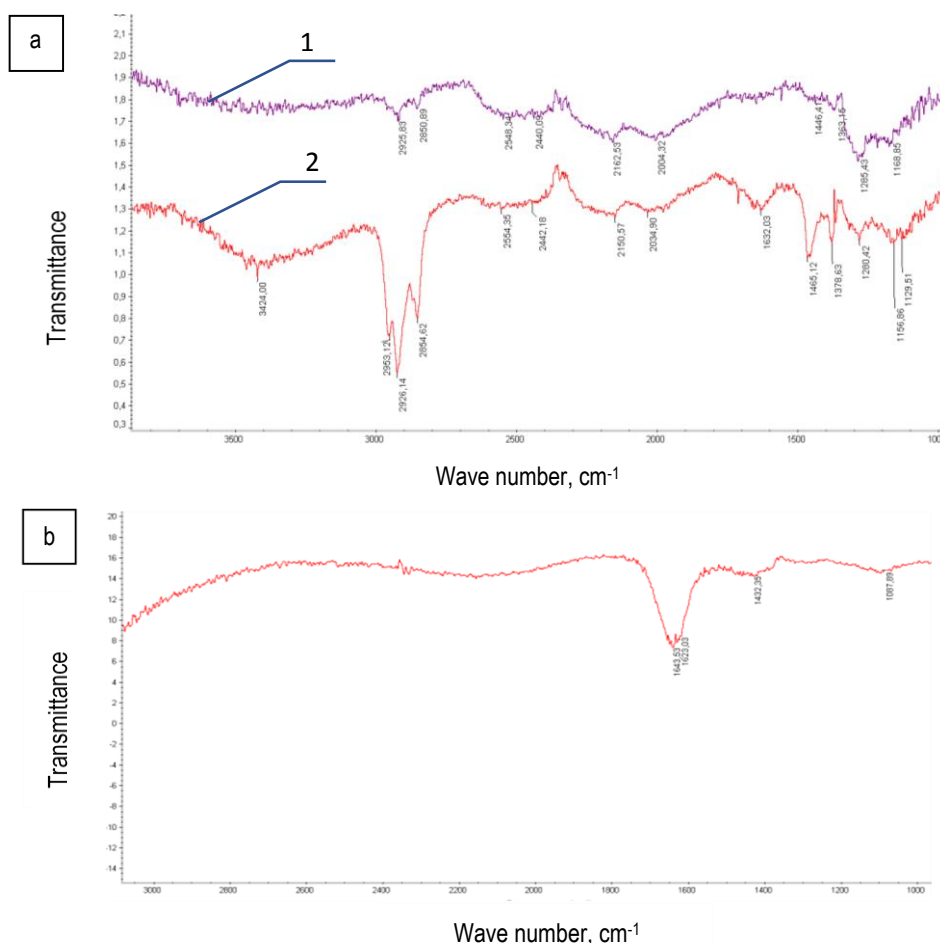


Figure 2 IR spectra of diamonds before (a1) and after (a2) treatment with collector and of diamond surfaces after extraction of collector with toluene (b)

The tests showed that using diesel fuel oil at the concentration of 200 mg/l provided the extraction of diamonds into the concentrate of 46.5%. When fuel oil was used as the collector, the recovery of diamonds increased significantly (Table 1, test 2). The extraction of the collector with toluene led to sharp decrease of floatability (test 3). The recovery of diamonds pretreated with fuel oil, extracted with toluene, and treated with diesel fuel oil amounted to 72.2% (Table 1, test 4). The test results confirmed that the presence of high-molecular weight fractions on the surface of diamonds increased the flotation performance. The difference in

floatability of diamonds after extraction without additives and with diesel fuel oil (Table 1, tests 3, 4) showed the importance of using low and medium molecular weight fractions, probably, as the phase promoting effective adhesion of diamonds to air bubbles [9].

The results obtained gave grounds to state that the best conditions for the fixation of collector on the surface of diamonds required the presence of both low-molecular and high-molecular hydrocarbon fractions in the collector composition. The spectral study data were in good agreement with the historical data of the process studies, which showed that the highest diamond

recovery into the foam separation concentrate was achieved at weight fraction of light distillates (LD) of 35-48%, hydrocarbon oils (HO) of 32-40%, resins and asphaltenes (RA) of 17-24% [10]. The area of oil fractions optimum proportions (in terms of mass fractions) in a compound collector is illustrated by the ternary diagram in Fig. 3, taken from [10].

