# **SECTION 2**

# **REACTOR MATERIALS ON HEAT AND FAST NEUTRONS**

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## **RADIOACTIVE METALS IN RED MUD**

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The radioactivity of alumina production materials was analyzed. The main information on the content of radioactive metals in bauxite and red mud is presented. Technologies for the extraction of radioactive metals from red mud are considered.

#### **INTRODUCTION**

During the processing of bauxite raw materials into alumina using the Bayer method, aluminum and most of the gallium are transferred into an aluminate solution, while a part of bauxite that is insoluble during leaching forms a precipitate, the so-called red mud (RM). At the same time, from 0.7 to 1.5 t of RM is formed for each ton of obtained alumina, depending on the composition of the processed bauxite. Such rare metals as vanadium, titanium, zirconium, niobium, and tantalum partially transfer from bauxite to RM. Rare-earth metals (scandium, yttrium, as well as lanthanum, cerium and neodymium) are almost completely transferred to RM. Radioactive metals such as uranium and thorium are contained in the form of microimpurities in RM. The technologies for extracting these metals from RM are described in conference proceedings, monographs, in particular, in [1-5] and other sources.

The study of the processing of radioactive metals from secondary raw materials has long attracted the possibility of obtaining a cheap and quality final product. The study and analysis of recycling secondary radioactive metals was generalized by us earlier in [6, 7].

### RADIOACTIVITY OF ALUMINA PRODUCTION MATERIALS

RM radioactivity is hereditary, obtained from maternal bauxite processed by Bayer. It is known that bauxites of many ore regions of the world are characterized by increased radioactivity. Thus, according to [8, 9], the concentration coefficient of thorium in bauxite is 3.4-4.2. In [10], it is indicated that bauxites (Brazilian) are the main source of radioactivity in the Bayer process. Using gamma-ray spectroscopy and neutron activation analysis, it was found that the studied bauxites had an activity of (37±12) Bq/kg for <sup>238</sup>U and (154±16) Bq/kg for <sup>232</sup>Th. It was reported in [11] that the Egyptian bauxites used in Australia show an activity of ~ 120 Bq/kg for <sup>235</sup>U and from 289 to 575 Bq/kg for <sup>40</sup>K. The average radioactivity of bauxites in Saudi Arabia (Kassim region) is about 107 Bq/kg for <sup>232</sup>Th and 192 Bq/kg for  $^{40}$ K [12]. A number of bauxite deposits on the territory of the Siberian Platform are also characterized by increased concentrations of thorium: from 25...50 to 200 g/t and higher [13].

The resulting RM has a concentration of radionuclides higher than that of bauxite. Thus, in [14], in the production of alumina from Jamaican bauxites, an increase in the content of uranium, thorium, actinium, and their descendants (lead, bismuth) in RM was established. According to [15], the UAZ's RM (Russia) sample contained 64 g/t of thorium, the BAZ's RM (Russia) sample contained 89 g/t (when the thorium content in the initial bauxite was 22 g/t). Uranium in the samples was less than 0.01 g/t. NGZ's RM (Ukraine) contains about 50...60 g/t of thorium and 20...30 g/t of uranium [16]. At the same time, our chemical analysis of RM produced by ZALK (Ukraine) showed that the radioactive elements were either absent or in the form of traces [17].

In [18], RM samples using mineralogy methods were divided into fractions. Thorium and uranium tended to accumulate in light fractions (159 mg/g versus 102 mg/g in a single sample for thorium and 31.3 mg/g versus 25 mg/g for uranium). Neither uranium nor thorium was concentrated in magnetic fractions, and thorium tended to accumulate in a fine fraction (-20  $\mu$ m) (120 mg/g versus 99.9 mg/g in a single sample) and was mainly in perovskite. The authors concluded that physical methods for extracting radioactive elements or reducing their content in RM are not effective.

As it is known, RM can be used as a material for the road and building industries, including for the manufacture of ceramics. Many researchers indicate that this direction is the most real for the utilization of RM. However, the presence of radioactive elements limits the use of RM for these materials [19, 20]. To reduce the radiation activity of RM to an acceptable level, a number of solutions have been proposed [21–24]. So, for example, in [23] it is recommended to add barium carbonate to the ceramic mixture, which promotes the formation of glassy phases absorbing radiation.

