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Use of Leaching Cake from Refractory Lining of Dismantled Electrolysers in Cement Production

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***Abstract* – A hydrometallurgical technology has been proposed for processing the refractory part of the lining of aluminum production electrolysers dismantled and disabled for overhaul. Fluorine-containing compounds are transferred to the solution, which allows obtaining cryolite for being used in the electrolysis process. It is recommended to use solid residue from leaching (cake) with a minimum content of alkalis and fluorine as an additive to the mixture for cement production. According to the results of experiments on obtaining portland cement by traditional firing technology, the limiting content of alkali metal oxides (not higher than 4.5% by weight, calculated as Na_2O) in the leaching cake was determined, the optimum content of this additive in the charge is from 5 to 12% depending on chemical composition of lime used.**

Keywords – aluminium production; electrolyser; cathode lining; spent firebrick lining; leaching cake; cement clinker.

I. INTRODUCTION

The aluminum industry is the largest non-ferrous metallurgy sector. The main industrial method for introducing primary aluminum is electrolysis of alumina-cryolite melts (Hall–Héroult process) [1, 2]. One of the drawbacks of this technology is a large amount of solid technogenic products (coal foam, flotation tailings, alumina strips) formed in the process [3-5]. Currently, the main type of solid technogenic raw materials in the production of aluminum is materials for dismantling the cathode device of electrolyzers being waste lining, which is saturated with electrolyte components during the entire service life. Every year in Russia up to 130 thousand tons of lining are dismantled and disconnected for major repairs of electrolyzers. The lining is stored in open landfills, interacts with water and air, forming alkaline fluorine-containing solutions and other toxic compounds. In this regard, the development of technology for processing fluorine-containing materials, in particular, refractory ones for the dismantling of electrolyzers with the objective to extract fluoride salts to return them to the electrolysis process of alumina-cryolite melts is an urgent task [6, 7].

II. PROBLEM SPECIFICATION

The cathode device of the electrolyzer used to produce primary aluminum is a lined bath designed to hold the melt and the cathode metal throughout the bath operating life [8]. Structurally, a cathode of the new electrolyzer consists of the following main parts:

- cathode casing in the form of a rectangular container with straight or inclined sides;

- bottom carbon conductive blocks with metal current-carrying rods (blooms) and side blocks of carbon or silicon carbide materials that make up the outer part of the cathode lining and are in direct contact with the melt;

- refractory (heat insulation) lining.

The refractory part of the cathode lining of the electrolyzer consists of a layer of refractory materials or dry barrier mixtures located under the coal blocks, a layer of thermal insulation materials (based on diatomite, perlite or vermiculite) and a leveling layer of powdered refractory material (most often chamotte crumbs) [9].

The refractory lining consists of natural inorganic materials (based on aluminosilicate bricks) and has a dual purpose, specifically, to resist the chemical and physical effects of the electrolyte and to insulate the bath. However, there are significant differences between them. As a rule, refractory bricks have a density of more than 1 g/cm^3 and, in general, they are more resistant to the effects of melt components. However, they have worse thermal insulation properties than low density materials (0.35-0.8 g/cm^3), which have lower rates of chemical and physical resistance. Bricks with a high content of alumina, that is, alumina-silica refractory materials (chamotte), consisting mainly of mullite ($3Al_2O_3$ $5SiO_2$) and the most resistant to the effects of melt components are used as the refractory materials [9]. Diatomaceous bricks of the types PD-350, PD-400, D-500 [10] and vermiculite of different composition [11] are widely used as thermal insulation materials.

The main purpose of the insulating component of the refractory part is to reduce heat loss to the environment and, as a consequence, to reduce power consumption for aluminum production. In addition, the correct device of the heat-insulating part of the refractory lining enables to maintain the required temperature of the melt and steel casing elements for electrolyzer operation in the optimal mode, increasing a bath operating life.

As a result of thermal and electrochemical factors, sodium and electrolyte vapors interact with the components of the spent lining. As is known, the electrolyte used to produce aluminum on the basis of the Hall–Héroult method consists of cryolite, alumina, and corrective additives, aluminum fluoride and calcium fluoride. In the process of electrolysis, the cathode lining is impregnated with fluoride salts, metallic aluminum and gases dissolved in the electrolyte, with the occurrence of complex physicochemical transformations. Thus, the cathode lining of the electrolysis cell of average power (150-160 kA) when disconnected for being subject to overhaul may contain up to 5 tons of fluorine [12].

