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**MINERAL PROCESSING  
OF NONFERROUS METALS**

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## **Recovery of Silicon and Iron Oxides from Alumina-Containing Sweepings of Aluminum Production**

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**Abstract**—The method of recovery of contaminating components from sweepings of the aluminum production for their further return into the electrolyzer is proposed. To concentrate the material, the following processing flowsheet is proposed: milling—classification—reverse flotation—thickening. For the most complete removal of silicon and iron oxides during the flotation of sweepings, the Flotigam 7266 flotation reagent produced by Clariant (Germany), which is a mixture of primary fatty alkyl amines, is used. To remove carbon particles, the combination of pine oil in a mixture with kerosene is used. Flotation is performed using a FML 0.3 flotation machine. The initial material, chamber product, and tails are analyzed for the content of carbon and aluminum, iron, and silicon oxides using X-ray spectral (XSA), X-ray phase (XPA), and chemical analyses. It is established that processing the total material mass does not make it possible to acquire a product with an acceptable content of silicon and iron oxides. Based on the XPA of various fractions of the initial material, it is proposed to process the material fractions containing the minimal amount of contaminating substances (carbon and silicon and iron oxides). Two fractions are selected for processing using the flotation method by the XRS results of various material fractions:  $-0.071$  mm and  $+5.0$  mm. When processing the first of them, the chamber product of the acceptable quality is acquired. A product with a high content of alumina and fluorinated components at low carbon and iron oxide concentrations but a considerable amount of silicon oxide is acquired from a coarse electrolyte-containing fraction ( $+5.0$  mm). The further use of this product is possible to fabricate aluminum–silicon alloys.

**Keywords:** alumina-containing wastes, sweepings, electrolytic aluminum production, flotation of sweepings, purification of sweepings, recycling

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### INTRODUCTION

Dusting and pouring of alumina and electrolyte occur during electrolysis aluminum production through leakages in construction elements of equipment, mechanisms, and motorized machinery. Herewith, the dust formed when loading the feedstock and performing the process operations, which mainly consists of electrolyte and alumina, is distributed on various surfaces in electrolysis building. Coarse particles deposit close to emission sources on working floors of the building, while fine ones either deposit on higher arranged surfaces or are exhausted from electrolysis building into the atmosphere [1, 2].

The alumina-containing material taken from the zero level of electrolysis buildings (sweepings) either is

collected, passes the sieving stage, and is returned into the production for smelting at acting electrolyzers with self-roasted anodes or is exhausted on landfills of industrial wastes due to substantial contamination with salts and silicon and iron oxides, which lowers the technical-and-economical performance of electrolysis [3]. For example, up to 5000 t/yr of alumina-containing wastes unacceptable for secondary use in electrolysis are formed during the operation of dust-removing machines (DRMs) at AO RUSAL Krasnoyarsk. This results in the intense filling of the plant slime fields, which consequently require constant purification or reconstruction. In addition to costs for these measures, there is a series of obligatory “environmental” fees for the arrangement of wastes. Here-

with, the environmental stress in the region increases each year, which requires an immediate solution on the development and introduction of wasteless technology in aluminum production plants [4].

The feedstock costs constantly grow and their fraction in the prime cost of electrolytic aluminum is considerable, while the policy of the Government of the Russian Federation in the field of formation and storage of production wastes toughens. Therefore, it seems reasonable to search for technologies of the recovery of contaminating components from alumina-containing material of DRMs with the purpose of its further return into the electrolyzer. Herewith, the main goal is a decrease in the weight fraction of impurities of iron to 0.2% (recalculated to  $\text{Fe}_2\text{O}_3$ ), silicon to 0.3% (recalculated to  $\text{SiO}_2$ ), and carbon, which will make it possible to return this material into electrolysis production.

Various manufacturing flowsheets are proposed for recycling electrolyte-containing wastes of aluminum production [5–16]; some of them are implemented in industry. The purification technology of removal impurities from the DRM material was not in fact considered, because definite difficulties appear due to variables of the chemical and granulometric compositions and, correspondingly, the distinction of the properties of this material even of the same category, which complicates its recycling possibilities. Our proposed processing technology of the DRM material is similar to the flowsheet implemented for the recovery of the fluorine–alumina concentrate from solid wastes of gas purification and coal froth into the production at the production area of fluoric salts (UPFS) of OAO RUSAL Bratsk: milling–classification–reverse flotation–thickening [16].