Increased attention is also paid to ensuring the radiation safety of RM during its storage and use for soil reclamation [25, 26].

In work [27], the safety of people and the environment from the presence of RM residues was studied at an industrial facility. Evaluation and dose of irradiation by <sup>238</sup>U and <sup>232</sup>Th isotopes were made using the neutron activation method. Phantoms were used to monitor the distribution of the radiation dose throughout the human body, demonstrating the radiation effect on each individual organ.

It was emphasized in [28] that it is essential that the

alumina used for the manufacture of electronic devices contains a minimally low level of  $\alpha$ -radiation, the main source of which in alumina is uranium and in a lesser degree thorium. It was shown that during the Bayer process, part of the uranium is precipitated together with aluminum hydroxide, and its content is controlled by the content of organic substances in the solution.

## EXTRACTION OF METALS FROM RED MUD

Today, the issues of RM utilization with the extraction of rare, rare-earth (REM) and radioactive metals from them are priority. Most methods for extracting valuable metals from RM are hydrometallurgical, although pyrometallurgical (after sludge dehydration) is also used. It is noted [29] that REMs are easier to leach from RM than from bauxite due to differences in their mineral composition and morphology.

In [30], the selective separation of thorium from the solutions formed during the processing of RM from the Ural plants was carried out by coprecipitation with calcium or barium sulfates. Since the sorption activity of barium sulfate is 10-20 times higher than the corresponding value for calcium sulfate, a smaller excess of sulfuric acid is required for the extraction of thorium with BaSO<sub>4</sub>. When processing solutions containing 40...80 mg/dm<sup>3</sup> of thorium, to extract it into the solid phase by 90...99%, 5...10 g/dm<sup>3</sup> of BaCl<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> must be introduced into the initial solution. The amounts of BaCl<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> introduced should provide an excess of sulfate ions with respect to the sum of barium and calcium contents and the formation of negatively charged particles in the solution. Since calcium is always present in solutions under production conditions, mixed precipitates of BaSO<sub>4</sub> and CaSO<sub>4</sub> containing thorium are released into the solid phase.

Using a selective complexing agent, RM was deactivated in [31] with extraction of uranium-thorium products for burial. It is assumed that during decontamination of RM in amount of 1 million tons/year, more than 2.5 t of uranium and 1.5 t of thorium will be able to be extracted with the associated production of up to 30 t/year of scandium compounds.

The authors of work [32] developed a complex technology of sulfuric acid processing of RM with the extraction of uranium, rare-earth metals and aluminum. For the extraction of radioactive and rare-earth metals from RM of Middle Timan (Komi Republic, Russia), sulfuric acid leaching was used at a temperature of 90-120 °C and a ratio S:L = 3:10. The extraction of rareearth metals, thorium, and scandium into the sulfate solution was 92...99%, and that of iron, aluminum, and phosphorus was 98%. Titanium, niobium, hafnium and strontium remain in the solid residue. Sorption separation of uranium-thorium products, as well as the associated production of scandium compounds and additional extraction of aluminum and rare-earth metals was carried out at pH = 1.5 on universal KU-2 cationite.

The most frequently extracted elements from RM are scandium and yttrium. In this case, scandium is the most valuable element among the rare-earth metals that are present in RM. According to the authors of [33], it accounts for more than 95% of the economic value of

rare-earth metals in RM.

If it is believed that bauxite contains an average of 0.001...0.01% Sc<sub>2</sub>O<sub>3</sub>, then its content in RM is already 0.01...0.02%. As was shown in [34], during the leaching of bauxite, rare-earth metals are concentrated in RM, and scandium passes from bauxite to RM by 95%.

The most promising method for scandium extraction, according to [16], is the sulfuric acid opening of RM with its subsequent sorption on ion-exchange resins AFI-21, AFI-22, SF-4, SF-5 and desorption by a Na<sub>2</sub>CO<sub>3</sub> solution. The authors managed to extract most of uranium and thorium from the sulfuric acid solution of RM together with scandium, the extraction of which amounted to 50%). Complete precipitation of scandium was achieved by adding excess (up to 20...30 g/l) alkali to the solution. The crude scandium concentrate after titanium extraction contained 5...7% Sc, 4.5% U, and 0.9% Th.