The main mechanism of sodium fluoride salts penetration to the refractory layer is capillary flow of the electrolyte through the permeable pores of the bottom coal blocks, interblock and peripheral joints, filled with bottom mass. This is due to the fact that the electrolyte viscosity (from 2.5 to 3.5 mPas) is comparable with the viscosity of water (1.0 mPas) at electrolysis temperatures. In practice, electrolyte fluids are considered to be one of the main reasons for the penetration of salts to the refractory lining in gaps, cracks and other hearth defects caused by firing when starting an electrolyzer. During electrolyzer operation at high temperatures, an electrolyte containing 40–50% fluorine and up to 30% sodium penetrates into the refractory part of the lining, thereby melting the chamotte brick to form lenses and destroying part of the bricks with fluoride [13].

Currently, there are the methods for processing the carbon-containing part of the spent lining, which is most impregnated with electrolyte components, in order to obtain fluoride salts for being used in the electrolysis process [14, 15]. There are practically no studies on effective processing of the refractory part of the spent lining with the objective to obtain cryolite (for its application in the electrolysis process) and solid residue, which can be sold to third-party consumers. Consequently, the purpose of our research was to obtain a leach cake (suitable for the production of a cement clinker) from the hydrometallurgical processing of refractory lining.

III. Object of Research

The spent refractory part is divided into four parts according to layout and composition: a layer under the blocks (a lens and a brick having reacted with electrolyte components), an intact fireclay brick, heat insulation (diatomite, vermiculite or perlite) and chamotte filling. The layer under the blocks is formed by electrolyte that has penetrated through the hearth and dissolved part of the chamotte brick. It represents a monolith of gray, light gray and yellow color interspersed with the following conglomerates of electrolyte components: cryolite Na_3AlF_6, chiolite $Na_5Al_3F_{14}$, alumina Al_2O_3. This part of the non-carbon lining is the richest in fluorine and sodium. The second part is a fireclay brick that has not reacted with electrolyte and sodium. A significant part of the brick is usually not destroyed and contains from 0.2 to 1.5% fluorine. This is due to the resistance of fireclay to fluorine vapor. The third part is heat-insulating materials containing up to 7% of fluorine, which is due to their porous structure and high-surface area. The fourth part is fireclay filling, which in most cases remains unchanged [9].

According to various literature data [16-18], the refractory part contains mullite, sodium fluoride NaF, cryolite, chiolite, silicon oxide SiO_2 (in the form of cristobalite, quartz, tridimite), calcium fluoride CaF_2, aluminum oxide, iron oxide Fe_2O_3, nepheline $NaAlSiO_4$ and albit $NaAlSi_3O_8$. It has been established [19, 20] that nepheline predominates in refractory materials with formed sodium aluminosilicates with an excess of alumina, and albite predominates in the ones with an excess of silica.

The average chemical composition of the refractory part of the spent lining according to different researchers [18, 21, 22] varies considerably in terms of weight (in %), respectively: C is from 0.1 to 6; F is from 5 to 15; Al is from 10 to 18; Na is from 5 to 15; Ca is from 0.5 to 1.0; Si is from 10 to 30; Mg is from 0.2 to 1.0; Fe is from 1.0 to 2.0; others are from 35 to 45. The last mentioned category is represented manly by oxygen. The differences in the chemical composition are due to the fact that the destruction of the refractory of this type of technogenic raw materials is individual for each cell.

We have selected the samples of a refractory part of dismantlement outputs of an electrolyzer (PJSC “RUSAL” Krasnoyarsk in Krasnoyarsk) disconnected to be subjected to major overhaul. A general view of the spent refractory lining is presented in Fig. 1.

Fig. 1. Dismantled refractory part of electrolyzer’s spent lining

Dismantling was carried out at the beating sites in electrolyzers overhaul shops using a so-called wet technology. This technology consists in the fact that after extracting a lumpy electrolyte to accelerate cathode cooling and facilitate disassembly, water is poured into the bath. The water enters into chemical interaction with the electrolyte, aluminum carbide and sodium embedded in the carbon lining, which leads to swelling and destruction of the lining. At the same time, salt slags (which it is possible to obtain fluoride salts from) are formed [23]. Next, the samples were subjected to grinding in SMD 108 crusher (produced in Russia). The chemical composition of the studied samples (according to the results of X-ray fluorescence analysis, performed on a Bruker S8 TIGER spectrometer (Germany), equipped with the software SPECTRAplus and QUANT EXPRESS), is given in Table 1.