An increase in quality of the fabricated chamber flotation product of sweepings due to the most complete removal of silicon and iron oxides can be attained with the help of selectively acting combinations of cation collectors (amines). For example, the authors of [17–20] showed the efficiency of their use to remove silicates during the reverse flotation of ferrous quartzites and iron ore concentrates in deep depression conditions of iron minerals with starch. Herewith, the investigation into electrokinetic properties of iron silicates and oxides showed that the applied collectors are actively adsorbed on them without the depression of iron minerals with alkali starch [17–19]. Ferrous silicates have close flotation characteristics with iron oxides, which gives grounds to assume the efficiency of removal with amines into the froth product.

In the course of this study, we performed the reverse flotation of sweepings during the use of the Flotigam 7266 flotation reagent produced by Clariant (Germany), which is a mixture of primary aliphatic amines with the general formula  $\text{R-NH}_2$ , as the cation collector.

A high and almost nonselective adsorption ability of carbon particles provides a wide choice of suitable flotation reagents under the condition that the latter

promote surface hydrophobization, do not destruct air bubbles, and cause no floatability of fluorine-containing and alumina-containing particles. Carbonaceous particles are able to adsorb substances dissolved or emulsified in water. Herewith, the amount of absorbed reagent is determined by their porosity and size of molecules or emulsion drops rather than by the specificity of its interaction with the carbon surface. It was established that the flotation of carbon particles is performed with the maximal efficiency with the combination of two types of reagents, notably, surfactants with a heteropolar molecular structure and apolar substances low-soluble in water. The application of turpentine, pine oil, or flotation oil in a mixture with kerosene is most efficient herewith from the viewpoint of lowering the product prime cost [5].

This study was targeted at the development of the process flowsheet of purification of the DRM material to remove impurities (iron and silicon oxides) with the formation of the fluorine–alumina concentrate and evaluation of the production reasonability of processing this material.

## EXPERIMENTAL PROCEDURES AND CHEMICAL ANALYSIS

Investigations were performed in laboratory conditions. DRM sweepings of AL RUSAL Krasnoyarsk were selected as the material under study.

### *Preparation of Sweepings to the Experiment*

The sieve analysis of sweepings (Table 1) showed an essential amount (80%) of the coarse and medium fractions +0.071; therefore, in order to perform the flotation, the material was milled for 1 h in a ball mill in an amount of 2 kg of sweepings per 8 kg of balls. This resulted in a material in which the part of fraction –0.071 increased to 80%.

### *Preparation of the Flotigam 7266 Aqueous Solution*

When performing the flotation, the Flotigam 7266 aqueous solution (5 g/L) was used; in order to prepare it, the flotation reagent was dissolved in distilled water at  $\sim 80^\circ\text{C}$  under continuous stirring for 30–40 min.

### *Flotation Process*

This process was performed using a FML 0.3 flotation machine with a chamber for 0.3 L. The sweeping charge was selected depending on the S : L ratio and mixed with water in an amount of 100 cm<sup>3</sup> in a pan at room temperature. The pulp was quantitatively transported into a flotation machine chamber and water was poured to the required volume with manual stirring. After the flotation machine was switched on, the pulp was stirred without air supply. The Flotigam 7266 solution was introduced into the pulp by a pipette in a required amount after 2-min stirring with the addition

**Table 1.** Granulometric and phase compositions of sweepings before and after milling

Fraction, mm	Average fraction content, g	Phase composition, wt %					
		Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Amorphous part*	C**	F***
Before milling							
Phase composition of the total mass		47.49	3.68	0.50	23.37	7.2	6.48
+10	0.81	30.58	0.45	0.09	28.71	9.8	16.91
-10 + 5.0	5.56	35.99	3.69	0.23	17.60	5.4	18.04
-5.0 + 2.0	7.39	18.51	3.66	0.69	44.57	39.5	11.29
-2.0 + 0.5	21.51	18.32	3.33	0.63	39.36	32.5	14.03
-0.5 + 0.071	44.72	52.71	3.27	1.03	19.46	5.4	6.16
-0.071 + 0.045	17.51	65.81	1.18	0.53	13.99	3.4	3.81
-0.045	2.49	64.22	1.53	0.71	13.27	1.8	4.57
After milling							
-10 + 5.0	5.24	Not determined					
-0.5 + 0.071	9.04	Not determined					
-0.071 + 0.045	45.38	Not determined					
-0.045	39.64	Not determined					

\* Amorphous part of the material (solid substance) having no crystalline structure and not identified by XPA.