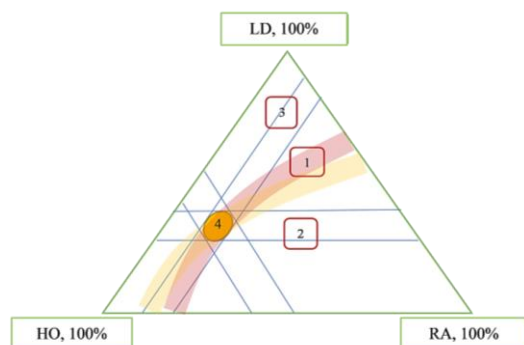


Figure 3 Diagram of three-fraction compound collector composition: LD — light distillates; HO — hydrocarbon oils; RA - resins and asphaltenes. 1 - colloidal solution of RA in LD and HO; 2 - dispersion of RA in LD and HO; 3 - true solution of RA in LD and HO; 4 - area of optimal ratio of fractions

To find the regularities of forming the oil products structure at various phase composition and then select the composition of collectors with increased collecting ability, oil products were studied using the method of combined optical microscopy. A feature of the method used is the ability to detect the presence of crystalline and colloidal forms of hardly soluble components in oil products, such as asphaltene fraction.

The oil structure analysis showed that adding hydrocarbon oil fraction and light distillates to fuel oil led to decreasing number of large crystals or agglomerates of asphaltenes and resins due to their transition into colloidal form and the subsequent dissolution [11, 12]. The specified changes in the structure of a compound collector of variable fractional composition are reflected in the diagram in Fig. 3. Such transformations can be the reason of changing collecting ability of oil products used in flotation of diamonds. In further researches we determined the regularities of behavior of high-molecular oil fractions at changing the ratio of low- and high-molecular fractions in the oil products used in flotation of diamonds.

Optical microscopy study using Micromed-3-LUM microscope at combined lighting conditions showed that

resins and asphaltenes were present in the disperse state (black dots and aggregates) and in the form of solution (the areas with yellow-green light).

Analysis of the images showed that the addition of 10% diesel oil fraction did not lead to significant dissolution of asphaltenes (Fig. 4a, b). When adding 20 and 30%, the aggregates of asphaltene crystals disintegrated, dispersed, and dissolved with the formation of fine-dispersed and colloidal forms (Fig. 4c, d).

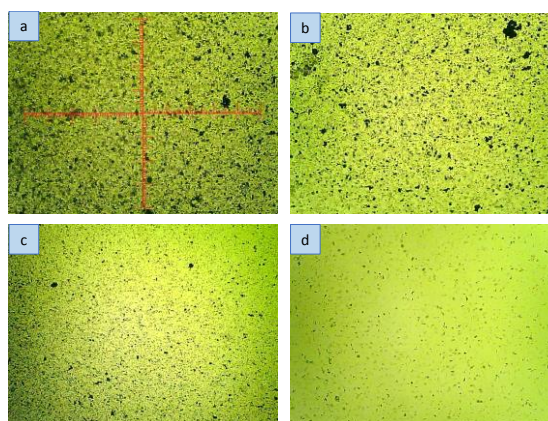


Figure 4 Images of a thin layer of M-40 fuel oil at combined lighting conditions at Micromed-3-LUM microscope before (a) and after dilution with diesel oil fraction: 10% (b); 20% (c), 30% (d)

Dilution of fuel oil by 30% (70% of fuel oil, 30% of diesel fuel oil) ensures the transition of the roughly dispersed structure into the colloidal or molecular solution of resins and asphaltenes (Fig. 4d). In this area the compound collector is characterized by optimal structure (colloids of resins and asphaltenes in the mixture of light distillates and hydrocarbon oils), ensuring manifestation of its best processing behavior.

Fuel oils used in flotation of diamonds are not optimal collectors (in their composition), and it is reasonable to modify their composition and properties to improve their foam separation performance. Based on the findings of physicochemical investigations, to increase collecting ability of M-40 and F-5 fuel oils, it was suggested to change state of the resin-asphaltene fraction from coarse-dispersed into colloidal or molecular solution by adding light distillates (Table 2). At the corresponding dilution of M-40 fuel oil (to 30-40% of diesel fuel oil, Table 2) or F-5 bunker fuel oil (to 10-20% of diesel fraction, Table 2), the collectors with the fractional composition justified in [10] were obtained.

