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Physicochemical parameters of a hydrochemical technology employing sodium chloride to obtain cryolite used in aluminium production

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Abstract. The paper aims to study the physicochemical parameters of a hydrochemical technology employing hydrofluoric acid and local mineral resources (sodium chloride) to obtain cryolite used in the electrolysis of cryolite-alumina melts. In order to determine the elemental chemical and phase compositions of initial, intermediate, and final products, titration and X-ray diffraction analysis (using an upgraded Dron-2 unit) were employed. The conducted studies indicate that the proposed process of cryolite production from hydrofluoric acid at 28–30% concentration using aluminium hydroxide and a concentrated sodium chloride solution occurs at 25°C for 10–15 min. The yield of cryolite reaches 87.6%, while ~12% of cryolite remains dissolved in the hydrochloric acid solution. With the temperature rising from 25°C to 95°C, the cryolite yield is shown to decrease from 87.6% to 69.3% due to its higher solubility in the formed hydrochloric acid. The cryolite production process was validated via X-ray diffraction analysis. The analysed sample was found to be consistent with the cryolite reference, i.e., indicating an interaction between sodium chloride and fluoroaluminic acid. The conducted studies served as a basis for developing a process flow diagram of hydrochemical cryolite production using hydrofluoric acid, aluminium hydroxide, and sodium chloride. The conducted studies revealed that the technology of cryolite production employing sodium chloride is easy to implement and cost-effective due to the use of local mineral resources and low energy consumption.

Keywords: aluminum production, cryolite, aluminum hydroxide, sodium chloride, electrolyte, hydrofluoric acid

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МЕТАЛЛУРГИЯ

Научная статья
УДК 66.012.2:66.012.3

Физико-химические аспекты технологии получения криолита, используемого для производства алюминия, гидрохимическим способом с использованием поваренной соли

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Резюме. Цель – изучение физико-химических параметров технологии производства криолита, используемого при электролизе криолит-глиноземных расплавов, гидрохимическим способом с применением производимой плавиковой кислоты и местного минерального сырья – хлорида натрия. Употреблены титриметрический и рентгеноструктурный (с использованием модернизированной установки Дрон-2) методы анализа с целью определения химического элементного и фазового составов исходного, промежуточного и конечного продуктов. Проведенные исследования показали, что процесс получения криолита предлагаемым способом из плавиковой кислоты 28–30% концентрации с использованием гидроксида алюминия и концентрированного раствора хлорида натрия протекает при температуре 25°C в течение 10–15 мин. При этом выход криолита достигает 87,6%, а ~ 12% криолита остается в растворе соляной кислоты в растворенном виде. Показано, что с повышением температуры в интервале от 25°C до 95°C наблюдается снижение выхода криолита с 87,6% до 69,3%, связанное с повышением его растворимости в образующейся соляной кислоте. Достоверность протекания процесса получения криолита с применением поваренной соли подтверждена результатами рентгенофазового анализа. Установлено, что анализируемая проба соответствует эталону криолита, свидетельствуя о взаимодействии хлорида натрия и фторалюминиевой кислоты. На основе проведенных исследований была разработана принципиальная технологическая схема производства криолита гидрохимическим способом с применением плавиковой кислоты, гидроксида алюминия и поваренной соли. В результате проведенных исследований установлено, что технология получения криолита с применением поваренной соли проста в осуществлении и экономически эффективна за счет использования местного минерального сырья и малой энергозатратности производства.

Ключевые слова: производство алюминия, криолит, гидроксид алюминия, хлорид натрия, электролит, плавиковая кислота

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INTRODUCTION

Aluminium is one of the world's most in-demand metals. Due to its low density and high strength, aluminium is widely used in construction, as well as in the manufacture of car parts, aircraft, etc., which results in an annual output of nearly 70 million tonnes of aluminium. For over 120 years, it has been produced using the Hall–Héroult process[8] [1–12].

The electrolyte in electrolyzers used for aluminium production performs several essential

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functions such as charge transfer, dissolution, and mass transfer of electrode products. The proper performance of these functions depends solely on physicochemical properties, namely electrical conductance, viscosity, vapour pressure, solidus and liquidus temperatures, density, surface tension, the solubility and dissolution rate of aluminium oxide, carbon oxide, and various compounds, as well as the following operating parameters: temperature, current density, anode-cathode distance, etc. The electrolyte affects the current output and cell voltage, which determine the specific energy consumption and the overall cost of aluminium [13–20].

Fluoride salts – cryolite and aluminium fluoride – constitute essential electrolyte components. Since natural cryolite is rare, it is produced synthetically from fluorite to meet the needs of the aluminium industry [21–25].