\*\* According to the chemical analysis data.

\*\*\* Recalculated to elemental fluorine.

of kerosene and pine oil. The value of pH of the solution was held at a level of  $9.0 \pm 0.5$ .

After pulp contacting with reagent for 1 min, the air supply was switched on and the froth products started to be removed. This process was continued throughout the entire flotation time. The specified pulp level in a chamber was supported by the periodic addition of water into it.

Upon finishing the flotation process, the chamber product was filtered using the Buchner funnel, and its quality was visually evaluated. The chamber product was dried in a drying oven at 120°C to a constant product weight. The hygroscopic humidity was determined separately and was not included into the sum of analysis results.

#### *Analysis Methods of the Chamber and Tail Products*

The dried material was analyzed for the content of carbon and aluminum, iron, and silicon oxides. The qualitative and quantitative analyses of the chamber and tail products were performed using X-ray spectral (XSA), X-ray phase (XPA), and chemical methods.

The carbon content in sweepings was evaluated according to the procedure of determining the amount of the coal froth in the electrolyte of aluminum electrolyzers. The method is based on the dissolution of the charge of the material under study in the hydrochloric acid solution with the subsequent isolation and weighing of the dried carbon-containing precipitate.

The Al<sub>2</sub>O<sub>3</sub> content was determined by dissolving the electrolyte charge in the hydrochloric acid solution in the presence of aluminum sulfate and isolation and weighing the calcined aluminum oxide precipitate according to the procedure attested at the laboratory of AO RUSAL Krasnoyarsk. The method of evaluating the content of elemental fluorine is based on the decomposition of the analyzed sample by fusion with potassium carbonate (sodium carbonate) with the addition of silicon oxide, leaching the fusion cake, transferring fluorine into a ternary complex with alizarin complexon and lanthanum, and measuring the optical density of the solution [21]. The determination of the iron (III) oxide content is based on its deposition in a form of Fe(OH)<sub>3</sub> by ammonium hydroxide [22], while that of silicon oxide is based on coagulation by decomposition of the sample by fusion with anhydrous soda and subsequent deposition of silicic acid with gelatin upon heating from concentrated solutions [23].

## RESULTS AND DISCUSSION

### *Analysis of the Granulometric and Chemical Compositions of Sweepings*

According to the results of the chemical analysis of the milled material, the initial carbon content in sweepings was 7.2 wt %<sup>1</sup> (Table 1). Recalculation to the elemental composition of sweepings (not allowing

<sup>1</sup> Here and below in the text, the content of components is given in wt %.

for metallic aluminum found by XSA) on the phase composition established that the alumina content in the material is ~47.5% on average, while that of iron and silicon oxides is 0.5 and 3.68%, respectively.

The sieve and X-ray spectral analyses of sweepings (Table 1) showed that various fractions of this material are presented by various compositions. For example, numerous pieces of the solidified electrolyte and coal material (most likely, cleavages from the anode), as well as aluminum globules, are observed in a coarse fraction (+5.0 mm) of the sample of sweepings. This fraction of the material contains a rather large amount of alumina (30–36%) and fluorinated components (16–18% recalculated to elemental fluorine).

The medium fraction (–5.0 + 2.0 mm) is coal froth with a high carbon concentration reaching 40%. Fraction –2.0 + 0.071 mm contains a considerable amount of silicon and iron oxides at a relatively low alumina content. A high content of silicon and iron oxides (3.3–3.7 and 0.6–1.0%, respectively) is found in fraction –5.0 + 0.071 mm according to the XSA data upon a relatively low alumina concentration in it.

