

ANNOTATION

dissertation on the topic:

"DEVELOPMENT OF TECHNOLOGY FOR OBTAINING A CONCENTRATE OF RARE AND RARE EARTH METALS FROM URANIUM-CONTAINING RAW MATERIALS"

submitted for the degree of Doctor of Philosophy (PhD)

specialty 6D070900 – "Metallurgy"

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The purpose of the dissertation is to substantiate and develop an effective technology for extracting rare and rare earth metals from the black shale ore of the Balasauskandyk deposit, to study the scientific foundations of their leaching and sorption, as well as to improve the technology for obtaining a concentrate of rare earth elements from man-made mineral formations from the processing of phosphate uranium ores of the Chalk deposit with the release of phosphorus compounds into a separate product.

Objectives of the research:

1. analysis of existing scientific and technical literature and patent information and selection of the direction of research to determine the method of processing raw materials;
2. physical and chemical studies of ore of the Balasauskandyk deposit and technogenic mineral formations from the processing of uranium ores of the Melovoye deposit;
3. thermodynamic analysis of the interaction of rock-forming and useful minerals of the initial ore of the Balasauskandyk deposit with ammonium hydrosulfate during sintering and the study of kinetic dependencies of leaching of rare and rare earth elements from the sinter;
4. study of the composition of productive solutions depending on the pH and optimization of the process of their purification from impurities by precipitation of aluminammonium alum;
5. studies of kinetic dependencies of hydrolytic iron deposition with optimization of this process;
6. study of the influence of the nature of rare earth elements on the sorption process and development of technological parameters of sorption-desorption of rare and rare-earth metals from productive solutions from leaching of ore of the Balasauskandyk deposit;
7. development of conditions and experimental evaluation of acid and alkaline methods of leaching of technogenic mineral formations from the processing of phosphate uranium ores;
8. study of the patterns of phosphorus leaching in the form of sodium orthophosphate and determination of optimal conditions for its release into a separate product;
9. substantiation and development of a method for deposition of rare earth elements in the form of a concentrate from productive solutions from the opening of

technogenic mineral formations;

- enlarged laboratory tests in the conditions of production of technology for the extraction of rare and rare earth elements from black shale ores of the Balasauskandyk deposit.

Research methods:

When performing dissertations work, the following methods of analysis were used: chemical analysis of samples was performed on an optical emission spectrometer with inductively coupled plasma Optima 2000 DV (USA, PerkinElmer), as well as on an atomic emission spectrometer (VARIANAA 240SSAO), X-ray fluorescence analysis was carried out on a spectrometer with wave dispersion Venus 200 PANalytical B.V. (PANalytical B.V., Holland), the results of X-ray phase analysis were obtained on the BRUKERD8 ADVANCE apparatus on copper radiation at an accelerating voltage of 36 kV, a current of 25 mA, IR - spectroscopic analysis was performed on the FTIR spectrometer "Thermo Nicolet" Avatar 370 FTIR Spectrometer, in the region of 400-3600 cm^{-1} , thermal analysis - on the synchronous thermal analysis device STA 449 F3 Jupiter, the mapping data of the elemental and phase composition of the samples were obtained on the electron-probe microanalyzer JXA-8230 of JEOL (Japan), electronically micro-optical studies were performed on the energy-dispersion spectrometer Inca Energy Energy Aboutxfor Instruments. Thermodynamic analysis of processes performed on HSC Chemistry version 5.1;

Description of the main results of the study:

1. The complex of physical and chemical studies has established that rare and rare earth elements in the ore under study are in various minerals in the form of inclusions in a siliceous-carbon matrix. This is one of the reasons for their incomplete leaching when testing various options for ore processing technology.

2. The study of the kinetics of the leaching process of P and RARE EARTH from the sinter of black shale ores showed that the process is limited by diffuse restrictions ($E_A = 4.57 \text{ kJ / mol}$);

3. The constants of the thermohydrolysis process and the activation energy value of 41.5 kJ/mol indicates the possibility of iron deposition in kinetic mode.

4. On the basis of IR spectroscopic study of saturated REE from a model solution of ionite KU-2-8n, it is assumed that in the matrix of the initial resin there is an interaction of the REE cation with ionite sulfo groups to form a complex whose size affects the degree of sorption of reE, while the size of the complex is in direct proportion to the atomic radius of the complexifier Based on a comparison of the result- It has been established that the influence of their nature on the magnitude of the degree of sorption can be judged by the magnitude of the intensity of the IR spectra of individual REEs at a wave number of 1220 cm^{-1} ;

Main provisions for protection:

1. the results of physical and chemical studies of the ore of the Balasauskandyk deposit and technogenic mineral formations from the processing of uranium ores of the Melovoye deposit;

2. the results of thermodynamic analysis of the interaction of the host rock of

the initial ore of the Balasauskandyk deposit and minerals containing valuable components with ammonium hydrosulfate;

3. the results of studies of the process of opening the ore of the Balasauskandyk deposit by sintering it with ammonium sulfate in the presence of concentrated sulfuric acid;

4. the results of studies of kinetic dependencies of leaching of rare and rare earth elements with a dilute solution of sulfuric acid;

5. the results of purification of productive solutions from impurities by precipitation of aluminammonium alum;

6. the results of studies of kinetic dependencies of the process of hydrolytic deposition of iron from productive solutions;

7. results of sorption extraction of rare and rare earth elements from productive solutions from ore leaching;

8. the results of the study of the processes of alkaline and acidic, as well as two-stage - alkaline-acid opening of technogenic mineral formations from the processing of phosphate uranium ores with the release of phosphorus compounds into a separate product.

