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# Rationale for the temperature regime of the foam separation cycle

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***Abstract***

***The research objective*** *is to determine the optimal temperature regimes for slurry preparation and foam separation of diamond-bearing kimberlites to ensure maximum diamond recovery under high selectivity of the process.*

***Methods of research*** *include the electron probe X-ray spectral analysis, IR spectrophotometry, and measurement of the contact angles of a collecting agent drop on diamonds or minerals. Technological studies were carried out on the setup for foam separation.*

***Research results.*** *It was shown that when the feedstock of the foam separation cycle is heated to a temperature of 80–85 °C the diamonds are effectively purified from hydrophilic coatings, which leads to the restoration of their natural floatability. Through contact angles measurement, the temperature range of 30–40 °C was determined in the feedstock reagent conditioning operation. It is shown that the maximum water repellence of diamonds is achieved in this temperature range without a significant increase in the kimberlite minerals water repellence. Laboratory experiments have shown that the best foam separation results are achieved when in the conditioning operation the feedstock with flotation reagents is maintained at a temperature of 30–38 °C. The flotation studies using F-5 bunker fuel oil as base collecting agent, as well as its compounds with diesel fraction and Machchobinsky oil, determined the optimal temperature of 14–24 °C directly in the process of foam separation. After data analysis, a temperature regime was proposed and tested, which includes the foam separation feedstock heating before the operation of slurry removal and conditioning with flotation reagents up to 85 °C and the subsequent use of accumulated heat in the operations of foam separation feedstock conditioning (30 °C) with reagents and the foam separation process itself (18 °C).*

***The prospects of the technology.*** *The test results of the selected temperature regime for the foam separation process on a test bench show the possibility of increasing the diamonds recovery into the concentrate by 2.3–4.5% when using applied and potential collecting agents, including F-5 bunker fuel oil and compounds based on it. The developed regime is recommended for commercial development in the foam separation cycle at Alrosa processing plants.*

***Keywords:*** *diamonds; kimberlites; foam separation; collecting agent; conditioning; hydrophobic properties; heat processing.*

**Introduction.** The main stages of processing, which ensure the production of commercial diamonds, are the cycles of foam separation and grease recovery. The main prospects for increasing commercial diamonds production at PJSC Alrosa are associated with improved efficiency of existing stages and the introduction of new technologies at processing plants [1].

Increased losses of diamonds are due to insufficient hydrophobization of their surface caused by both the spread of hydrophilic mineral coatings on the diamond crystal surface and the reduced activity of the collecting agents [2, 3]. Heat processing is a promising direction for increasing the efficiency of diamond-bearing raw materials foam separation [4, 5]. The mechanism of processes on the surface of diamonds in the course of the temperature rise during foam separation was studied extensively in [6]. It is shown that the diamond-bearing kimberlite materials processing at a temperature of 60–85 °C provides purification and prevention of repeated hydrophilization of the diamond surface.

Temperature rise during conditioning caused by the feedstock heat processing can contribute to a more efficient interaction between minerals and flotation reagents, primarily collecting agents [7, 8]. It is equally important to determine and maintain the optimal temperature regime in the very process of foam separation [9]. Therefore, the objective of the present research is to both carry out a study and determine the conditions for diamond surface hydrophilization prevention, and create conditions for effective interaction with collecting agents of various compositions in the course of heat processing [10, 11]. At the same time, the research task was to establish the relationship between the temperature regimes of the processes of slurry preparation, conditioning, and foam separation [12].

