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Results of Researches with Revealing of Technological Parameters of Processes of Recycling and Neutralization of the First and Second Cut of the Spent Lining of Electrolyzers for Reception of Aluminum Fluoride by Pyrolytic and Hydro Chemical Method

Kondratiev V.V.[1], Petrovskiy A.A.[2], Ershov V.A.[3], Sysoeva T.I.[4] and Karlina A.I.[5]

[1]PhD, Head of Innovative Technologies Center, Irkutsk National Research Technical University,

[2]Senior Researcher of Innovative Technologies Center, Irkutsk National Research Technical University,

[3]Associate Professor, Ph.D., Irkutsk National Research Technical University,

[4]Researcher of Innovative Technologies Center, Irkutsk National Research Technical University,

[5]Deputy Head of Innovative Technologies Center, Irkutsk National Research Technical University,

[1,2,3,4,5] Suit T-202, Irkutsk National Research Technical University, 83, Lermontov street, 664074, Irkutsk, Russia.

Abstract

Leaching is carried out in a thermostat with a stirring device using a fluoroplastic reactor. The thermostat is preheated to the temperature providing the set temperature in the reactor (determined experimentally due to the low thermal conductivity of the fluoroplastic). A solution of a given composition is prepared and the reactor is placed in a thermostat. After reaching a predetermined temperature in the reactor, a portion of the refractory lining is poured into it and a stirring device is added to ensure intensive mixing. After a predetermined time, the suspension is filtered. The precipitate is washed on a filter and dried in an oven at 105-1100 ° C. Wet and dry sediments are weighed together with a filter of known weight.

For flotation, the input product undergoes preliminary repulpation and conditioning. For these operations, a cavitation installation is used in which mixing with water and adding the necessary reagents occurs. The next stage, the finished pulp is fed to the first stage of flotation in the first two-chamber flotation machine, then, the foam product of the first stage of flotation is transferred to the second two-chamber flotation machine for further enrichment. The final stage, the foam product of the second stage of flotation is transferred to a single-chamber flotation machine for the final stage of enrichment.

Keywords: aluminum electrolytic; lining waste; recycling, flotation.

INTRODUCTION

The spent lining (SPL) of aluminum electrolyzers contains significant amounts of carbon, refractories, fluorides. In addition, in the SPL contains metals, nitrides, carbides, hydroxides, carbonates, and free cyanides and iron cyanides. According smelters data, composition of SPL varies within the following ranges (Table. 1).

Table 1: Fluctuations in the composition of PF

Component	Content, % wt.
Carbon	9,6 – 51,0
Sodium	7,0 – 20,0
Aluminum	4,7 – 22,1
Fluorine	6,0 – 18,9
Calcium	1,1 – 2,9
Lithium	0,3 – 1,1
Magnesium	0,3 – 0,9
Silicon	0,0 – 12,3
Iron	0,3 – 2,1
Sulfur	0,1 – 0,3
Cyanide	0,02 – 0,44

Water leaching

Relevance and purpose

Thermal insulation or refractory lining is an important structural part of the C-8B, S-8BM, S-8BME electrolytic cells installed at the Krasnoyarsk Aluminum Smelter.

In the process of exploitation, the lining undergoes thermal, electrochemical and chemical effects of aggressive fluorine-containing electrolysis, which leads to partial destruction, impregnation of fluoride salts and the formation of cyanides.

The amount of spent refractory lining reaches 20-45 kg / t of A1 per one cell. In connection with the content of cyanides and fluorides in the majority of foreign countries, it belongs to the 1st class of danger (highly hazardous waste) in addition to the refractory part accounts for about 25.0% of all losses of fluorine.

In Russia, almost all the refractory part is stored in specially equipped dumps. In Russia, works on the composition, neutralization and disposal of valuable components in the last decade have not been carried out.

The purpose of this work is to obtain the initial data and technological parameters of neutralization and utilization of valuable components by a hydrochemical method for performing a comparative analysis of thermal, flotation and hydrochemical methods of processing.

Characteristics of hydrochemical waste processing.

Selected samples, after determining the elemental and phase composition, are crushed and grinded to specified parameters. Grinded samples are leached with various solvents for specified parameters. Based on the analysis of residues and solutions, optimal processing conditions are determined and conclusions are drawn about the expediency of further use of the products obtained. The main directions are:

- water leaching;
- alkaline leaching;
- acid leaching;
- salt leaching;

Variable parameters: temperature, duration, ratio S: L, granulometric composition.