A fine fraction (–0.071 mm) mainly consists of alumina (~65%) and coal froth. The material contains numerous fine and medium-size aluminum globules. The composition of a fine fraction corresponds to the usually represented composition of sweepings containing 65% alumina and of about 1.0–1.5 and 0.5–0.7% silicon and iron oxides, respectively.

According to the XPA data (Table 2), a considerable part of fluorine contained in the material of this batch enters the composition of cryolite and chiolite and aluminum oxide is mainly presented by corundum; silicon and iron enter the composition of native oxides SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Fe<sub>3</sub>O<sub>4</sub>. Albite is also present in the material in the amount of 2.13%. However, the XPA showed a high content of various phases and mutual peak overlap, which introduced a considerable error into the results.

#### Selection of Consumption of Flotation Reagents

The range of working concentrations of the Flotigam 7266 flotation reagent was selected based on producer recommendations: the consumption of 0–150 g/t with a step of 50 g/t. Starting from investigations into the efficiency of the coal froth at various consumptions of flotation reagents [5], optimal results were acquired for the mixture of pine oil with kerosene in ratio 1 : (8–10). Therefore, to perform the flotation of DRM sweepings, we selected the ratio of pine oil with kerosene of 1 : 10.

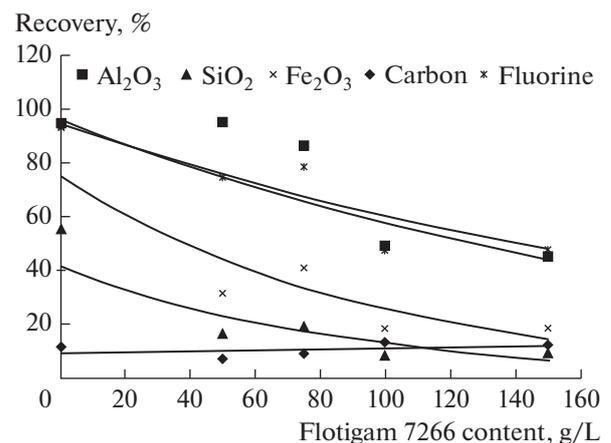
Based on the above, we determined flotation modes of the material (Table 3) at which the data presented in Table 4 and in the Figure 1 were attained (the yield of the chamber product and the content and recovery of C, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and F into the chamber product).

**Table 2.** Data of semiquantitative analysis of sweeping without the content of metallic aluminum

Phase	Name	Content, %
Na <sub>3</sub> AlF <sub>6</sub>	Cryolite	27.12
Na <sub>5</sub> Al <sub>3</sub> F <sub>14</sub>	Chiolite	10.80
Al <sub>2</sub> O <sub>3</sub>	Corundum	47.49
Na <sub>2</sub> Ca <sub>3</sub> Al <sub>2</sub> F <sub>14</sub>	–	3.00
Na[AlSi <sub>3</sub> O <sub>8</sub> ]	Albite	2.13
SiO <sub>2</sub>	Cristobalite	0.28
SiO <sub>2</sub>	Quartz	3.91
CaMg(CO <sub>3</sub> ) <sub>2</sub>	Dolomite	1.18
K <sub>2</sub> NaAlF <sub>6</sub>	Elpasolite	0.73
Na <sub>2</sub> Ca <sub>3</sub> Al <sub>2</sub> F <sub>14</sub>	–	1.66
MgSO <sub>4</sub>	–	1.13
Fe <sub>2</sub> O <sub>3</sub>	Hematite	0.35
Fe <sub>3</sub> O <sub>4</sub>	Magnetite	0.21
KO	–	2.422

It is established that the content of alumina and fluorinated components (recalculated to elemental fluorine) in the chamber product decreases with an increase in the Flotigam 7266 consumption from 0 to 150 g/t, while that of silicon and iron oxides increases. This fact explains the acquired abrupt decrease in the yield of the suitable product from 81.1% (without the participation of flotation reagent, Table 1, experiment 1) to 45.6% (with the Flotigam consumption of 150 g/t, experiment 5).

The recovery of alumina into the chamber product with the Flotigam consumption up to 50 g/t remains at



**Fig. 1.** Recovery of C, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and F into the chamber product during the flotation of sweepings with various consumptions of the Flotigam 7266 flotation reagent.