As can be seen from the data presented in Table 1, the layer under the coal blocks (fireclay reacted with the electrolyte and the first fireclay layer) is a fireclay brick decomposed under the influence of a penetrated electrolyte melt and contains sodium, aluminum, fluorine and silicon. The second part is chamotte brick (the second and third layer of chamotte). A significant part of the brick remains intact and contains an insignificant amount of fluorine, specifically, from 0.2% to 1.3%. This is due to the resistance of fireclay to fluoride penetration. The third part is thermal insulation (diatomite) and it contains 6.9% of fluorine, which is stipulated by the porous structure of the material as it was stated above.

The main phases recorded in the studied samples of the technogenic refractory lining spent by X-ray analysis with the application of a Shimadzu XRD-7000S powder diffractometer (Japan) are as follows: $3Al_2O_3$, $2SiO_2$, NaF, Na_3AlF_6, $Na_5Al_3F_{14}$, SiO_2 (in the form of cristobalite, quartz, tridymite), CaF_2, Al_2O_3, $NaAlSiO_4$, $NaAlSi_3O_8$.

TABLE I. CHEMICAL COMPOSITION OF THE COMPONENTS OF THE REFRACTORY PART OF THE FINISHED LAYER

Components of spent lining	**Content, % mass**									
	C	***F***	***Na***	***Mg***	***Al***	***Si***	***Ca***	***Fe***	***K***	***Other****
Chamotte reacted with electrolyte	3.20	20.60	17.20	0.12	16.30	16.80	0.70	0.70	0.90	23.48
The first layer of chamotte	2.80	12.80	19.10	0.14	16.00	19.50	0.90	1.50	0.70	26.56
The second layer of chamotte	0.00	1.30	3.60	0.23	16.50	26.10	0.27	0.78	0.44	50.78
The third layer of chamotte	0.00	0.20	2.20	0.30	16.50	26.90	0.68	1.90	0.44	50.88
Diatomite	0.00	6.90	3.00	8.40	4.50	22.40	0.47	7.20	3.00	44.13

a. * Other components are mainly represented by oxygen in the form of Al_2O_3, SiO_2.

Fluorine in refractory spent lining is present in the form of four compounds: NaF, Na_3AlF_6, $Na_5Al_3F_{14}$, CaF_2; sodium fluoride accounts for more than 50% of the total fluorine. Aluminosilicates, calcium fluoride and aluminum oxide are practically insoluble in water, cryolite and chiolite are slightly soluble, but NaF dissolves almost completely until the solubility limit is reached in this water-salt system. Thus, most of the fluorine can be extracted by dissolving NaF during the water treatment of fluorine-containing refractory materials. Cryolite, chiolite and calcium fluoride are almost completely left in the cake under the conditions of water leaching.

As a result of studying the composition and properties of the components of the refractory spent lining, it became necessary to study the complex issues of its processing, namely: studying the influence of the main parameters of fluorine leaching from this man-made material with transfer to sodium fluoride solution and obtaining silica-containing cake suitable for further use in the construction industry, specifically, cement clinker production.

IV. EXPERIMENTAL WORKS

As follows from the above data, all NaF contained in the sample under study goes into solution in the process of water leaching. There is no NaF in the leaching cake, which indicates that under the given conditions of hydrometallurgical processing of the refractory spent lining it passed into the solution completely. Processing of the crushed sample of the refractory spent lining was carried out with distilled water or salt solutions that simulate gas cleaning solutions formed in the process heated to a certain temperature in a fluoroplastic beaker placed in a TERMEX M01 liquid thermostat. Stirring was performed using a BIOSAN MM-1000 top-drive laboratory mixer with a two-bladed attachment. Rotation frequency was set at 800 min^{-1} (circumferential speed of 0.2 m/s), which means that mixing was quite intense and distribution of solid particles in the volume of the reactor was the most uniform. Process parameters are as follows: W: T = 7-11:1, leaching temperature is 60-80°C and leaching time is 360 minutes. After a specified time, the slurry was filtered under vacuum on a Buchner funnel with a double blue ribbon filter. The pH value and concentrations of sodium fluoride, carbonate, and sodium bicarbonate were determined in the leaching solution. With the objective to determine the degree of fluorine extraction, data on its content in the original sample, the cake and the solution after leaching were used. For this purpose, the samples were investigated using X-ray fluorescence (XRF) and X-ray analysis methods.

The solid residue (cake) from the water-treated sample of the refractory part of the lining when dismantling electrolyzers (as a by-product when converting water-soluble sodium fluoride into solution) was proposed to be used in the composition of the raw mix to produce cement clinker. With the objective to study the possibility of its use, we carried out research, the purpose of which was to determine the optimal amount of additive in the leaching cake and to select the parameters for obtaining cement clinker from the obtained raw material mixture.