SUBJECT MATTER OF THE STUDY

In Tajikistan, hydrofluoric acid is currently produced from local minerals (fluorspar) by TALCO Chemical, which is used to obtain cryolite employing imported aluminium and sodium hydroxides [26].

At this plant, along with concentrated hydrofluoric acid, acid at 30% concentration is produced during the sulphatisation of fluorite. Thus, the hydrochemical production of cryolite using hydrofluoric acid at 30% and sodium chloride was studied.

Under laboratory conditions, parameters for processing hydrofluoric acid at 28–30% concentration using aluminium hydroxide to produce fluoroaluminic acid were determined as per the following equation:

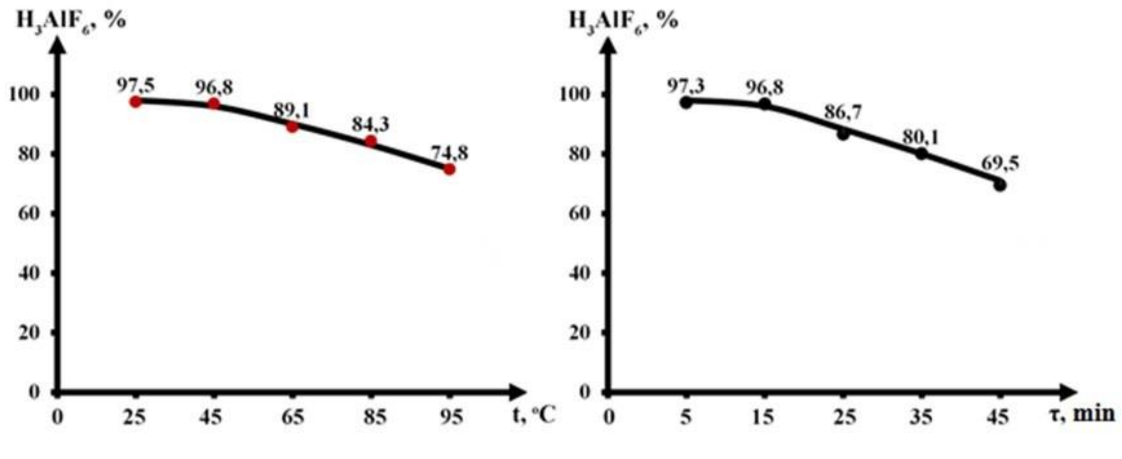
$$6HF + Al(OH)_3 = H_3AlF_6 + 3H_2O. \qquad (1)$$

The resulting fluoroaluminic acid was treated with sodium chloride to produce cryolite according to the reaction equation:

$$H_3AlF_6 + 3NaCl = Na_3AlF_6 + 3HCl. \qquad (2)$$

The main parameters in the production of fluoroaluminic acid include temperature and process duration.

The temperature dependence of the fluoroaluminic acid yield was investigated within a temperature range of 25°C to 95°C at a constant process duration of 10 min. Within this temperature range, a yield reduction from 97.5% to 74.8% was observed (fig. 1 *a*).



a *b*

Fig. 1. Dependence of the fluoroaluminic acid yield on temperature (a) and process duration (b)
Рис. 1. Зависимость выхода фторалюминиевой кислоты от температуры (a) и продолжительности процесса (b)

It can be seen in fig. 1 *a* that the yield of fluoroaluminic acid decreases at higher process

[8] Zelberg B. I., Ragozin L. V., Barantsev A. G., Yasevich O. I., Grigoriev V. G., Baranov A. N. Metallurgist's Handbook. Production of aluminum and alloys based on it. St. Petersburg: MANEB; 2013, 143 p. / Зельберг Б. И., Рагозин Л. В., Баранцев А. Г., Ясевич О. И., Григорьев В. Г., Баранов А. Н. Справочник металлурга. Производство алюминия и сплавов на его основе. СПб.: МАНЭБ, 2013. 143 с.

temperatures, which can be attributed to the partial evaporation of hydrofluoric acid. Hydrofluoric acid starts to evaporate from the solution at 50–60°C, with the process intensifying at temperatures above 95℃.

Fig. 1 *b* shows the dependence between the yield of fluoroaluminic acid and the process duration. With the process duration varying from 5 to 45 min, a decrease in the yield of fluoroaluminic acid from 97.3% to 69.5% is observed.

The exothermic reaction between aluminium hydroxide and hydrofluoric acid exhibits a reduction in the yield of fluoroaluminic acid as the temperature reaches 60°C or higher due to the evaporation of hydrofluoric and fluoroaluminic acids.

Thus, the rational parameters of fluoroaluminic acid production via the interaction of hydrofluoric acid and aluminium hydroxide are as follows: initial temperature of 25–40°C; duration of 10–15 min.