Research performing.

Means of measurement, auxiliary equipment, reagents.

Tiger s8 spectrometer;

The Shimadzu XRD700 diffractometer;

Electronic scanning microscope JEOL JIB Z-4500;

Analytical equipment for determination of cyanides according to HDPE F 16.1: 2: 2.2: 2.3: 3.70-10; PH-meter;

Analytical balance;

Drying cabinet DC 80-01;

Thermostat TERMEX M01;

Fluoroplastic reactor with a cover;

Stirring device ESRO ES-8300;

Installation for filtration (vacuum pump, Bunsen flask, Buchner funnel);

Press the manual;

High pressure press;

Press Herrog HTP-40/60;

Chopper - planetary mill FRITCSH pulvezzisette7;

Porcelain cups for evaporation;

Filter paper;

Reagents NaOH, Na_2CO_3, Ah (SO_4) $18H_2O$, YCl, H_2SO_4 of marks "Pure for analysis" and "Chemically pure";

Water tap and distilled.

Preparation of samples for research.

For the tests, samples of the refractory part of the spent lining of the S8BM electrolyzers of the Krasnoyarsk Aluminum Smelter with a long service life are used. Samples are taken throughout the area of the bottom. Each point is analyzed separately, after which an average sample is formed. In total, three types of samples are formed: impregnated chamotte, lenses and diatomite. Large pieces of samples are crushed by a press of high pressure, after which they are crushed and grinded to the specified parameters.

All samples of the refractory are analyzed:

Elemental analysis - C, F, Na, Ca, Mg, Fe, Si, S, CN.

Phase analysis - NaF, Na_3AlF_6, $Na_5Al_3F_14$, Al_4C_3; SiO_2.

Conduct leaching.

Leaching is carried out in a thermostat with a stirring device using a fluoroplastic reactor. The thermostat is preheated to the temperature providing the set temperature in the reactor (determined experimentally due to the low thermal conductivity of the fluoroplastic). A solution of a given composition is prepared and the reactor is placed in a thermostat. After reaching a predetermined temperature in the reactor, a portion of the refractory lining is poured into it and a stirring device is added to ensure intensive mixing. After a predetermined time, the suspension is filtered. The precipitate is washed on a filter and dried in an oven at 105-1100 ° C. Wet and dry sediments are weighed together with a filter of known weight.

Analytical support:

Measurement of pH of the initial solution and filtrate

Elemental analysis - C, F, Na, Ca, Mg, Fe, Si, S, CN

Fase analysis - NaF, Na_3AlF_6, $Na_5Al_3F_{14}$, Al_4C_3; SiO_2.

Analysis of the solution - pH, NaF, Al_2O_3, $NaSO_4$, Na_2CO_3, Na_2O, SiO_2, CN.

Experiment plan and management factors.

Table 2: Experiment plan and management factors

№ п/п	Solvent. Management Factors	Levels of factors			Range of variation
1	Water exhaust				
1.1.	S: L basic by weight	2:1	6:1	10:1	4:1
1.2.	Temperature, degree C		80	95	15
1.3.	Duration, min.	30	60	90	30
1.4.	Degree of grinding, mm		-0,2	-1,0	0,8
2	Caustic solution NaOH				
2.1.	S: L basic by weight	5:1	10:1	15:1	5:1
2.2.	Concentration of NaOH solution g / dm3	5,0	10,0	15,0	5,0
2.3.	Temperature, degree C		80	95	15
2.4	Duration, min.		60	300	240
2.5.	Degree of grinding, mm		-0,2	-1,0	0,8
3	Solution of sulfuric acid H_2SO_4				
3.1.	S: L basic by weight	5:1	10:1	15:1	5:1
3.2.	Concentration of H_2SO_4 solution g / dm3	5,0	10,0	15,0	5,0
3.3.	Temperature, degree C		80	95	15
3.4.	Duration, min.	30	60	120	60
3.5.	Degree of grinding, mm		-0,2	-1,0	0,8
4	Solution of aluminum sulfate Al $(SO_4)_3$				
4.1.	S: L basic by weight	5:1	10:1	15:1	5:1
4.2.	The concentration of Al $(SO_4)_3$ solution g / dm3	5,0	10,0	15,0	5,0
4.3.	Temperature, degree C		80	95	15
4.4.	Duration, min.	30	60	120	60
4.5.	Degree of grinding, mm		-0,2	-1,0	0,8

Processing of refractory part

The technology of processing the refractory part is the least developed. For neutralization, heat treatment, treatment with sulfuric acid and lime is used. In addition, it is possible to use in production of cement, bricks and building materials.