**Table 3.** Flotation modes for determining the consumption of the Flotigam 7266 flotation reagent

Experiment no.	S : L	Weight of the chamber product, g	Flotation time, min	Flotigam 7266 consumption, g/t	Addition, kg/t	
					pine oil	kerosene
1	1 : 10	30	10	0	0.22	2.2
2	1 : 10	30	10	50	0.22	2.2
3	1 : 10	30	10	75	0.22	2.2
4	1 : 10	30	10	100	0.22	2.2
5	1 : 10	30	10	150	0.22	2.2

**Table 4.** Product yield and recovery of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, C, and F into the chamber product and tails during the flotation of sweepings with various consumptions of Flotigam 7266 jointly with pine oil and kerosene (1 : 10)

Experiment no.	Flotigam consumption, g/t	Product	Product yield		Content, g					Recovery, %				
			g	%	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	C	F	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	C	F
1	0	Sweepings	30	100	47.49	3.68	0.50	4.20	6.48	100.00	100.00	100.00	100.00	100.00
		Chamber product	24.33	81.10	55.59	2.52	0.58	1.10	7.47	94.92	55.49	94.37	21.24	93.55
		Tails	5.67	18.90	12.77	8.68	0.15	17.50	2.21	5.08	44.51	5.63	78.76	6.45
2	50	Sweepings	30	100	47.49	3.68	0.50	4.20	6.48	100.00	100.00	100.00	100.00	100.00
		Chamber product	21.03	70.10	64.38	0.90	0.23	0.80	6.92	95.03	17.19	31.98	13.35	74.85
		Tails	8.97	29.90	7.90	10.20	1.13	12.17	5.45	4.97	82.81	68.02	86.65	25.15
3	75	Sweepings	30	100	47.49	3.68	0.50	4.20	6.48	100.00	100.00	100.00	100.00	100.00
		Chamber product	20.37	67.90	60.66	1.07	0.30	1.00	7.53	86.72	19.70	41.45	16.17	78.91
		Tails	9.63	32.10	19.65	9.22	0.90	10.97	4.26	13.28	80.30	58.55	83.83	21.09
4	100	Sweepings	30	100	47.49	3.68	0.50	4.20	6.48	100.00	100.00	100.00	100.00	100.00
		Chamber product	14.75	49.17	47.70	0.67	0.19	2.00	6.28	49.38	8.97	18.75	23.41	47.68
		Tails	15.25	50.83	47.29	6.60	0.79	6.33	6.67	50.62	91.03	81.25	76.59	52.32
5	150	Sweepings	30	100	47.49	3.68	0.50	4.20	6.48	100.00	100.00	100.00	100.00	100.00
		Chamber product	13.68	45.60	47.13	0.84	0.21	2.00	6.61	45.25	10.38	19.08	21.71	46.55
		Tails	16.32	54.40	47.80	6.07	0.74	6.04	6.37	54.75	89.62	80.92	78.29	53.45

the same level, being 95%; the recovery of alumina decreases to 45% with a further increase in the flotation additive consumption to 150 g/t. The recovery of fluorine into the chamber product with an increase in the Flotigam consumption in a range of 0–150 g/t abruptly decreases from 93.5 to 46.5%. Thus, this flotation additive causes the floatability of fluorinated and alumina-containing material particles.

The minimal content and recovery of silicon and iron oxides into the chamber product (the SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> content was 0.67 and 0.19%, respectively, and their recovery was 9 and 19%) are attained with a

Flotigam concentration of 100 g/t, and no further variation in these characteristics was observed with its increase to 150 g/t.

The carbon content in the chamber product increases from 1 to 2% with an increase in the Flotigam concentration from 0 to 150 g/t.