Table 2 Composition and characteristics of collectors prepared based on M-40 fuel oil and diesel fuel oil, F-5 fuel oil and diesel oil fraction (DF)

Collector composition		Density, g/cm ³	Dynamic viscosity at 50 °C, mPas	T _{pour point} , °C	Weight percentage of fractions			Diamond recovery, %
M-40	Diesel fuel oil				LD	HO	RA	
100	0	1.053	135.6	+12	10.3	55.5	34.2	68.1
90	10	0.981	128.3	+5.3	17.6	51.6	30.8	70.2
80	20	0.956	71.7	-1.0	24.9	47.7	27.4	74.5
70	30	0.945	36.7	-5.2	32.2	43.8	24.0	84.5
60	40	0.935	23.6	-10.1	39.5	39.9	20.6	83.5
50	50	0.931	19.2	-17.2	46.8	36.0	17.2	78.4
40	60	0.914	14.2	-24.4	54.2	32.1	13.7	73.0
F-5	DF							
100	0	0.948	36.2	-6.0	31.5	40.9	27.6	80.5
90	10	0.939	24.1	-12.3	36.7	38.46	24.8	82.0
85	15	0.933	15.5	-15.6	39.3	37.2	23.5	84.7
80	20	0.929	12.5	-18.8	41.9	36.0	22.1	82.3

LD — light distillates; HO — hydrocarbon oils; RA - resins and asphaltenes. Collectors with optimal fractional composition are highlighted in gray.

Consideration of M-40 and F-5 fuel oils mix as the basic collector was due to the emerging task of replacing F-5 fuel oil by the less scarce M-40. The selection of diesel fuel oil and diesel fraction as a modifier was caused by organizational factors only. No significant differences in their effect on fuel oil dilution were found.

Verification of collecting properties of the initial and diluted fuel oils was carried out using a Hallimond tube, a non-frothing flotation unit. In the flotation tests, a 200 mg weight of diamonds of -1+0.5 mm fineness was used. The flotation test duration was 4 min; the air consumption was 50 ml. For diamond agitation, the collector was used as an emulsion prepared by dispersion of 4.0 mg of the collector in 40 ml of aqueous phase.

The flotation tests showed that the highest diamond recovery was achieved just with the recommended fractional composition of the collector, namely, at the weight percentage of M-40 fuel oil (in the mixture with diesel fuel oil) of 60-70 and the weight percentage of F-5 fuel oil (in the mixture with diesel fraction) of 80-90 (Table 2). Comparison of the data on the fractional composition and diamond extraction (Table 2) allowed concluding that the best flotation performance was reached at the recommended ratios of the basic collector fractions.

An important parameter of foam separation is medium temperature in the initial feed conditioning with flotation agents and in foam separation process [13, 14]. Depending on the climatic conditions and peculiarities of the applied process mode, the ambient temperature in these operations varied significantly [15]. The temperature fluctuations resulted in decreasing diamond recovery and increasing reagent consumption. To determine optimal temperature conditions, Hallimond tube flotation tests were carried out at the conditioning and flotation temperatures of 10, 14 and 24 °C. The selected temperature interval corresponds to foam separation conditions at industrial plants at different seasons. In the tests, F-5 bunker fuel oil (produced by "Bologonefteprodukt" LLC) and its compositions with diesel fraction were used.

In the tests, the best results were achieved at temperature of 14 and 24 °C. At 14 °C, the best results were obtained for F-10 and F-14 diluted fuel oils obtained by diluting F-5 bunker fuel oil with 10 and 14% of diesel fraction (diamond recovery of 78.4% and 77.9%, respectively). This was 3.8-4.5% higher than for the best basic collector, F-5 bunker fuel oil (Table 3).

Recovery of diamonds into the concentrate in non-frothing flotation process at temperature of 14-24 °C with the collector-reagents based on F-5 bunker fuel oil exceeded the recovery at 10 °C by 3.3-9.9% (Table 3).

Table 3 Recovery of diamonds in flotation with F-5 bunker fuel oil and its compositions with diesel oil fraction as collectors at different temperatures

Collector	Diamond recovery into concentrate, %		
	10 °C	14 °C	24 °C
F-5 bunker oil	70.6	74.6	80.5
Diluted F-10 bunker oil (10% of diesel oil fraction)	75.4	78.4	84.2
Diluted F-14 bunker oil (14% of diesel oil fraction)	74.3	77.9	83.2
Diluted F-18 bunker oil (18% of diesel oil fraction)	72.0	75.3	81.5

The obtained results were tested at the foam separation unit (Figure 3) operating in a near-industrial mode (collector consumption of 1000 g/t, butyl aerofloat consumption of 50 g/t, frothing reagent consumption of 150 g/t). This unit (Figure 5) simulates the actual process technology very adequately.