Water leaching

It is based on the solubility of sodium fluoride with limited solubility of other components including silicon. Water leaching allows reducing the content of fluorine and sodium in waste by 40-70%, followed by the utilization of fluorosols from solutions. In addition, water leaching is combined with neutralization of cyanides due to natural oxidation and the introduction of neutralizing additives. Two blocks of research have been completed: the first unit - water leaching of fireclay bricks, the second block - water leaching of the mixture of refractory waste in an approximate proportion of the content with subsequent flotation.

When water leaching chamotte bricks, which are visually undamaged, the solution contains several milligrams of fluoride and is not subject to processing. Obviously, fluoride is not sorbed on chamotte. The results of aqueous leaching with subsequent flotation of a mixture of refractory wastes consisting of a lens, reacted bricks and fireclay bricks are shown in the experiment sheet and balances of leaching and flotation.

Flotation experiment of mixed refractory lining with preliminary leaching (lens + impregnated chamotte brick + crushed fireclay bricks)

Table 3:

1. Composition of the initial mixture of refractory lining parts

	O	F	Na	Mg	Al	Si	K	Ca	Ti	Fe	Total
SPLR900	46,82	8,50	8,42	0,87	14,38	18,21	0,55	0,55	0,50	1,20	100

2. Leaching

Sample	Quantity		№ sample
SPLR700 -50%+SPLN712-25%+SPLN719-25%	50	g	SPLR900
H_2O	500	ml	
Temperature in the reactor	90	degrees, C	
Holding time	60	min	
The total weight of the initials	50	g	

3. Filtering, washing on the filter, drying, evaporation,

PH of the filtrate	9		
Filtrate weight	666,6	g	
Filter residue, wet	56,4	g	
Filter residue, dry	46,7	g	**SPL1021**
Vapor	3	g	**SPL1022**
Total Product Weight	49,7	g	
Difference	-0,3		

Table 4: The water leaching balance of the mixed refractory lining
(lens + impregnated chamotte brick + crushed fireclay brick) per 1 ton of lining

Income					Outcome				
№	Name	F, kg/%	Na, kg/%	Si , kg/%	№	Name	F, kg/%	Na, kg/%	Si,kg/%
1	Refractory lining 1 ton	85/100	84,2/100	182,1 / 100	1	Sediment	57.7/67.88	53.2/69.0	173.88/96.79
					2	Solution	27.7/32.59	25.6/30.4	3.12/1.71
					3	Discrepancy	-0.4/-0.47	0.5/0.6	7.2/ 1.5
	Total	85/100	84,2/100	182,1 / 100		Total	85/100	84,2/100	182,1/100

The balance of flotation of the mixed refractory lining after preliminary aqueous leaching (lens + impregnated chamotte brick + crushed fireclay brick) per 1 ton of lining

Flotation conditions

1. Reagent consumption: Oleic acid - 0.2 g. per 100 g. of lining
Pine oil - 40 mg. per 1 liter of water

2. Composition of the initial mixture of refractory lining parts

	O	F	Na	Al	Si	K	Ca	Ti	Fe	Total
SPL1021	57,31	4,94	5,69	13,86	15,65	0,55	0,53	0,41	1,06	100

3. Flotation machine: FL-67

4. Fraction of refractory lining: -0.2 mm (table 5).

Table 5

Income					Outcome				
№	Name	F, kg/%	Na, kg/%	Si , kg/%	№	Name	F, kg/%	Na, kg/%	Si , kg/%
1	Refractory lining 1 ton	57.7/ 1 0 0	56.9/100	186/100	1	Foam product	7.63/13.2	9.6/16.87	12.53/6.74
					2	Solution	9.52/16.5	3.4/5.9	0.85/0.45
					3	Chamber product	39.35/68.3	41.9/73.6	177.62/92.76
					4	Discrepancy	1.2/2.0	2.0/3.63	1.0/0.05
	Total	57.7/100	56.9/100	186/100		Total	57.7/100	56.9/100	186/100

Flotation experiment of mixed refractory lining with preliminary leaching (lens + impregnated chamotte brick + crushed chamotte brick)

1. Composition of the initial mixture of refractory lining parts

	O	F	Na	Mg	Al	Si	K	Ca	Ti	Fe	Итог
SPLR900	46,82	8,50	8,42	0,87	14,38	18,21	0,55	0,55	0,50	1,20	100

