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Morozov V. V. et al. Current trends of improving the efficiency of froth separation.

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#### BENEFICIATION AND PROCESSING OF NATURAL AND TECHNOGENIC RAW MATERIALS

Research paper

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#### Current trends of improving the efficiency of froth separation of diamond-bearing kimberlites

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#### Abstract

Along with the introduction of new froth separation sections at new and existing enterprises, the basis for increasing the output of small diamonds is the reduction of their losses at the existing froth separation sections. The results of the studies of diamond surface composition under conditions of technogenic hydrophilization made it possible to establish the influence of the effects of crystallization of carbonate and silicate mineral films and the fixation of sludge fractions on the hydrophobicity and floatability of diamonds. It was proposed to use combined regimes of conditioning of ore and recycled water to increase floatability of diamonds, providing removal of hydrophilizing coatings and restoration of natural hydrophobicity of diamonds. The application of methods of acoustic, thermal, electrochemical, and reagent treatment of water-mineral disperse systems, as well as their combinations to increase floatability and reduce losses of hydrophilic diamonds in the process of froth separation was considered and substantiated.

Based on the study of the effect of the temperature factor in the preparation and froth separation processes, the optimal temperature regime of froth separation cycle operations was substantiated, providing the use of the heat consumed for thermal treatment of the initial diamond-containing material at a temperature of 85–90°C to maintain the required temperature in the conditioning operations with a collector and immediately in the froth separation and flotation operations.

It was shown that the regulation of phase composition of an apolar collector by additives of low- and mediummolecular fractions provides increase of its collecting ability due to transition of asphaltene-resin fraction into adhesion-active form and occurrence of processes of the collector autodispergating in aqueous phase.

On the basis of the statistical analysis of froth separation process indicators depending on changing the share of recycled water in the processes the reason for worsening the indicators (performance) was determined, which consisted in a significant increase in the concentration of sludges. The optimum degree of recycled water use (85%) was determined, which ensures decreasing the used collector consumption by 8% without decreasing diamond recovery and concentrate quality.

#### Keywords

diamonds, kimberlites, coatings, conditioning, hydrophobization, collector, froth separation, closed-loop water recycling, ALROSA

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#### ОБОГАЩЕНИЕ, ПЕРЕРАБОТКА МИНЕРАЛЬНОГО И ТЕХНОГЕННОГО СЫРЬЯ

Научная статья

## Современные направления повышения эффективности пенной сепарации алмазосодержащих кимберлитов

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#### Аннотация

Наряду с внедрением новых отделений пенной сепарации на новых и действующих предприятиях ресурсом повышения выпуска мелких алмазов является снижение их потерь на действующих переделах пенной сепарации. Исследование состава поверхности алмазов в условиях техногенной гидрофилизации позволило установить влияние эффектов кристаллизации пленок карбонатных и силикатных минералов, а также закрепления шламовых фракций на уменьшение гидрофобности и флотируемости алмазов. Предложено использовать для повышения флотируемости алмазов комбинированные режимы кондиционирования руды и оборотной воды, обеспечивающие удаление гидрофилизирующих покрытий и восстановление природной гидрофобности алмазов. Рассмотрено и обосновано применение способов акустической, тепловой, электрохимической и реагентной обработки водно-минеральных дисперсных систем, а также их комбинаций для повышения флотируемости и снижения потерь гидрофильных алмазов в процессе пенной сепарации.

На основании исследования влияния температурного фактора в процессах подготовки и пенной сепарации обоснован оптимальный температурный режим операций цикла пенной сепарации, использующий тепло, расходуемое для тепловой обработки исходного алмазосодержащего продукта при температуре 85–90 °C, для поддержания требуемой температуры в операциях кондиционирования с собирателем и непосредственно в операциях пенной сепарации и флотации.

Показано, что регулирование фазового состава аполярного собирателя добавками низко- и среднемолекулярных фракций обеспечивает повышение его собирательной способности за счет перевода асфальтен-смолистой фракции в адгезионно-активную форму и протекания процессов автодиспергирования собирателя в водной фазе.