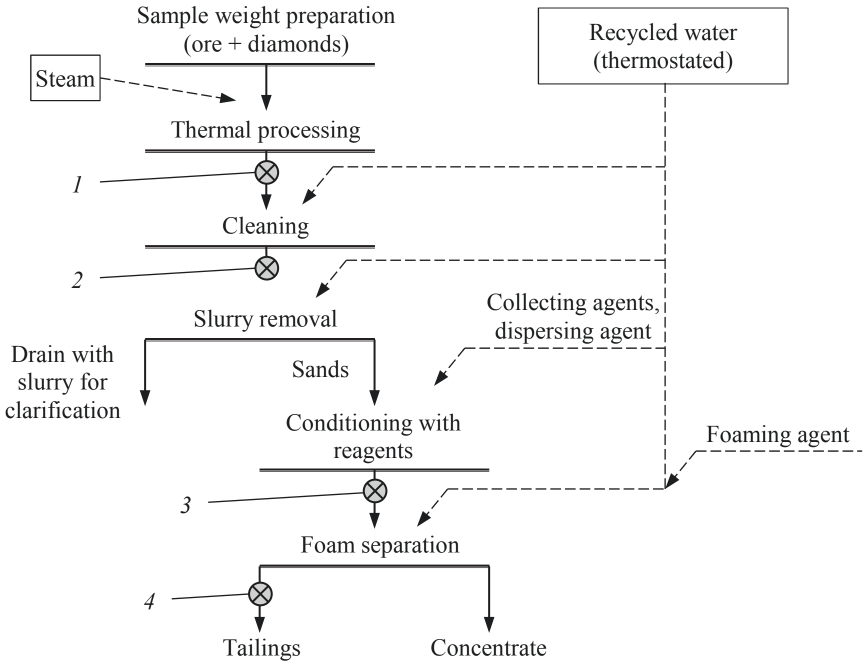


Figure 1. Scheme of the experiment of diamond-bearing products foam separation using feedstock heat processing. Temperature control points: *1* – in the operation of thermal conditioning; *2* – when cleaning; *3* – when conditioning with reagents; *4* – in foam separation

Рисунок 1. Схема опыта пенной сепарации алмазосодержащих продуктов с применением термической обработки исходного питания; точки контроля температуры: *1* – в операции теплового кондиционирования; *2* – при оттирке; *3* – при кондиционировании с реагентами; *4* – в пенной сепарации

**Methods of research.** The elemental composition of surface mineral formations on diamonds was analyzed by the method of electron probe X-ray spectral analysis (EPXRSMA) with a Jeol-5610 LV electron microscope [13]. Information on the mineral composition of the solid phase was obtained from the IR spectrophotometry data analysis within the wavenumber range of 400–4000 cm$^{-1}$ [14].

The method of measuring the contact angles of a collecting agent drop on diamonds or minerals with an OCA 15EC device was used for the research [15].

To carry out laboratory studies of diamond-bearing ore foam separation process, a module equipped with specialized process equipment was used, namely a trough conditioner with a stirring device in the form of an inclined spiral, a foam separator with a chamber volume of 1 l, and a steam generator.

The process flow scheme of foam separation, shown in Figure 1, was implemented with the laboratory module.

Prior to the study, the feedstock was manually selected from the foam separation feed before it was treated with reagents. Diamonds were extracted from the samples with the Kristall X-ray luminescent apparatus. The prepared diamond-free samples were proportionated and divided into sample weights of a certain mass (30 g). Twenty diamond crystals were added to each sample.

**Table 1. Change in the surface composition of diamonds after heat processing in a sample with kimberlite at different temperatures of the medium**

**Таблица 1. Изменение состава поверхности алмазов после тепловой обработки в пробе с кимберлитом при различной температуре среды**

| Element | Mass fraction of elements on a diamond, % | | | |
|---|---|---|---|---|
| | no processing | under 60 °C | under 80 °C | under 90 °C |
| C | 78.11 | 84.12 | 88.23 | 89.64 |
| $O_2$ | 6.22 | 4.76 | 3.42 | 3.04 |
| Na | 0.52 | 0.41 | 0.25 | 0.23 |
| Ca | 3.65 | 2.40 | 1.94 | 1.70 |
| Cl | 0.06 | 0.05 | 0.04 | 0.03 |
| Fe | 0.32 | 0.26 | 0.17 | 0.15 |
| Si | 2.43 | 1.87 | 1.32 | 1.16 |
| Mg | 4.56 | 3.24 | 2.46 | 2.15 |
| Al | 1.80 | 1.33 | 0.93 | 0.79 |
| Other | 2.33 | 1.55 | 1.24 | 1.11 |
| *Total* | 100.00 | 99.99 | 100.00 | 100.00 |

To reproduce the conditions of technogenic mineralization that occurs when diamonds contact with a mineralized aqueous phase, during sample preparation, the prepared sample was preliminarily held in recycled water and stirred in an open container for 60 min [16].

During the experiment, the prepared sample was heated and held at a temperature of 60–95 °C for 1 min. Then the container with the sample was placed in an ultrasonic unit and processed for 2 min. After the ultrasonic cleaning, the temperature of the medium was measured and the drain with the slurry fraction was removed.