Experiments were carried out to produce cryolite through the interaction of a fluoroaluminic acid solution with a 25% sodium chloride solution in a stoichiometric ratio according to (2).

The interaction of fluoroaluminic acid with sodium chloride produces hydrochloric acid, whose concentration amounts to 10–12%. Of note is that hydrochloric acid comprises from 10% to 27% of produced cryolite in dissolved state.

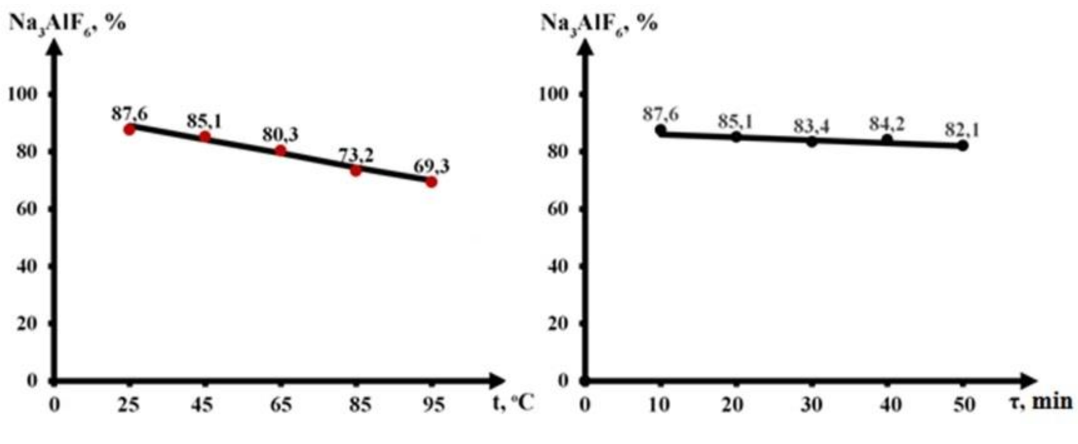
The temperature dependence of cryolite yield was also studied (fig. 2 *a*) at a constant process duration of 10 min. Here, with a temperature increase from 25°C to 95°C, a reduction in the cryolite yield from 87.6% to 69.3% is observed.

With rising temperature (see fig. 2 *a*), the yield of cryolite decreases due to its higher solubility in hydrochloric acid formed in (2).

The dependence of the cryolite yield on process duration was also examined. With the interaction time of reactants increasing from 10 to 50 min, a reduction in the cryolite yield from 87.6% to 82.1% is observed (see fig. 2 *b*). At a longer process duration, a slight decrease in the cryolite yield is clearly also attributable to the dissolution of cryolite in the hydrochloric acid.

Thus, the following rational parameters for hydrochemical cryolite production from hydrofluoric acid using aluminium hydroxide and sodium chloride are determined: temperature – 25°C; duration – 10–15 min; concentration of sodium chloride solution – 25%. Under these conditions, the yield of cryolite reaches 87.6%, while ~12% of cryolite remains dissolved in hydrochloric acid.

Of note is that the used hydrofluoric acid solution contains up to 1.5% of hexafluorosilicic acid that reacts with the aluminium hydroxide to produce silica gel and an aluminium fluoride solution. Following the separation of silica gel via filtration, the aluminium fluoride remains in the solution.



a *b*

Fig. 2. Dependence of the cryolite yield on temperature (a) and process duration (b)
Рис. 2. Зависимость выхода криолита от температуры (a) и продолжительности процесса (b)