На основании статистического анализа показателей процесса пенной сепарации в условиях изменения доли направляемой в технологические процессы оборотной воды определена причина снижения показателей, заключающаяся в существенном возрастании концентрации шламов. Определена оптимальная степень замыкания водооборота (85%), обеспечивающая снижение расхода применяемого собирателя на 8% без уменьшения извлечения алмазов и снижения качества концентратов.

#### Ключевые слова

алмазы, кимберлиты, покрытия, кондиционирование, гидрофобизация, собиратель, пенная сепарация, замкнутый водооборот, АК «АЛРОСА»

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#### Introduction

One of the promising areas for increasing the output of fine diamond size classes at ALROSA's enterprises is their extraction from kimberlites using a method of froth separation developed at ALROSA under the guidance of PhD M.N. Zlobin [1, 2]. The importance of the froth separation and flotation process is due to the fact that it enables achieving beneficia-

tion of -2+0.5 mm size class, to which more than 40% of the total quantity of diamonds in the ore belong, which constitute up to 10% of the value of all commercial products.

Along with the introduction of new froth separation sections at new and existing enterprises, the basis for increasing the output of small diamonds is the reduction of their losses at the existing froth separa-



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tion sections, which currently reach 20%. The losses are primarily caused by hydrophilic mineral coatings present on a diamond surface, formed under conditions of hypergenic processes in a deposit or due to technogenic hydrophilization [3, 4]. A promising way to remove hydrophilizing coatings and restore the natural hydrophobicity of diamonds is using a combination of different types of physical and physicochemical methods of treatment, the most effective of which are thermal, acoustic treatment of pulp, and electrochemical treatment of recycled water [5, 6]. At the same time, a necessary condition for achieving a positive result is to ensure a selective regime of diamond hydrophobization, which is achieved by using dispersing agents for sludges and agent-regulators of crystallization of hydrophilizing coatings [7].

Another important trend in increasing the efficiency of the froth separation cycle is optimization of the composition of collectors, which are presented by various petroleum products [8, 9]. This solves the problems of both increasing diamond recovery and reducing the consumption and, consequently, the cost of reagents. An important condition for increasing diamond recovery is a reasonable selection of the temperature conditions of the processes of conditioning a diamond-containing product with collectors and the processes of froth separation and flotation as such [9].

The selected directions of improving the process of froth separation and flotation allow achieving maximum performance with the correct selection of the process parameters, taking into account the mechanism of interaction of mineral surface with ionic-molecular components of the pulp liquid phase and with a collector, as well as changes in the composition of the aqueous phase under conditions of closed-loop water circulation [10, 11]. Therefore, the main scientific goal of the research was to establish the regularities of changes in surface properties and floatability of diamonds when using different types of conditioning of water-disperse systems in the processes of froth separation and flotation of diamond-bearing kimberlites.

#### 1. Research techniques

The elemental composition of the surface mineral coatings on diamonds was analyzed using the method of scanning X-ray electron spectrometry at a JSL-5610LV Jeol scanning electron microscope [12]. The information on the mineralogical composition of the solid phase was obtained by analyzing the data of infrared spectrophotometry in the wave number range of 400–4000 cm<sup>-1</sup> using a Specord 75 IR instrument [13].

An improved technique of measuring the threephase wetting contact angle of a collector drop on minerals using an OCA 15EC instrument was used to study the effect of a collector composition and preparation regimes on the collector fixation and hydrophobicity of diamonds and kimberlite minerals [14].

The method of combined optical microscopy was used to study the phase composition and structure of the collectors [15]. The images of a thin layer of the petroleum products were obtained by a Micromed-3-LUM microscope. Visiometric analysis and determination of size distribution of grains of asphaltene-resin and paraffin fractions were carried out using VideoTesT 4.0 program package<sup>1</sup>. The peculiarity of the applied technique is the possibility to determine the mass fraction of both asphaltenes and petroleum resins in a collector in the form of solid formations.