V. RESULTS

The leaching cake after hydrometallurgical processing of the averaged sample of the spent lining refractory part had the following composition (according to XRD data), % to mass: C was 0.39; F – 3.49; Al – 22.59; Na – 2.61; Ca – 0.92; Si – 23.85; Mg – 0.32; Fe – 0.98; other components made 44.85. The main compounds present in the test sample are (according to x-ray diffraction analysis), % to mass: mullite, nepheline, cristobalite, quartz, fluorite (Fig. 2). Alumina, chiolite, tridymite, albite were also stated in some samples.

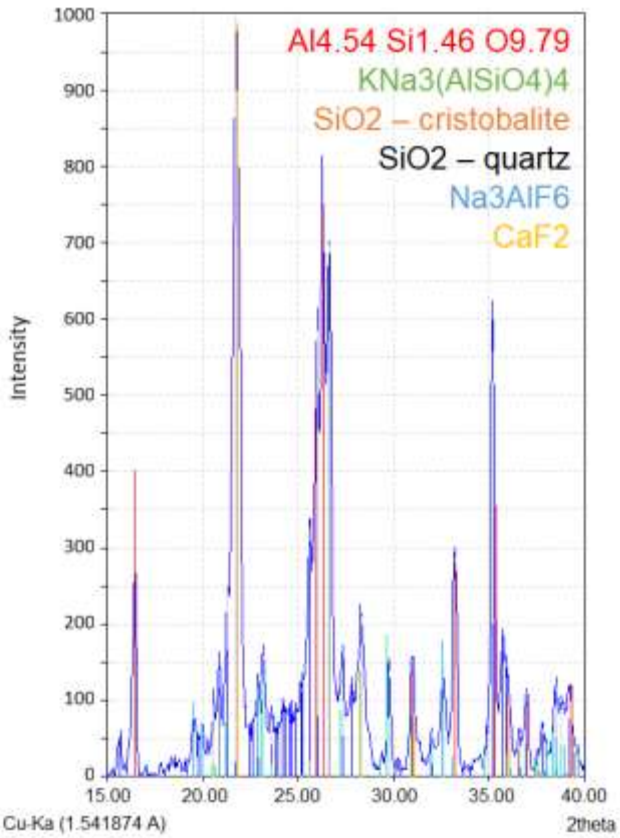

Fig. 2. Diffractogram of sample leach cake

Traditionally, a charge for cement clinker production consists of, % to mass: from 70 to 80 carbonate rocks (limestone); from 15 to 25 clay rocks (clays, marls); from 3 to 5 corrective additives (iron component). The use of the spent lining refractory part as the clay component of raw material mixture without any processing is limited due to the high content of alkali metals in it. Their total content in cement clinker should not exceed 1.2% (in terms of Na_2O) according to GOST 10178-85 "Portland cement and slag portland cement. Technical conditions." But at the same time presence of fluorine in a spent lining reduces the firing temperature of a clinker, reducing energy costs thereby.

The composition and properties of portland cement clinker are predetermined by the following characteristics: clinker chemical composition, saturation coefficient values of silicate ($MSiO_2$) and alumina (MAl_2O_3) modules, and the content of basic clinker minerals.

The content of oxides in the clinker varies in the following ranges, % mass: CaO from 62 to 67, SiO_2 from 20 to 24, Al_2O_3 from 4 to 7, Fe_2O_3 from 2 to 5, MgO, SO_3, other ones from 1.5 to 4. The value of saturation coefficient varies from 0.8 to 0.95, $MSiO_2$ from 1.7 to 3.5, and MA_2O_3 from 1.0 to 3.0. Values of saturation coefficient and modules are determined by the following formulas:

$$KH = CaO - 1.65Al_2O_3 - 0.35Fe_2O_3 - 0.7SO_3/2.8SiO_2;$$
$$M_{SiO2} = SiO_2/(Al_2O_3 + Fe_2O_3);$$
$$M_{Al2O3} = Al_2O_3/Fe_2O_3,$$

where CaO, SiO_2, Al_2O_3, Fe_2O_3, SO_3 denote clinker oxide content, % mass.

The content of basic clinker minerals in an ordinary clinker varies in the following range, % to mass: tricalcium silicate $3CaO{\bullet}SiO_2$ (alit) from 40 to 60; dicalcium silicate $2CaO{\bullet}SiO_2$ (white) from 15 to 35; tricalcium aluminate $3CaO{\bullet}Al_2O_3$ from 4 to 14; tetracalcium aluminoferrite $4CaO{\bullet}Al_2O_3{\bullet}Fe_2O_3$ from 10 to 18.