Reagents (black mineral oil and aerofloat) were added to the rest of the sample, and the sample was mixed for 2 min. Upon completion of conditioning, the temperature was measured. The prepared sample was fed to the feed tray and then directed to the foam layer of the separator. The resulting foam and flotation tail were dehydrated. The separated aqueous phase was returned to the backwater basin, where the temperature was measured and, if necessary, adjusted.

After drying, diamonds were manually extracted from the foam and flotation tail to calculate the weight balance.

**Results and discussion.** ***To select the regime of thermal conditioning for the foam separation feed,*** the method of electron probe X-ray spectral analysis was used. The analysis results showed that the heat processing of diamond-bearing material, i.e. foam separation feed, provides diamond surface purification from mineral coatings. The results of X-ray photoelectron spectroscopy of the diamonds surface show that, with the temperature rise, the content of elements within the hydrophilic coatings decreases significantly (Table 1).

The surface concentration of mineral formations is noticeably decreased at temperatures above 45 °C (thermomechanical mechanism), and the diamonds are most intensively purified at more than 80–85 °C, when the thermochemical mechanism is realized [6]. Taking into account the significant acceleration of the cleaning process, heating to 85 °C is preferable, when both mechanisms of hydrophilizing surface formations destruction are realized.

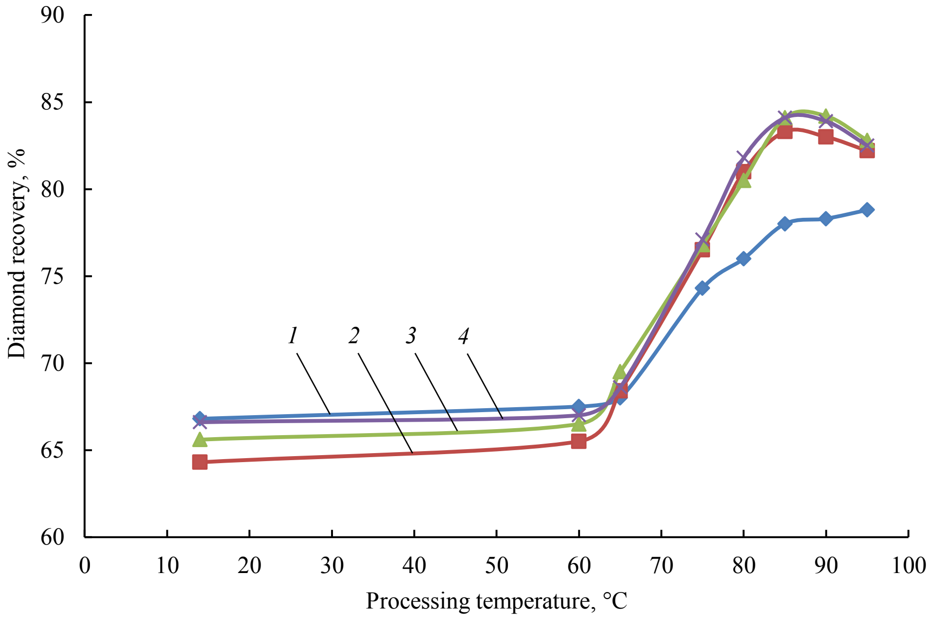


Figure 2. Recovery of diamonds into a foam separation concentrate using heat processing of the initial sample: *1* – 30 s; *2* – 60 s; *3* – 90 s; *4* – 120 s

Рисунок 2. Извлечение алмазов в концентрат пенной сепарации при использовании тепловой обработки исходной пробы: *1* – 30 с; *2* – 60 с; *3* – 90 с; *4* – 120 с

Experiments on foam separation showed that the best result from the heat processing of diamond foam separation feed is achieved at 85–90 °C (Figure 1), when diamonds recovery into concentrate increases by 18–20%.

Comparison of the surface composition (Table 1) and floatability (Figure 2) results shows that diamond recovery increase is due to the effective purification of their surface from mineral coatings at 60 °C and more. The results also showed that in order to achieve the maximum diamond recovery increase, the required duration of heat processing is 60 s.

***In order to choose the temperature regime for conditioning the feedstock of the foam separation with flotation reagents,*** a method was used to measure the contact angles of a collecting agent drop on diamond or kimberlite mineral surfaces in the aqueous phase. This method makes it possible to evaluate the oil-receptivity of minerals, which determines the effective fixing of apolar collecting agents with mineral surface. In accordance with modern concepts, oil-receptivity and water-repellence of minerals are the closely interrelated and proportionally changing parameters [17, 18].