The collecting properties of the studied collectors in relation to diamonds were tested using a frothless flotation cell, Hallimond tube [16]. For laboratory studies of the process of diamond-containing kimberlite product froth separation, a froth separator equipped with a conditioner with reagent dosers, a unit for electrochemical treatment of recycled water, and a steam generator were used. The initial material with the coarseness of 0.5 to 2 mm, which was taken from the feed of the froth separation cycle, diamond-containing gravity concentrate, was used in the research.

To determine the regularities of interaction of the collector with minerals, the methods of extraction-spectral analysis of the collector distribution between the liquid and solid phases of the flotation system were used [17].

Scaled-up tests of the froth separation process were carried out at a LFM-001C automated unit of the Yakutniproalmaz Institute using closed-loop water cycle. The reagent regime used was consistent with the plant reagent regime. In the scaled-up tests, conditioning of a sample with a collector for two minutes was performed after pretreatment. A prepared sample of diamond-containing material was fed onto the separator froth bed. The resulting froth and cell products were dewatered on a sieve. The separated aqueous phase was returned to the recycled water tank. Diamonds were extracted from the froth separation products after drying them for weighing and recovery calculation.

<sup>&</sup>lt;sup>1</sup> VideoTesT-Master (Structure) 4.0: specification. Saint Petersburg 2002, 15 p.

### 2. Restoration of natural hydrophobicity and floatability of hydrophilized diamonds

In order to select the conditions for preventing technogenic hydrophilization of diamonds, special studies were conducted, and quantitative regularities of formation of hydrophilizing coatings on their surface were determined. For this purpose, surface composition studies were carried out while ageing a sample in desludged and non-desludged recycled water. The surface composition was determined by scanning X-ray electron spectroscopy. At the same time, the floatability of diamonds was determined by flotation of a sample weighing 200 mg with grain size of 0.5 to 1 mm in a frothless flotation unit for 4 min at a total air consumption of 50 ml. Prior to flotation, conditioning of the diamond sample in a collector emulsion was carried out.

To reproduce the conditions of formation of technogenic mineralization occurring at the contact of diamonds with mineralized aqueous phase in sample preparation operations, the prepared sample before conditioning with a collector was pre-aged in recycled water in an open container for 180 min.

The tests results showed that ageing diamonds in the process water of the processing plant No. 3 of the Mirny Mining and Processing Complex leads to a continuous increase in the mass fraction of mineral coatings on the surface of diamond crystals (Fig. 1) and a decrease in the diamond floatability (Fig. 2).

The high rate of changing surface composition and decreasing floatability allows concluding that the initial cause of diamond hydrophilization is the crystallization of carbonate and silicate mineral films [6]. The comparison of the dependences shows that ageing diamonds in non-desludged pro-



**Fig. 1.** Changes in the mass fraction of elements of mineral coatings on the surface of diamonds when they are aged in desludged (1, 2) and non-desludged (3, 4) recycled water: 1, 3 - calcium; 2, 4 - silicon

cess water leads to a significantly more intensive (4-5.5 times) increase in the concentration of mineral coatings on the diamond surface (see Fig. 1) and a significant decrease in the floatability of minerals (by 10.1%, see Fig. 2). The most probable mechanism of technogenic hydrophilization of diamonds is fixation of sludge fractions on the surface of floating minerals hydrophilized by mineral films.

To solve the problem of restoring the natural hydrophobicity and floatability of diamonds, taking into account the significant contribution of sludges, the application of acoustic activation was considered [18, 19]. Taking into account the results of our own earlier studies [5, 6, 9], it was proposed to use combined regimes, including the application of both acoustic and thermal treatment to increase the floatability of diamonds.

The tests were performed with diamond-containing samples at a laboratory froth separator. The diamonds were extracted from selected kimberlite samples of +0.5–2 mm grain size at an X-ray luminescence separator. The diamond-free samples of kimberlite products were then averaged and divided into 30 g subsamples. 20 diamond crystals were added to each subsample. A prepared subsample in the presence of sodium hexametaphosphate was treated with a collector (F-5 bunker fuel oil and butyl aerofloat at consumptions of 1,000 and 25 g/t, respectively) and fed to the froth separator. The frothing agent, methylisobutylcarbinol (MIBC), was fed into the aqueous phase of the froth separation process.