2. Weight of SPLR900: 100 grams

3. Reagent consumption: Oleic acid - 0.2 g. per 100 g of lining

Pine oil - 40 mg. per 1 liter of water

4. Flotation machine: FL-67

5. Results:

Foam part				
Dry foam part	**3,7**		**SPL1009**	
Filtrate weight	160			
PH of the filtrate	9			
Vapor	**0,5**		**SPL1010**	

Solution				
Residue on the filter	**2,7**		**SPL1011**	
Filtrate weight	1210			
PH of the filtrate	9			
Vapor	**3,4**		**SPL1012**	

Chamber product				
CP washed and dryied	**76,3**		**SPL1013**	
Filtrate weight	361			
PH of the filtrate	8	-	-	-
Vapor	**0,3**	**-**	**SPL1014**	

Mass of reaction products	**86,9**
Weight of the vapor	**4,2**
Discrepancy	**-13,1**

Table 6: SEM analysis by means of scanning electron microscopy (averaged values):

	O	F	Na	Mg	Al	Si	K	Ca	Ti	Fe	Total
SPL1009	33,46	19,91	9,21	0,19	15,45	15,88	0,71	2,61	0,63	1,96	100
SPL1010	4,63	46,09	48,45			0,48		0,12			100
SPL1013	45,48	7,26	8		14,57	20,6	0,7	0,61	0,74	2,04	100

Flotation of the refractory part

The input product of flotation is ground refractory SPL. Flotation occurs in three stages, for the first and second, two-chamber flotation machines are used, for the third - one-chamber flotation. Each of the stages of flotation is aimed at increasing the concentration of fluorosols in the foam product.

For flotation, the input product undergoes preliminary repulpation and conditioning. For these operations, a cavitation installation is used in which mixing with water and adding the necessary reagents occurs. The next stage, the finished pulp is fed to the first stage of flotation in the first two-chamber flotation machine, then, the foam product of the first stage of flotation is transferred to the second two-chamber flotation machine for further enrichment. The final stage, the foam product of the second stage of flotation is transferred to a single-chamber flotation machine for the final stage of enrichment. The hardware scheme of flotation is shown in Figure 1.

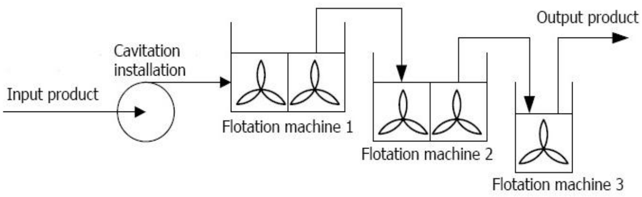

Figure 1: Flotation hardware scheme

The parameters for performing flotation are selected according to the experimental matrix

The first series of experiments on the selection of the optimal S: L ratio showed that the optimal values of the given lie in the range 1: 9 - 1:11. Within this range, the flotation process is most efficient. When out of range, the efficiency is significantly reduced.

The second series of experiments on the selection of the optimum flotation time showed that the optimal time is 15 minutes. A shorter time does not provide sufficient extraction of the target product, longer time does not lead to a significant increase in the yield of the target product.

The third series of experiments on the selection of the optimum amount of added flotation agent - kerosene showed that the optimum concentration of kerosene is 2 kg / t.

The fourth series of experiments on the selection of the optimum amount of added flotation agent - sodium oleate showed that the optimum concentration of kerosene is 2 kg / t.

The fifth series of experiments on the selection of the optimum amount of foaming agent - pine oil showed that the optimum concentration is 25 g / t.

CONCLUSION

To assess the efficiency of flotation, a final series of 10 experiments with optimal parameters was carried out.

As can be seen from the data given in the table, at each stage of flotation, there is a marked increase in the amount of fluorine, sodium and aluminum. The amount of silicon decreases significantly after each stage. Three stages of flotation make it possible to obtain the desired product with the necessary concentration of fluorosols and a low content of silicon dioxide.

The obtained results of the experiments are processed with the derivation of conclusions and the determination of the possibility of patenting. Particular attention is paid to the content of cyanides and fluorides.

In the case of high cyanide content, additional studies are carried out to neutralize cyanides and use iron salts ($FeSO_4$ and others) or other methods.

Based on the data obtained, an environmental assessment of the application of hydrochemical methods is performed in comparison with thermal and flotation methods.

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