To confirm the possibility of using cake from water-treated refractory spent lining in cement production, we conducted an experiment on drawing our own raw material composition (with adding leaching cake) and firing it in a SNOL 12/16 type kiln (Russia) to obtain cement clinker. The standard raw material charge of OJSC "Angarskcement" (Angarsk, Irkutsk Region), currently used in cement production, was burned as a control sample in the same furnace, simultaneously with the charge under investigation.

The recommended composition of the raw material mixture was prepared from lime, leaching cake of the refractory part of the spent lining and iron sand. Chemical composition of a leaching cake (51.8% to SiO_2 mass) does not fully correspond to the composition of the clay component of the raw material mixture, which should contain 75% of SiO_2 mass. To increase the silicate component, silica fume (with an average 92% to SiO_2 mass) was used as a corrective additive, specifically, dust from the gas cleaning system of the production of crystalline silicon of JSC Silicon, UC "RUSAL" (the town of Shelekhov, Irkutsk Region). The resulting ratio of leaching cake and silica fume is 3:4. That is, in our raw material blend composition, the clay component is represented by two types of technogenic materials of metallurgical production being leaching cake (from the refractory part of the spent lining dismantled and disconnected for the overhaul of aluminum production electrolyzers) and microsilica (gas cleaning dust when producing metallurgical silicon [24]).

The content of the components in the charge was calculated by the traditional method by setting the values of saturating efficiency (SE) and M_{SiO2}. The charge calculation consists in determining the ratios between its components based on the chemical composition of the raw materials and the required characteristics of the clinker. The following values were set: SE = 0.9 and M_{SiO2} = 2.3. The ratio between the components was calculated, % to mass, respectively: lime 82.90, leaching cake 5.90, silica fume 7.86; quartz sand 3.34. The components were ground in a ball mill to a particle size of -80 μm, the resulting fine mixture was moistened to 8% and pressed into briquettes on a $VP_{10}M$ hand screw press. The briquettes were burned in a furnace together with the control samples with regards to the technological parameters of a tubular rotary kiln of the active roasting shop of OJSC "Angarskcement" (bypassing the drying zone since the charge does not contain excess moisture): from 100 to 800°C for 45 min; from 800 to 1100°C for 50 min; from 1100 to 1300°C for 10 min; from 1300 to 1450°C for 20 minutes.

The clinker specks were ground in a jaw crusher, and then in a ball mill to a particle size equal to -80 μm. Grinding was performed together with an added amount of 5% to mass by $CaSO_4 \cdot 2H_2O$ dihydrate gypsum to prevent false setting. Specimens for performing tests according to GOST 310.1-76 "Cements. General provisions." were prepared from the obtained cements by definition of various characteristics in accordance with GOST 30744-2001 "Cements. Test methods using polyfractional sand". The test results of cements (control cements and cements with the addition of man-made materials) are presented in table 2.

TABLE II. CHARACTERISTICS OF RECEIVED CEMENTS SAMPLES AND TEST RESULTS

Characteristics, measuring units	**Sample view**	
	containing man-made materials	***control***
True density, g/cm^3	3.0	3.03
Pour density, g/cm^3	1.085	1.100
Grinding fineness: sieve residue 0.08%	13.8	13.1
Normal density, % to mass	28.50	25.75
Setting start min.	220	200
Setting end min.	430	470
Water separation rate,%	28.1	29.0
Compressive strength at 28 days point (average result for testing three samples), MPa	29.6	29.8

Studies have confirmed the possibility of using solid residue from fluorine water leaching out of the spent lining refractory part as the clay component of the raw charge for the production of cement clinker. Cement obtained in the laboratory conditions corresponds to the grade PC-300.

Cement brands PC-400 and PC-500 are usually obtained from the charge (control sample) at the factories of JSC "Angarskcement". The reduction of grade of the cements obtained from the mixture containing technogenic materials is due to the low content of alite in a clinker, which is caused by the fact that the samples were fired in a stationary mode in the chamber furnace without providing sufficient contact for the solid particles of the mixture at high temperature. The mixture undergoes this mode in rotating tube furnaces with mixing.

VI. Conclusion

According to our findings, we determined the limiting content of alkali metal oxides should not exceed 4.5% of mass (in terms of Na_2O) in a leaching cake to be recommended for being used in cement production. The optimal content of this additive in raw material mixture was also determined, specifically, from 5 to 12% depending on the chemical composition of the lime used. It was recommended to adjust the silicate component of the silica leaching cake in order to observe the necessary values of the saturation coefficient and modular characteristics of the raw material mixture for cement clinker production.

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