In the course of the laboratory tests at the froth separation unit, an initial subsample was heated and aged at a temperature of 60-95 °C for 1 min. Then acoustic (ultrasonic) treatment was carried out at an



**Fig. 2.** Recovery of diamonds in frothless flotation after ageing them in low-salinity (1), desludged (2) and non-desludged (3) high-salinity recycled water



IL 100-6/1 unit for 1-2 min. After removal of excess water phase with sludge fraction, reagents (the fuel oil and aerofloat) were added to the subsample and agitated for two minutes.

The prepared subsample was fed to the froth bed of the laboratory separator. The concentrate (froth product) and tailings (cell product) obtained in the froth separation process were dewatered. The separated aqueous phase was returned to the recycled water tank. Diamonds were extracted from the froth separation products after drying them for weighing and recovery calculation.

The results of the analysis showed that the joint application of thermal and ultrasonic treatment led to a 1.8–4.2-fold decrease in the proportion of diamond surface with mineral coatings. The earlier studies showed that the maximum increase in diamond recovery was achieved with the use of a combination of ultrasonic treatment and heating a sample to 85–90 °C [20].

The study of reagent methods of intensifying diamond desludging by conditioning of froth separation feed also showed the effectiveness of using sodium polyphosphate, oxyethylene diphosphonic acid (OEDP), and sodium metasilicate. The results of X-ray-electron spectral analysis showed that the use of dispersing agents allowed to reduce the surface concentration of mineral contaminant components by 1.3–1.5 times and promotes the removal of sludges from the diamond surface. Good results were achieved with the simultaneous application of ultrasonic scrubbing and additions of oxyethylene diphosphonic acid to the aqueous phase at a consumption of 500 g/m<sup>3</sup> [20]. The maximum positive effect was achieved when ultrasonic, thermal, and reagent treatment processes were used together. The results of the tests showed that the combination of ultrasonic (UST) and thermal treatment of the initial feed with the addition of oxyethylene diphosphonic acid (OEDP) or sodium polyphosphate provided an increase in diamond recovery by 7.5% with a significant decrease in the yield of kimberlite into the concentrate (Table 1).

A significant effect on hydrophobization and increase of floatability of diamonds is provided by electrochemical conditioning of recycled water [6, 21]. To assess the effect of a combination of factors (changing diamond hydrophobicity and the saturation of the aqueous phase of the pulp with gas), a test was conducted to determine the collector retentivity in relation to diamond crystals of flotation size (+0.6–1 mm). In the test, a droplet of the fuel oil interacted with a subsample of diamond crystals (100 mg) at the bottom of a glass cuvette and then recycled water were added. The fuel oil droplet concentrated at the aqueous phase-air interface and floatated diamonds fixed at the fuel oil-aqueous phase interface (Fig. 3, a).

Table 1

and source polyphosphate on noth separation outcomes								
No.	Processing conditions			Recovery to concentrate, %				
	UST duration, sec.	Temperature, °C	Dispersing agent concentration, mg/L	Diamonds	Kimberlite			
	Using OEDP							
1	_	24	-	82.5	0.86			
2	60	24	100	90.0	0.40			
3	60	85	200	90.0	0.33			
Using sodium polyphosphate								
4	60	24	100	80.0	0.44			
5	60	85	200	90.0	0.36			

Effect of ultrasonic and thermal treatment and additives of oxyethylidene diphosphonic acid (OEDP) and sodium polyphosphate on froth separation outcomes



**Fig. 3.** An aggregate of a F-5 bunker fuel oil droplet and diamonds on the surface of recycled water: *a* – in ordinary recycled water; *b* – in recycled water after electrochemical conditioning



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tailings

**Fig. 4.** Scheme for preparation of diamond-containing product for froth separation process, including thermal treatment of ore pulp and electrochemical treatment of recycled water

The results of observations showed that when using recycled water that has undergone electrochemical conditioning, a droplet of the fuel oil spreads to a greater extent along the interface between the aqueous phase and air, and a 40-50%greater number of diamond crystals are fixed on it (Fig. 3, *b*). At the same time, a large number of gas microbubbles are observed on the surface of diamonds, which indicates the saturation of the treated water with gas phase and the tendency of the gas phase to form bubbles on the surface of diamond crystals.