During the tests the recycled water temperature was regulated by its cooling or heating. As the performance criterion, the selectivity index calculated as a function of diamond recovery (ϵ) and kimberlite yield into the concentrate (γ) was used:

$$S = \epsilon - 1.3 \gamma \quad (1)$$

The index value at $\gamma = 1.3$ was selected as the ratio of increasing costs for aftertreatment of the concentrate with increasing the concentrate dilution to the cost of additionally recovered diamonds.

The bench-tests results showed that using the compound collectors based on F-5 bunker fuel oil at 24 °C provided diamond recovery of 80-83.9% with high selectivity ($S = 76.1-80.1\%$, Table 4). Diamond recovery at 24 °C was 1.8-7.7% higher than in the basic mode in the temperature interval of 10-14 °C.

**Figure 5** Automated device for foam separation of diamond-containing kimberlites**Table 4** Key process parameters of foam separation at optimal conditions

No.	Collector used	Temperature, °C	Diamond recovery into concentrate, %	Kimberlite yield into concentrate, %	Selectivity, %
1	F-5 bunker fuel oil	10	74.0	1.5	72.05
2	F-5 bunker fuel oil	14	78.2	1.6	76.12
3	F-5 bunker fuel oil	24	79.4	1.7	77.19
4	F-5 bunker fuel oil	28	80.5	4.0	75.19
5	F-10 collector (10 % of diesel fuel)	24	81.7	1.9	79.23
6	F-14 collector (14 % of diesel fuel)	24	83.9	2.9	80.13
7	F-14 collector (14 % of diesel fuel)	28	85.4	5.7	78.0
8	F-18 collector (18 % of diesel fuel)	24	80.0	2.1	77.27
9	F-18 collector (18 % of diesel fuel)	28	81.5	3.9	76.43

Further increasing the temperature to 28 °C increased diamond recovery by 1.5%, but this also increased the yield of kimberlite minerals into the concentrate and decreased the process selectivity to 78-79.5%. Decreasing the temperature to 10 °C led to decreasing the recovery by 5.5-7%.

Based on the findings of studying temperature effect on the foam separation performance we recommend using heating recycled water and collector to maintain the conditioning and foam separation temperature at 24 °C. In accordance with the recommendations made, the regulations for recycled water supply in the foam separation cycle at Processing Plant No. 3 of the Mirninsky GOK have been developed and adopted for industrial development.

4. Conclusion

The regularities of a collector low and high molecular weight fractions distribution between solid and liquid phases in foam separation process were determined using extraction-spectrophotometrical technique. Besides, the importance of fixation of resin and asphaltene fractions on the surface of diamonds was established. The proportion of M-40 fuel oil (60-70%) and diesel fuel oil (30-40%), as well as those of F-5 fuel oil (82-90%) and diesel fraction (10-18%), providing the optimal collector fractional composition were determined. It was shown that the selected ratios of fuel oil and diesel fractions enable maximizing diamond recovery into concentrate. The optimal temperature of 24 °C for the initial ore feed conditioning and foam separation providing high diamond recovery into the concentrate with the required selectivity was determined. Applying compound collectors of optimal fractional composition based on F-5 bunker fuel oil and diesel fraction at the selected temperature conditions enabled increasing diamond recovery with high selectivity. This allowed recommending heating recycled water and reagents in foam separation circuit.

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IZBOR SASTAVA KOLEKTORA I TEMPERATURNIH USLOVA ZA SEPARACIJU DIJAMANATA PROCESOM FLOTIRANJA

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Izvod

Ekstrakciono-spektrofotometrijskim ispitivanjima utvrđene su kvantitativne i kvalitativne karakteristike frakcija male i velike molekulske mase kolektora između čvrste i tečne faze u procesu flotiranja. Utvrđena je doza kolektora sa optimalnim odnosom frakcija lakih destilata, ugljovodoničnih ulja, smole i asfaltena mešanjem mazuta M-40 sa dizelom u određenim odnosima. Pokazalo se da je primena kolektora smeše sa masenim procentom mazuta M-40 60-70 omogućila povećanje iskorišćenja dijamanta za 2,7-3,5%. Slično povećanje je postignuto i pri korišćenju brodskog loživog ulja F-5 razblaženog za 10-18% frakcijom dizel goriva. Određena je optimalna temperatura od 24 °C za početno kondicioniranje rude flotacionim kolektorima, koja obezbeđuje maksimalno izdvajanje dijamanta u koncentrat, kao i visoku selektivnost. Predloženi kolektori i temperaturni uslovi omogućili su povećanje iskorišćenja dijamanta.

Ključne reči: Dijamanti; Flotacija; Ekstrakcija; UV spektroskopija; IR spektroskopija; Složeni kolektori; Mazut; Dizel; Srednja temperatura.