During testing, the original sample of a polished diamond or a section of kimberlite minerals was soaked for 1 h in recycled water in contact with air. After that the sample was heated with recycled water under thermostatic conditions at 85 °C. After the sample was processed, the hot solution was drained and 10–60 °C recycled water was added, which ensured the sample holding and follow-up experiments in the temperature range of 14–60 °C.

**Table 2. Change in the contact angles of diamond and kimberlite minerals by a collector's drop with temperature variation**

**Таблица 2. Изменение краевых углов смачивания алмаза и минералов кимберлита каплей собирателя при варьировании температуры**

| The temperature of medium, °C | Contact angle of minerals, degrees | | | | |
|---|---|---|---|---|---|
| | diamond | phlogopite | kimberlite | calcite | olivine |
| 14 | 91–95 | 47–67 | Detachment | Detachment | Detachment |
| 24 | 92–97 | 54–68 | Detachment | 40–50 | 40–55 |
| 30 | 94–101 | 57–65 | Detachment | Detachment | Detachment |
| 40 | 94–100 | 57–66 | Detachment | Detachment | Detachment |
| 50 | 91–96 | 58–68 | Fragmental, 40–75 | Detachment | Detachment |
| 60 | 90–93 | 60–70 | Fragmental, 45–75 | Detachment | Detachment |

The composition of the recycled water corresponded to the recycled water of processing plant No. 3 of the Mirny GOK. F-5 bunker fuel oil was used as a collecting agent. The research results showed that the contact angle for a drop of F-5 bunker fuel oil, which characterizes the diamond water repellence and tendency to interact with the collecting agent, increases in the temperature range of 14–40 °C by 3.5 angular degrees (Table 2). At temperatures above 40 °C, the oil-receptivity diamonds decreases.

Initially, kimberlite (phlogopite) hydrophobic minerals and a diamond increase in oil-receptivity with the temperature rise. The hydrophilic minerals of kimberlite (olivine, calcite) decrease in oil-receptivity with the temperature rise, up to the termination of drop retention on the mineral surface at a temperature of 30 and more degrees (Table 2). It is impossible to accurately measure the contact angle on the surface of a kimberlite section because the collecting agent is held in separate areas of the surface, where grains of naturally hydrophobic minerals are visually detected.

When conducting the technological studies, the initial processing with a diamond-bearing sample was carried out at a temperature of 85 °C. After that, the ultrasonic cleaning and slurry removal were carried out. To create the required temperature in the conditioning operation, an aqueous phase with a temperature of 10 to 60 °C was used. Due to this, the temperature in the conditioning operation was maintained in the range from 10 to 42 °C. Foam separation was carried out at a constant temperature of 24 °C.

Laboratory experiments have shown that the best result of foam separation is achieved by maintaining the temperature of the medium in the conditioning operation with reagents from 30 to 38 °C (Figure 3).

With no preliminary heat processing of the feedstock, the level of diamond recovery is lower, but the type of dependence with the maximum diamonds recovery at 30–38 °C is preserved (Figure 3).

***To select the optimal temperature regime for foam separation,*** experiments were carried out at a setup for foam separation at a conditioning and flotation temperature from 10 to 28 °C. The selected temperature range corresponds to the conditions of foam separation at industrial enterprises in different seasons without special heat processing (10–14 °C) or with its intended use (24–28 °C). When conducting the research, F-5 bunker fuel oil and its compounds with a diesel fraction were used, as well as Machchobinsky oil and its compound with F-5 bunker fuel oil.

As can be seen from the data presented in Table 3, the best foam separation results are achieved at temperatures of 14 and 24 °C.

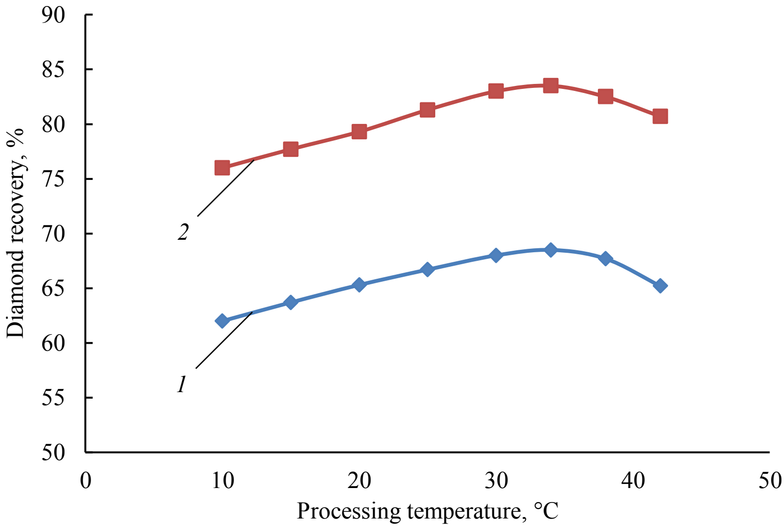


Figure 3. Dependence of diamond recovery into foam separation concentrate on the temperature of the medium in the conditioning operation with flotation reagents: *1* – without preliminary heat processing; *2* – with preliminary heat processing