To evaluate the effectiveness of the combination of the thermal and electrochemical conditioning, a scheme was tested, including heating of the froth separation feed before the flotation reagents conditioning operation and the subsequent use of the accumulated heat in the operations of the froth separation feed conditioning with the reagents and properly the froth separation process (Fig. 4).

The flotation tests were carried out using electrochemical conditioning of recycled water. Electrochemical conditioning was carried out in a diaphragmless electrolyzer at a current density of 100 A/m<sup>2</sup> and an electricity consumption of 1.5 kWh/m<sup>3</sup>. To clarify the mechanism of the electrochemical conditioning effect on diamond properties, tests with hydrophobic and technogenically-hydrophilized diamonds were performed. Hydrophilization of diamonds was carried out by ageing them in sludge-containing recycled water of the froth separation cycle in contact with air for three hours.

The analysis of the developed regime testing at the laboratory froth separation unit showed that when using the combined thermal and electrochemical treatment (at a temperature of 85-90 °C), the diamond recovery (82.5%) is noticeably higher than the recovery when using these treatment methods separately (58.5% and 69%, Table 2). The yield of kimberlite into the concentrate remained stable in all tests (0.47–0.6%) [20].

The analysis of the conducted studies outcomes showed that floatability of hydrophilic diamonds after thermal and electrochemical treatment increased by 49% and became comparable with the recovery of naturally hydrophobic diamonds. This result allows concluding that the reason for the increase in floatability of diamonds is the restoration of their natural hydrophobicity due to the removal of hydrophilizing films. The observed increase in the recovery of naturally hydrophobic diamonds (up to 18%) indicates the manifestation of the effect of additional aeration of the medium by electrolysis gases, which is typical for the conditions of application of electrolysis products (see Fig. 3) and, possibly, due to other effects, for example, an increase in the activity of a collector.

#### Selection of temperature regime of conditioning of diamond-containing product with collector

The temperature of a medium in the preparatory and basic process operations is an important parameter of froth separation cycle [9, 17]. To determine the rational thermal regime, wetting contact angle measurements were performed on diamonds and kimberlite in the temperature range of 14 to 60 °C.

Table 2

#### Effect of temperature on wetting contact angles of diamonds and kimberlite by a droplet of collector in aqueous medium

No.	Medium temperature, °C	Wetting contact angle on minerals, deg. (minimum–maximum/average)			
		Diamond	Kimberlite		
1	14	91-95/93	Breakaway		
2	24	92-97/94.5	Breakaway		
3	30	94-101/97.5	Breakaway		
4	40	94-100/97.0	Breakaway		
5	50	91-96/93.5	Piecewise, 40–75/57.5		
6	60	90-93/91.5	Piecewise, 45–75/60		

The influence of temperature on the adhesive activity of a collector to the surface of a diamond and kimberlite in aqueous medium was assessed by measuring the wetting contact angles using a special technique, including the application of a droplet of F-5 fuel oil to the wetted surface of a mineral and the subsequent rise in the liquid level in the cuvette, described in detail in [9]. The results showed that the wetting contact angle, which characterizes the hydrophobicity of a diamond and its tendency to interact with a collector, increases in the temperature range of 14–40°C and decreases with further temperature increase (Table 2). The fixation of the collector on the surface of kimberlite is observed fragmentarily and manifests itself at temperatures above 40°C.

When the thermal conditioning of the initial feed of the froth separation cycle is carried out at 80-90 °C, the temperature in the subsequent operation of conditioning with reagents is 25–30°C, and in the froth separation operation, 20-22 °C. The higher medium temperature in the conditioning and froth separation operations with the use of thermal conditioning relative to the medium temperature in the control test  $(14 \,^{\circ}\text{C})$  is provided by the heat energy expended in the initial feed thermal conditioning operation. The froth separation tests confirmed the results of the tests on studying the effect of temperature on the adhesive activity of a collector in relation to diamonds and showed that maintaining the medium temperature in the operation of conditioning the diamond-containing product with the collector up to 30-40 °C led to an increase in diamond recovery by 6.2-7.3% (Table 3).

