

ALKALINE AUTOCLAVE TECHNOLOGY FOR MONAZITE CONCENTRATE DECOMPOSITION

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Rare earth elements (REE) are essential raw materials for advanced high-tech industries such as: nuclear, automotive, electronic, aviation, space, metallurgical, oil refining, and are also used in key defense technologies - in the production of lasers, aircraft, missiles, communications, etc.

Currently, Russia is 80% dependent on imports of rare earth elements, while without REE it is impossible to implement 14 of the 27 critical technologies approved by the Decree of the President of the Russian Federation.

One of the main sources for the production of rare earth elements is monazite concentrate.

Currently, in the world, the processing of monazite concentrate is carried out by sulfuric acid and nitric acid methods. Each monazite processing method used has its advantages and disadvantages. The sulfuric acid method of processing monazite does not require fine grinding, while its complexity and multistage isolation of valuable components in the original version necessitated the development of alkaline technology for monazite decomposition.

It should also be noted that over the past 3-4 years, in the world practice of processing monazite, there is a tendency to use a simplified scheme on an industrial scale, when only the ultimate rare earth concentrate is extracted, and the other elements (uranium, thorium, phosphorus) are sent to "tailings," which will be processed when necessary. This greatly simplifies the process scheme, however, this simplification causes a decrease in the through extraction of REE into the total product to 65-70%.

This circumstance led us to develop a simplified version of the alkaline scheme, when the product of monazite processing is deactivated ultimate rare earth concentrate and uranium, and the radioactive solid product (Th and Ra) is sent to the tailings storage for long-term storage.

Krasnoufimsk storage places contain 83 thousand tons of monazite concentrate (MC), which presents more than 40 thousand tons of REE in terms of total oxide (REO). At world prices for rare earths on 01.05.2016, more than 800 million dollars has been accumulated there.

In addition, the MC of Krasnoufimsk contains 7980 Ci of radioactivity, that is unacceptable from an environmental point of view. Therefore, the creation of an economical and environmentally favorable technological scheme for its processing

with the simultaneous immobilization of the radioactive component is an urgent task.

The purpose of the dissertation is to develop a complex technology for processing monazite concentrate by the method of alkaline autoclave decomposition to obtain a deactivated concentrate of the sum of REE and chemical concentrates of thorium and uranium.

The following objectives had to be solved to achieve this goal:

- determine mineral and material composition of a sample of Krasnoufimsk monazite concentrate (MC);
- determine optimal conditions of autoclave decomposition of MC sample with provision of REE extraction degree not less than 95%, Th - not less than 98%, U - not less than 90%, phosphorus - not less than 96%;
- set optimal process modes of pulp separation and sediment washing during MC decomposition;
- determine conditions for conversion of REE and other components into nitric acid solution during processing of hydroxide cakes;
- conduct researches on purification of radium and uranium nitrate solutions;
- develop a process flow chart for the production of deactivated concentrate of the sum of REE and chemical concentrates of thorium and uranium;
- develop a process scheme for immobilization of the radioactive waste.

Scientific novelty

As a result of the studies carried out on autoclave decomposition with the use of caustic soda, the following new technical solutions are proposed:

1. Optimal conditions of autoclave alkaline decomposition of monazite concentrate were determined: grinding size - 44 μm ; alkali (sodium hydroxide) consumption - 2 kg/kg MC; contact time - 3 hours; process temperature - 170 $^{\circ}\text{C}$, pressure - 2-3 atm.

2. Conditions for dissolution of hydroxide cakes in nitric acid are determined to obtain a solution containing 150-200 g/l of the sum of REE and a minimum residual concentration of HNO_3 (0.3-0.5 mol/l).

3. The possibility of deep purification of nitrate solutions from radium radionuclides in the "head" of the technological process by co-precipitation with the precipitate of barium sulfate is determined.

4. For the first time in the process of MC processing, the possibility of separating solid and liquid phases using a filter-press with squeezing membranes and a centrifuge is shown.

5. A process flow chart for processing monazite concentrate has been developed to obtain deactivated concentrate of sum of REE, thorium oxide and chemical concentrate of uranium.

Practical significance and implementation of the results of the work

1. The developed method of alkaline autoclave decomposition of monazite concentrate using standard autoclave equipment makes it possible to simplify processing scheme due to reduction of quantity of technological equipment and increase productivity due to reduction of processing time.

2. Proposed method of separation of solid and liquid phases after alkaline treatment of monazite using filter-press with pressing membranes makes it possible to achieve higher degree of sediment washing and to reduce its final humidity.

3. The use of centrifugation during filtration of solutions after nitric acid leaching makes it possible to significantly reduce the time for obtaining the target products, reduce the moisture content of the precipitate and eliminate the additional operation of clarifying the solution.

4. The extraction of radium radionuclides at the initial stages of the process significantly (~ 5 times) reduces the amount of radioactive waste generated.

5. The proposed technological scheme of monazite concentrate processing using alkaline autoclave method of decomposition allows organizing complex processing of raw materials with extraction of all valuable elements: REE, thorium and uranium. The developed technology can be recommended as the basis for obtaining REE and thorium from the Krasnoufimsky monazite concentrate.

Provisions to be defended:

- modes of autoclave alkaline decomposition of monazite concentrate sample;
- conditions of the pulp separation and washing of sediments obtained after autoclave leaching of monazite and treatment of hydrate cakes with nitric acid;
- results of the studies on Ra extraction in the "head" of the process flow sheet;
- process flow sheet of complex processing of monazite concentrate to obtain deactivated concentrate of the sum of REE, thorium oxide and chemical concentrate of uranium.