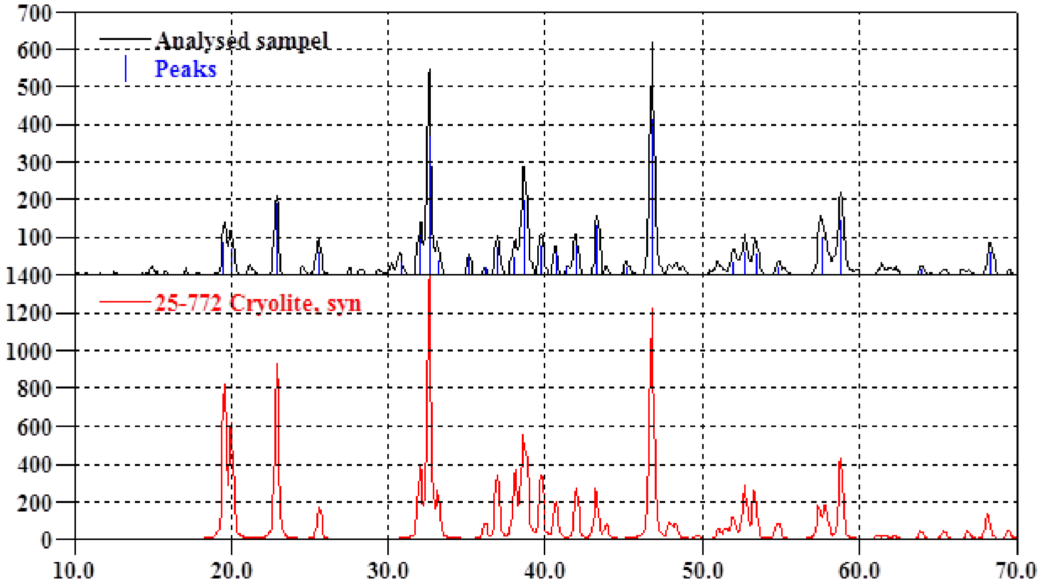


Fig. 3. XRD pattern of synthetic cryolite obtained using sodium chloride
(upper graph – analysed sample; lower reference graph – cryolite)
Рис. 3. Рентгенограмма синтетического криолита, полученного с использованием хлорида натрия
(верхний график – анализируемая проба, нижний эталонный график – криолит)

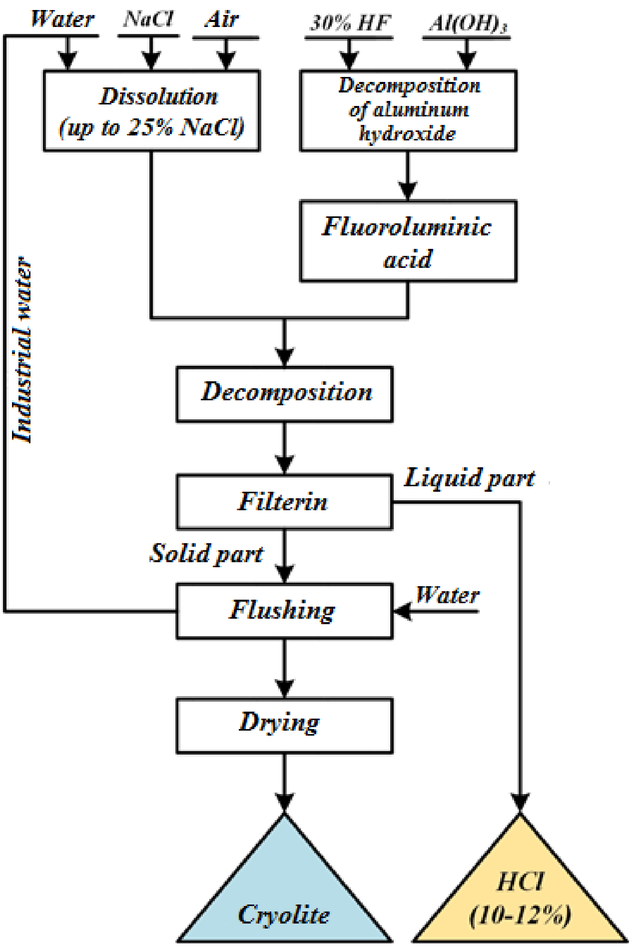


Fig. 4. Process flow diagram for the production of cryolite using sodium chloride
Рис. 4. Принципиальная технологическая схема получения криолита с использованием хлорида натрия

In order to validate the cryolite production process that employs sodium chloride, an X-ray diffraction (XRD) analysis was performed using an upgraded Dron-2 unit (fig. 3).

The obtained XRD pattern shows that the analysed sample (upper graph) is consistent with the reference sample (lower graph), which confirms the interaction between sodium chloride and fluoroaluminic acid resulting in the formation of cryolite.

Thus, the conducted studies served as a basis for developing a process flow diagram of hydrochemical cryolite production using hydrofluoric acid, aluminium hydroxide, and sodium chloride (fig. 4).

According to the provided process flow diagram, the first step involves the decomposition of aluminium hydroxide with hydrofluoric acid to obtain fluoroaluminic acid; the process occurs at 25–30°C for 10–15 min. Then, a 25% sodium chloride solution is prepared followed by the decomposition of fluoroaluminic acid at room temperature for 15–20 min. The resulting slurry is separated via filtration, while the solids are dried.

The liquid part comprising 10–12% HCl and 12% Na_3AlF_6 in dissolved form can be directly applied as an acidic composition to treat the bottom-hole zone of hot water reservoirs. Cryolite used in the electrolytic production of aluminium is obtained by evaporating the solution, while acid vapours condense or are absorbed by water to produce commercial hydrochloric acid.

CONCLUSION

The conducted studies revealed that the technology of cryolite production using sodium chloride is easy to implement and cost-effective due to the use of local mineral resources and low energy consumption.

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