Рисунок 3. Зависимость извлечения алмазов в концентрат пенной сепарации от температуры среды в операции кондиционирования с флотационными реагентами: *1* – без предварительной тепловой обработки; *2* – с предварительной тепловой обработкой

At 14 °C, the highest diamonds recovery into the concentrate was 78.4% and 77.9% when using the KM-10 and KM-14 collecting agents, respectively, obtained by diluting the F-5 fuel oil with a diesel fraction with a volume fraction of the diesel fraction of 10% and 14%, which is 2.8%–3.7% higher than that of F-5 bunker fuel oil, the best basic collecting agent (Table 3).

The recovery of diamonds into concentrate, achieved during nonfrothing flotation at a process temperature of 24 °C using reagent collecting agents based on F-5 bunker fuel oil, exceeds the corresponding recovery values at a flotation process temperature of 14 °C by 2.1–4.5% (Table 3). With a further temperature growth in the foam separation operation up to 28 °C, there is practically no increase in diamond extraction, while the kimberlite minerals recovery into the concentrate increases.

Thus, based on the results of the laboratory research, the rational thermal regime of foam separation cycle operations involves maintaining 85–90 °C in the operation of feedstock heat processing, the temperature of 30–38 °C in the operation of conditioning with a collecting agent, and the temperature of 14–24 °C in the operation of foam separation.

***The selected foam separation temperature regime was tested*** on an automated setup for foam separation LFM-001C which operated under the flow rate of the collecting agent of 1000 g/t, butyl aerofloat of 50 g/t, foaming agent of 150 g/t. The setup operated on recycled water.

When carrying out bench tests, regimes without feedstock heat processing (control mode) and with foam separation cycle feedstock processing at a temperature of 85 °C were chosen. Without feedstock heat processing, the temperature in the cleaning operation was 18 °C, in the conditioning operation the temperature was 20 °C, and in the foam separation the temperature was 16 °C.

**Table 3. Recovery of diamonds using different collecting agents with varying pulp temperature during foam separation**
**Таблица 3. Извлечение в концентрат алмазов с использованием различных собирателей при варьировании температуры пульпы при пенной сепарации**

| Collecting agent | Recovery into concentrate, % | | | |
|---|---|---|---|---|
| | 10 °C | 14 °C | 24 °C | 28 °C |
| | *Diamonds* | | | |
| F-5 bunker fuel oil | 72.6 | 77.8 | 82.2 | 82.4 |
| KM-10 | 76.4 | 82.4 | 85.6 | 85.4 |
| KM-14 | 77.8 | 82.9 | 85.0 | 84.8 |
| Machchobinsky oil | 68.6 | 72.6 | 75.7 | 75.2 |
| F-5 fuel oil + Machchobinsky oil 1 : 1 | 74.2 | 78.5 | 82.8 | 81.9 |
| | *Kimberlite* | | | |
| F-5 bunker fuel oil | 0.9 | 1.2 | 1.4 | 1.6 |
| KM-10 | 1.0 | 1.2 | 1.5 | 1.7 |
| KM-14 | 1.1 | 1.3 | 1.5 | 1.7 |
| Machchobinsky oil | 0.8 | 1.4 | 1.7 | 1.9 |
| F-5 fuel oil + Machchobinsky oil 1 : 1 | 1.0 | 1.3 | 1.5 | 1.7 |

Feedstock heating resulted in temperature rise in all operations. The results of temperature measurements in operations showed the following. With the selected initial sample heat processing regime, the temperature in the cleaning operation is 35 °C, the temperature in the conditioning operation is 30 °C, and the temperature in the foam separation is 18 °C (under the makeup water temperature of 16 °C). Such results confirm the possibility of maintaining the optimal temperature regime in all operations of feedstock preparation and in the very process of foam separation due to the accumulated heat.