The obtained results (achieving the maximum positive effect at a temperature of  $40 \,^{\circ}$ C) showed that in order to achieve the best outcomes, it is advisable, along with thermal conditioning of the froth separation cycle feed, to conduct additional pulp heating in the operation of conditioning of the froth separation feed. At the same time, the required temperature regime of the froth separation and flotation operation (24 to 28 °C) is to be maintained [9].

Table 3

Effect of medium temperature in the collector conditioning operation on diamond and kimberlite recovery in the froth separation operation

No	Medium	Flotation recovery, %		
INO.	temperature, °C	Diamond	Kimberlite	
1	14 (without thermal conditioning)	79.1	1.7	
2	24	83.6	1.7	
3	30	85.3	1.7	
4	40	86.4	1.6	
5	50	85.0	1.6	

The efficiency of the selected temperature regime of the froth separation process was tested on a LFM-001C automated froth separation unit of the Yakutniproalmaz Institute. In the tests, the initial diamond-containing product was heat treated at a temperature of 85 °C. Due to the accumulated heat, the temperature of the medium in the operation of conditioning with the collector was 34–40 °C, and in the froth separation operation, 24 °C. The tests results showed that the diamond recovery to the concentrate achieved at thermal conditioning of the initial feed and subsequent increase of the medium temperature in conditioning and froth separation operations exceeded the corresponding recovery values in the control test at the medium temperature in conditioning and froth separation operations of 14 °C by 3.5%.

Thus, according to the results of scaled-up studies conducted at the automated unit, the temperature regime of the froth separation cycle operations for the processing plants of Alrosa was recommended, which implied maintaining the temperature in the heat treatment operation of the initial feed at 85-90 °C, in the conditioning operation with the collector, at 30-40 °C, and in the froth separation operation, at 20-24 °C.

#### **Optimization of collector fractional composition**

Fuel oils used in diamond flotation are not optimal collectors. This is due to the fact that GOSTs and TU for petroleum products determine their composition and properties to satisfy the requirements of consumers, which are power engineering and transportation facilities. Earlier studies have shown that F-5 and M-40 fuel oils contain a large amount of inactive resin-asphaltene fraction present as crystallized solids [9].

In order to increase the collecting ability of M-40 and F-5 fuel oils, it was proposed to transfer the resin-asphaltene fraction from the coarse-dispersed state to the form of colloidal or molecular solution by adding light ends of petroleum refining [17]. To determine the collector structure formation regularities at its dilution and the subsequent selection of the fractional composition, the modified M-40 fuel oil was studied using the method of combined optical microscopy. The technique application allows to diagnose the presence of crystalline and colloidal forms of insoluble components, for example, the fraction of petroleum resins and asphaltenes in a petro leum product [22, 23].

The studies carried out by optical microscopy method using a Micromed-3-LYuM microscope in combined lighting regime showed that the resins and asphaltenes are present in the dispersed state (black crystals and aggregates) and in the form of solution in medium- and low-molecular components (areas with yellow–green glow). The analysis of the results showed that dilution of M-40 fuel oil with diesel oil cut (DOC) by 20 and 30% results in dispergating of the aggregates of asphaltene crystals and their dissolution with formation of fine-dispersed and dissolved forms (Fig. 5).

To determine the efficiency of fixing the collector on diamonds, the technique of UV-spectral analysis of the distribution of a collector between the liquid and solid phases of the flotation system was used, including operations of extraction of organic substances from the components of the aqueous dispersed system diamond – aqueous phase, measurement of concentrations, and the collector balance calculation [17].

The collecting properties of the studied collectors in relation to diamonds were tested using a frothless flotation cell, Hallimond tube. The diamonds of the coarseness of 0.5 to 1 mm, which were taken from diamond-containing gravity concentrate, were used in the research.