When sulfuric acid leaching of RM, yttrium and other rare-earth metals are extracted together with scandium. To extract yttrium, it is proposed to treat RM by a 5...7% HCl solution at 85...95 °C and the ratio L:S = (3...5):1. The proposed technology allows, along with the separation of the uranium-thorium product and scandium salts, to obtain a rare-earth concentrate containing 15...25% La<sub>2</sub>O<sub>3</sub> [16].

According to the technological scheme of sorption leaching uranium and scandium from RM formed during the processing of Guinean and Jamaican bauxites by the Bayer method, titanium cake (30...40% of titanium), scandium oxide of the grade OS-99, and ironaluminum coagulant are obtained. At that, a solution containing radionuclides of uranium and thorium is sent for utilization.

When scandium is extracted/sorbed from acidic solutions on phosphorus-containing extractants/ion exchangers [35], the extraction of scandium from the eluate to the concentrate exceeds 97%. The impurities of thorium salts and other elements contained in the precipitate are separated by filtration. When extracting scandium, other rare-earth metals, thorium, zirconium and titanium, the extract is washed with a 4.5 mol/dm<sup>3</sup> solution of H<sub>2</sub>SO<sub>4</sub>. Reextraction was carried out with an alkali solution of 0.25 mol/dm<sup>3</sup>.

Selective extraction of thorium from scandium concentrates is complicated by the similarity of the physicochemical properties of thorium and scandium ions. To solve this problem, the authors of [36] use the differences in the sorption of hydrolyzed thorium ions and scandium ions on a mixture of KU-23 sulfocationite with anion exchange resin from solutions with  $pH \sim 3$  at a temperature of 60...90 °C. As a result, up to 95...98% Th passes into cation exchanger. Impurities of scandium ions from cation exchanger are removed by washing with 0.5...2.0 n solutions of salts of NaCl, KCl, etc. Thorium desorption is carried out by NaCO<sub>3</sub> solutions. A similar effect was also achieved on KU-23 with the introduction of 50...150 g/dm<sup>3</sup> MgCl<sub>2</sub> into the initial solution. The resulting products contained  $1...10 \text{ g/dm}^3$ of scandium and 1...2 g/dm<sup>3</sup> of thorium.

A significant disadvantage of extraction methods for extracting scandium from solutions together with radioactive elements is the need to introduce additional processing steps for purifying scandium concentrates from them.

In the works [37, 38], a carbonization technology was proposed for extracting scandium from RM pulp, which makes it possible to reduce the toxicity of sludge fields. Subsequent two-stage hydrolysis using a ZnO support (co-precipitator) makes it possible to obtain an intermediate product with a content of up to 5%  $Sc_2O_3$ .

During the carbonization of RM, a number of elements pass into the solution, including uranium, thorium, and others tended to the formation of carbonate compounds. Sodium gallate, titanium, zirconium, uranium, and thorium hydroxides introduced with RM pulp interact with bicarbonate and form a double basic carbonate [39] Na<sub>2</sub>O(Al, Ga)<sub>2</sub>O<sub>3</sub>·2CO<sub>2</sub>·nH<sub>2</sub>O, as well as soluble complexes  $[UO_2(CO_3)_3]^{4+}$  and  $[UO_2(CO_3)_2$ ·  $2H_2O]^{2-}$ . The content of elements in the productive solution after the first cycle of carbonation of RM by flue gases (duration of aeration is 24 h) is following, mg/l: 25.0 Ti; 5.4 Sc; 0.17 Th; 0.34 U [40].

On a pilot scale, as a result of multiple carbonatehydrocarbonate treatment of RM (both carbon dioxide and reactive NaHCO<sub>3</sub>), scandium, titanium, zirconium, uranium, thorium, as well as a fine suspension of aluminum, iron, silicon, calcium, and other elements hydroxides pass into the solution [41]. Scandium dissolved in the clarified solution was hydrolyzed in the second stage at 100 °C for 2 h. During hydrolysis and precipitation of scandium, sodium zincate which was formed upon introduction of ZnO into the alkaline solution, was used as a precipitant. The precipitate from the second stage is the primary scandium concentrate, which contains, %: 2.26 Sc; 0.007 U; 0.014 Th.