The test results of the selected temperature regime of the foam separation process on a test bench showed the possibility of increasing diamonds recovery into concentrate by 2.3–4.5% when using almost all types of collecting agents (Table 4).

Based on the results obtained the developed technology of thermal conditioning and temperature regime of foam separation operations was recommended for use in the development of foam separation technological regime at processing plants No. 3 and No. 14 of PJSC Alrosa.

**Conclusions.** The optimal temperature regime for slurry preparation operations and diamond-bearing kimberlites foam separation were determined, namely, foam separation cycle feedstock should be heated to 85 °C, the temperature of 30–38 °C should be maintained during the reagent conditioning operation, and the temperature of 14–24 °C should be maintained during the foam separation. It is shown that the selected

**Table 4. Recovery during foam separation process on a bench installation at the basic (BR) and proposed (PR) temperature regimes**

**Таблица 4. Показатели извлечения в процессе пенной сепарации на стендовой установке при базовом (BR) и предлагаемом (PR) температурном режиме**

| Reagent collecting agent | Diamond, % | | Kimberlite, % | |
|---|---|---|---|---|
| | BR | PR | BR | PR |
| F-5 fuel oil | 74.9 | 79.4 | 1.7 | 1.7 |
| KM-10 | 77.2 | 81.7 | 1.9 | 1.9 |
| KM-14 | 81.6 | 83.9 | 2.2 | 2.9 |
| F-5 fuel oil + Machchobinsky oil 1 : 1 | 75.5 | 78.8 | 1.8 | 1.8 |

mode involves the use of the heat accumulated in the heat processing operation at 85 °C to maintain the required temperatures in the conditioning operations with reagents (30 °C) and in the very foam separation process (18 °C). The results of bench testing of the selected temperature regime of the foam separation process show the possibility of increasing diamonds recovery into concentrate by 2.3–4.5% under the use of the F-5 bunker fuel oil and compound collectors based on it as a collecting agent.

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# Обоснование температурного режима цикла пенной сепарации

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***Реферат***

***Цель работы*** *заключалась в определении оптимальных температурных режимов операций пульпоподготовки и пенной сепарации алмазосодержащих кимберлитов, обеспечивающих максимальное извлечение алмазов при высокой селективности процесса.*

***Методология.*** *Основными методами исследований выбраны электронно-зондовый рентгеноспектральный анализ, ИК-спектрофотомерия и измерения краевых углов смачивания капли собирателя на алмазах или минералах. Технологические исследования проводились на установке пенной сепарации.*

***Результаты.*** *В результате проведенных исследований показано, что при подогреве исходного питания цикла пенной сепарации до температуры 80–85 °С достигается эффективная очистка алмазов от гидрофильных покрытий, что приводит к восстановлению их природной флотируемости. Результатами измерения краевых углов смачивания определен интервал температур в операции реагентного кондиционирования исходного питания, составляющий 30–40 °С. Показано, что в данном температурном интервале достигается максимальная гидрофобность алмазов и не происходит существенного увеличения гидрофобности минералов кимберлита. Лабораторными опытами показано, что наилучшие результаты процесса пенной сепарации достигаются при поддержании в операции кондиционирования исходного питания с флотационными реагентами температуры 30–38 °С. Результатами флотационных исследований с применением в качестве базовых собирателей мазута флотского Ф-5, а также его компаундов с дизельной фракцией и Маччобинской нефтью определена оптимальная температура непосредственно в процессе пенной сепарации, составляющая 14–24 °С.*

*При анализе результатов проведенных исследований предложен и апробирован температурный режим, включающий нагрев питания пенной сепарации перед операцией обесшламливания и кондиционирования с флотационными реагентами до 85 °С и последующее использование накопленного тепла в операциях кондиционирования питания пенной сепарации (30 °С) с реагентами и непосредственно технологического процесса пенной сепарации (18 °С).*
***Перспективы применения технологии.*** *Результатами испытаний выбранного температурного режима процесса пенной сепарации на стендовой установке показана возможность повышения извлечения алмазов в концентрат на 2,3–4,5 % при использовании применяемых и перспективных собирателей, включая мазут флотский Ф-5 и компаунды на его основе. Разработанный режим рекомендован к промышленному освоению в цикле пенной сепарации на обогатительных фабриках АК «Алроса».*

***Ключевые слова:*** *алмазы; кимберлиты; пенная сепарация; собиратель; кондиционирование; гидрофобность; тепловая обработка.*

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