Fig. 5. Change of mass fraction of asphaltene-resin phases at dilution of M-40 fuel oil with diesel oil cut:
1 – calculated; 2 – in solid form according to the results of visiometric analysis; 3 – in dissolved and emulsion forms (as a difference of the first two values)

Table 4

Amount of fixed collector and recovery of diamonds to concentrate in Hallimond tube frothless flotation of diamonds

No.	Collector used	Share of collector fixed on diamonds, %	Diamond recovery into concentrate, %
1	M-40 fuel oil	45	68.4
2	M-40 fuel oil+20% DOC	61	78.6
3	M-40 fuel oil+20% DOC+DEK (KSM-1)	82	92.2
4	M-40 fuel oil+20% DOC+EMK (KSM-2)	87	92.5
5	M-40 fuel oil+20% DOC+DMK (KSM-3)	80	88.7

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The research results showed that the compound collector, obtained by dilution of M-40 fuel oil by 20% DOC, demonstrated the best process performance: an increase in the proportion of collector fixed on the surface of diamonds by 16%, and a significant increase in their floatability (Table 4, test 2).

Another effective approach to modify the characteristics of the collector is the addition of reagents to its composition, providing autodispergating of the collector in aqueous phase. It was confirmed that the use of ketone additives (DEK – diethyl ketone, EMK – ethyl methyl ketone, DMK – dimethyl ketone) in the compound M-40 fuel oil-based collector composition provided an increase in the proportion of the collector fixed on diamonds from 45 to 87% (Table 5, tests 3, 4, 5). The proportion of collector fixed on kimberlite minerals increases insignificantly in this case.

The results of the flotation tests showed (see Table 4) that the maximum recovery of diamonds using the compound of M-40 fuel oil and aliphatic ketones was achieved in the area of mass fraction of ketones of 8-20%.

Collectors KSM-1 and KSM-2, which are compounds of M-40 fuel oil, diesel oil cut, and ketones, were also tested at a LFM-001S froth separation unit, operating in close to industrial regime (collector consumption of 1000 g/t, butyl aerofloat consumption of 50 g/t, a frothing agent consumption of 150 g/t). Bench test results showed that in the temperature range of 14-24 °C, the diamond recovery reached 90-95% with a selectivity of 82.2-89.25% [17]. The comparison of the flotation tests results showed that the use of KSM-1 and KSM-2 collectors allowed achieving the recovery of 90% at half less consumption of the collector (Fig. 6). This is very important in terms of both reducing costs for flotation agents and decreasing environmental impact.

#### Selection of water recycling scheme parameters in froth separation cycle

The proportion of recycled water in a water consumption in the froth separation cycle is a key parameter determining the level of accumulation of soluble salts, sludges, and flotation reagents in the aqueous phase of beneficiation processes [24, 25].

The main reason for worsening froth separation performance at increasing the share of recycled water in the total water balance is the process of sludge accumulation. An increase in the share of recycled water leads to an increase in the concentration of sludge, which is due to the deterioration of conditions and decreasing efficiency of the recycled water clarification operation.

On the other hand, increasing the share of recycled water increases residual concentrations of

collector by 25–40%. This not only does not worsen flotation performance, but also reduces reagent consumption by 10-15%.

The results of the regression analysis showed that sludge concentration is the most significant adverse factor leading to lower diamond recovery, which results from a significantly stronger negative association (PCC = -0.56, Table 5) compared to the association with other parameters (PCC = -0.31-0.32, Table 5).

The results of the regression analysis are confirmed by a comparative analysis of the dependence of sludge accumulation and diamond recovery to the concentrate on the proportion of recycled water in the froth separation cycle. As can be seen from Fig. 7, when the proportion of recycled water reaches 85%, a gradual increase in the concentration of sludge in recycled water and a decrease in diamond recovery are observed. Therefore, the proportion of recycled water in the water balance in the froth separation cycle of 85% was established as the maximum permissible value (Fig. 7).