#### *Selection of the Optimal Fractional Composition of the Material*

Based on the results of previous investigations, it was determined that processing the total mass of the DRM material does not make it possible to prepare

**Table 5.** Flotation modes of selected fractions of the DRM material

Experiment no.	S : L	Fraction, mm	$\tau$ , min	Flotigam consumption, g/t	Addition, kg/t	
					pine oil	kerosene
6	1 : 10	+5.0	10	50	0.22	2.2
7	1 : 10	-0.071	10	50	0.22	2.2

**Table 6.** Product yield and recovery of  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ , C, and F into the charged product and tails during the flotation of selected fractions of the DRM material

Experiment no.	Flotigam consumption, g/t	$\tau$ , min	Fraction, mm	Product	Product yield		Content, g					Recovery, %				
					g	%	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	$\text{Fe}_2\text{O}_3$	C	F	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	$\text{Fe}_2\text{O}_3$	C	F
6	50	10	+5.0	Sweepings	30	100	35.99	3.69	0.23	5.40	18.04	100.0	100.0	100.0	100.0	100.0
				Chamber product	10.75	35.83	63.21	0.98	0.13	1.90	32.05	62.93	9.51	20.35	12.61	63.66
				Tails	19.25	64.17	20.79	5.21	0.28	7.35	10.22	37.07	90.49	79.65	87.39	36.34
7	50	10	-0.071	Sweepings	30	100	65.81	1.18	0.53	13.99	3.81	100.0	100.0	100.0	100.0	100.0
				Chamber product	13.68	45.60	89.60	0.35	0.18	2.60	5.21	62.09	13.51	15.55	8.47	62.28
				Tails	16.32	54.40	45.86	1.88	0.82	23.54	2.65	37.91	86.49	84.45	91.53	37.72

the product with an acceptable content of silicon and iron oxides. Therefore, based on the XSA results (Table 2), the two fractions containing the minimal amount of contaminants (C,  $\text{SiO}_2$ , and  $\text{Fe}_2\text{O}_3$ ) were selected for processing: +5 mm (with the highest amount of electrolyte-containing materials and the lowest content of silicon and iron oxides) and -0.071 mm (with the alumina content of about 65%).

The flotation was performed under the following conditions: the concentration of the Flotigam 7266 reagent was 50 g/t and the flotation duration was  $\tau = 10$  min (Table 5). The yield of the chamber product and the content and the recovery of C,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ , and F into the chamber product during the flotation are presented in Table 6.

Processing the alumina-containing fraction -0.071 mm of the DRM material resulted in the preparation of the chamber product with rather high alumina (of about 90%) and elemental fluorine (of about 5%) concentrations. We succeeded to lower the content of silicon and iron oxides to 0.35 and 0.18%, respectively. When processing the coarse electrolyte-containing fraction (+5.0 mm), the product contained a large amount of alumina and fluorinated components at a low content of carbon and minimal content of iron oxide (0.13%). However, a considerable amount of silicon oxide (of about 1.0%) is present in it in this case. The further use of this product is possible for the pro-

duction of aluminum-silicon alloys or ceramic materials [15, 24].

## CONCLUSIONS

(i) To concentrate sweepings of dust-removing machines, the following processing flowsheet is proposed: milling-classification-reverse flotation-thickening. A collector based on a mixture of primary fatty alkyl amines-Flotigam 7266 produced by Clariant (Germany)-is used for the more complete recovery of iron and silicon oxides from the material.

(ii) The composition of the sweeping sample is determined by XPA. It is established that a considerable part of elemental fluorine contained in the material enters the composition of cryolite and chiolite, aluminum oxide is mainly presented by corundum, and silicon and iron enter the composition of native oxides  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$ ; in addition, a low content of albite is found.

(iii) Flotation of the total material mass does not make it possible to prepare the product with an acceptable content of silicon and iron oxides. It is recommended to classify the material before its milling with the selection of material fractions containing the minimal amount of contaminants (C,  $\text{SiO}_2$ , and  $\text{Fe}_2\text{O}_3$ ). Based on qualitative and quantitative analyses of the fraction, the alumina-containing fraction

–0.071 mm and electrolyte-containing fraction +5.0 mm are selected for further investigations.

(iv) Processing the alumina-containing fraction – 0.071 mm of the material resulted in the formation of a chamber product with acceptable concentrations of silicon and iron oxides and a high content of alumina (of about 90%) and elemental fluorine (of about 5%).

(v) When processing the coarse electrolytic fraction +5.0 mm, the chamber product contained a considerable amount of silicon oxide, which makes it possible to use it for the production of aluminum–silicon alloys.

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