To select scandium, accopmanying products (aluminum-iron coagulants, enriched iron concentrate), other rare-earth metals and radioactive elements from RM, such methods are also used: pulp density separation with subsequent magnetic separation; leaching of 30%  $H_2SO_4$ , followed by extraction using D2EHPA and reextraction by NaF solution to obtain the Na<sub>3</sub>ScF<sub>6</sub> salt; biosorption [42] (with recovery up to 60% of scandium and 70% of yttrium); ion exchange; extraction by solid extractants. Also the introduction of the metal-collector into the solution remaining after leaching, extraction and concentration of scandium is used, for which a solution of aluminum or zinc oxide in sodium hydroxide is used. The formed precipitate is washed, dried and calcined.

In [43], ionic liquid was used to leach RM. It was shown that this method can be used to extract up to 70-85% rare-earth metals in solution with low ( $\sim$  3%) leaching of iron and 30% additional extraction of aluminum. At that, the ionic liquid can be regenerated and returned to RM leaching.

A detailed review of pyrometallurgical technologies for processing RM is presented in [44]. The data obtained during reduction smelting (temperature 1300...1400 °C, graphite as reducing agent) of RM of the Ural aluminum factories [15] are of interest. As a result of the melting, rare-earth metals (Ce, La, Sc, Sm, Yb, Eu, Lu) as well as thorium pass into slag. After extraction of  $Al_2O_3$  from slag, a 5...15-fold degree of enrichment in rare-earth metals and thorium is achieved; the concentration of rare-earth metals in the final product rises to 6.0...6.5 kg/t.

According to the data of [45], scandium fluoride successfully (by 92.7%) precipitates when an excess (~130%) of cryolite Na<sub>3</sub>AlF<sub>6</sub> or AlF<sub>3</sub> is introduced into the solution. With a 300% excess of precipitant, 99.6% scandium precipitation is achieved. Further concentration is based on the sharply distinctive solubility of scandium fluorides, other rare-earth metals and thorium in a solution of ammonium fluoride. With appropriate treatment, scandium passes into the solution in the form of the H<sub>3</sub>[ScF<sub>6</sub>] complex, and fluorides of other rareearth metals, uranium, and thorium remain in the residue. For poor solutions, sodium and potassium fluorides, HF, and H<sub>2</sub>SiF<sub>6</sub>, oxalic acid, sodium pyrophosphate are recommended as precipitants.

#### SUMMARY

As a result of the analysis it is established:

1. RM is characterized by increased radioactivity inherited from bauxite and due to the presence of mainly thorium and to a lesser extent uranium. Since the radioactive elements in the Bayer process almost completely transfer to RM, their concentration in RM is higher than in the initial bauxite.

2. Huge accumulations of RM around the world require the creation and implementation of industrial technology for their processing. Increased attention when storing and processing RM should be given to ensure radiation safety.

3. Physical methods for extracting radioactive metals from RM are not effective.

4. The extraction of metals from RM in most cases is based on the use of hydrometallurgical and to a lesser extent pyrometallurgical processes.

5. Promising are the complex technologies for processing RM, which make it possible to extract rare, rare earth (mainly scandium) and radioactive metals in one technological process, and use the resulting residue as building material.

6. It should be noted that the technologies described in the work have not yet found a proper economic justification. Most of them are characterized by factors that impede their implementation, including not only economic, but also environmental ones.

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## РАДИОАКТИВНЫЕ МЕТАЛЛЫ В КРАСНЫХ ШЛАМАХ

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Проанализирована радиоактивность материалов глиноземного производства. Приведены основные сведения о содержании радиоактивных металлов в бокситах и красном шламе (КШ). Рассмотрены технологии извлечения радиоактивных металлов из КШ.

### РАДІОАКТИВНІ МЕТАЛИ В ЧЕРВОНИХ ШЛАМАХ

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Проаналізовано радіоактивність матеріалів глиноземного виробництва. Приведено основні відомості про вміст радіоактивних металів у бокситах та червоному шламі (ЧШ). Розглянуто технології радіоактивних металів з ЧШ.