The increase in the proportion of recycled water from 70 to 85% recommended based on the findings of the present research was tested and implemented when modernizing the scheme of the froth separation cycle at Processing Plant No. 3 of Mirny Mining and Processing Complex. The task of reducing the concentration of sludge in recycled water was achieved with the use of single-stage clarification of the -2-+1 mm coarseness initial feed thickening overflow and final tailings of the froth separation. The results of the tests showed the feasibility of reducing the collector consumption by 7% with the proportion of recycled water of 85% while maintaining diamond recovery and concentrate quality at the same level (Table 6).

To increase the proportion of recycled water in the water balance of the froth separation cycle up to 90% (in order to reduce the consumption of flotation agents), it is necessary to use effective methods to decrease the concentration of sludge in recycled water. This result can be achieved by using a two-stage scheme for clarification of the recycled water or by adding coagulants or flocculants that do not adversely affect the froth separation process. Reduction of sludge concentration

by 20–30%, achieved through the application of a twostage clarification scheme, will allow increasing the proportion of recycled water up to 90% without reducing diamond recovery and achieving a total reduction in reagent consumption by 11%.



Fig. 6. Diamond recovery in froth separation with the use of collectors:
1 - M-40 fuel oil; 2 - KSM-1 collector; 3 - KSM-2 collector; 4 - KSM-3 collector



**Fig. 7.** Effect of the proportion of recycled water in the water balance of froth separation cycle on: *1* – sludge concentration and *2* – diamond recovery; *3* – limiting value of the proportion of recycled water

Table 5

Pair correlation coefficients (PCC) between diamond recovery and parameters of recycled water of froth separation cycle

Descaled water norm store	Diamond recovery, %			PCC between diamond recovery	
Recycled water parameters	Minimum	Maximum	Medium	and recycled water parameters	
Proportion of recycled water	65	90	74	-0.31	
Salinity, g-ion/l	0.4	0.6	0.47	-0.32	
Sludge concentration, g/l	3.1	5.8	4.0	-0.56	

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Table 6



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<b>Recycled</b> water	Collector consumption, g/t		Diamond recovery into	Kimberlite yield into	
proportion, %	F-5 bunker fuel oil	Butyl aerofloat	concentrate, %	concentrate, %	
Single-stage desludging					
70	1,100	25	87.9	0.65	
75	1,050	23.5	87.9	0.64	
85	1,020	22.8	87.8	0.63	
90	970	21.5	85.8	0.62	
Two-stage desludging					
85	1,020	22.8	87.9	0.60	
90	970	21.5	87.8	0.60	

#### Froth separation cycle indicators when changing recycled water proportion

#### Conclusions

It was shown that technogenic hydrophilization of diamonds is caused by the processes of crystallization of films of carbonate and silicate minerals and fixation of sludge fractions on the hydrophilized surfaces of diamonds. It was proposed to use combined regimes of conditioning of ore and recycled water using acoustic, thermal, electrochemical, and reagent treatment to increase diamond recovery, providing an increase in floatability of hydrophilic diamonds by 30–35% by reducing or preventing their hydrophilization and removal of sludges.

A rational temperature regime of froth separation cycle is proposed, including thermal treatment of the initial feed and assuming the use of heat consumed in the cycle to increase the temperature of the medium in the operations of conditioning with the collector and froth separation of a size class beneficiated. The feasibility of reducing diamond losses due to maintaining the optimal thermal regime by 3.5% was established. It was shown that an effective way to increase the performance of froth separation is the modification of phase composition of fuel oils by additives of low- and medium-molecular fractions, which ensured the transition of asphaltenes and oil resins into an adhesive-active form, as well as provided autodispergating the collector in the aqueous phase. Tests of KSM-1 and KSM-2 reagents with additives of alkyl ketones confirmed their greater efficiency in comparison with F-5 bunker fuel oil.

The optimum proportion of recycled water (85%) was determined, which was achieved by applying a water recycling scheme including single-stage clarification of the initial feed thickening overflow and final tailings of the froth separation cycle, providing an 8% reduction in collector consumption while maintaining diamond recovery and concentrate quality at a high level. Application of a two-stage clarification scheme is proposed to reduce the concentration of sludge in water and increase the proportion of recycled water use.

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