9. the results of enlarged laboratory tests in the conditions of production of the technology of processing black shale ore of the Balasauskandyk deposit with the extraction of rare and rare earth elements.

Substantiation of the novelty and importance of the results obtained: The studies carried out during the implementation of the dissertation work made it possible to study in detail the material and phase composition, on the basis of which to simulate the locations of P and rare earth metals in the ore, to theoretically substantiate and experimentally confirm the possibility of processing black shale ores of the Balasauskandyk deposit by sintering, leaching and sorbent concentration of P and rare earth metals. From the existing modern methods of processing black shale ores is the opening of black shale ore by sintering with ammonium sulfate in the presence of concentrated sulfuric acid and subsequent leaching of the sinter. This approach will increase the degree of extraction of rare metals, as well as simultaneously extract REE in the form of a concentrate, which will help to increase the complexity of the use of mineral raw materials and expand the range of products.

The scientific novelty of technical solutions is confirmed by the issuance of a patent of the Republic of Kazakhstan No. 33153 Method of extracting rare earth elements from siliceous raw materials, publ. No bul.38. 15.10.2018. and patent of the Republic of Kazakhstan No. 34620 Method of processing carbon-silica ores, publ. No bul. 46 – 20.11.2020.

Compliance of the direction of development of science or state programs. In this paper, a new method of opening black shale ore by sintering with ammonium hydrosulfate and subsequent leaching of the sinter is proposed. This approach will increase the degree of extraction of rare metals, as well as simultaneously extract REE in the form of a concentrate, which will contribute to increasing the complexity of the use of mineral raw materials and expand the range of products.

An improved method for extracting REE, in the form of a concentrate, from technogenic mineral formations from the processing of phosphate uranium ores has

been developed, which makes it possible to isolate phosphorus into a separate semi-product, contributing to increasing the complexity of the use of raw materials and the profitability of production.

The research work corresponds to the priority direction of the development of science "Rational use of natural, including water resources, geology, processing, new materials and technologies, safe products and structures".

Doctoral student's contribution to the preparation of each publication

1 Sultan Yulusov, Tatyana Y. Surkova, Vladlen A. Kozlov, Madina Barmenshinova Application of hydrolytic precipitation for separation of rareearth and impurity elements, Journal of Chemical Technology and Metallurgy, 53, 1, 2018, C 27-30: preparation and direct participation in research, analysis and generalization of the obtained results, writing the text of the article;

2 Sultan Yulusov, Tatiana Y. Surkova, Leila U. Amanzholova, Madina B. Barmenshinova, On sorption of the rare-earth elements, Journal of Chemical Technology and Metallurgy, 53, 1, 2018, C 79-82. scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the article;

3 Yulusov S.B., Kozlov V.A., Yegorov N.B., Surkova T.YU., Baygenzhenov O.S. Fiziko-khimicheskiye issledovaniya uransoderzhashchego syr'ya mestorozhdeniya Balasauskandyk // Vestnik KazNITU, 2019, № 1(130), S. 377-383: preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the article;

4 B.K.Kenzhaliev, T.Yu. Surkova, S.B. Yulusov, E.A. Pirmatov, A.P. Dulenin, Obtaining a concentrate of rare earth elements from waste and middlings of the uranium industry, KIMS No. 1 2017, Almaty, P. 70-77: scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the article;

5 Baygenzhenov O.S., Yulusov O.B., Khabiyev A.T., Sydykanov M.M., Akbarov M.S. (Almaty, Kazakhstan). Issledovaniye protsessa vyshchelachivaniya redkikh i redkozemel'nykh metallov iz chernoslantsevykh rud Bol'shogo Karatau // KIMS, 2019, №3, S. 76-80: scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the article;

6 Yulusov S.B., Kozlov V.A., Egorov N.B., Surkova T.Yu., Itygina D.R.. Thermodynamic assessment of the interaction of the main components of polymetallic carbon-siliceous ores with ammonium hydrosulfate // XIII International Scientific and Practical Conference International Trends in Science and Technology. -Warsaw, 2019. P. 20-22: scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the report;

7 Kengaliev B.K., Surkova T.Yu., Yulusov S.B., Pirmatov E.A., Dulenin A.P.. Rare-Earth concentrate production from technogen mineral formations // ASRTU Conference proceedings international conference with Elements of School for Young Scientists on Recycling and utilization of tehnogenic Formations. - Ekaterinburg, 2017. P. 152-156: preparation and direct participation in conducting

research, analyzing and summarizing the results obtained, writing the text of the report;

8 Yulusov S. B., Surkova T. Yu., Kozlov V. A. Behavior of rare earth and impurity elements in the process of their hydrolytic precipitation from solutions of processing of uranium-containing raw materials // International Conference: Satpaev readings "Scientific heritage of Shakhmardan Yessenov" - Almaty 2017. From 424-427: scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the report;

9 Kozlov V.A., Baigenzhinov O.S., Yulusov S.B., Zhumakynbai N., Dagubaeva A.T. Prospects for the production of rare and rare earth metals from carbon-silica polymetallic ores of the Greater Karatau // International scientific and practical conference "Effective technologies for the arbitrary use of non-ferrous, rare and noble metals." - Almaty, 2018. From 273-275: scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the report;

10 Yulusov S.B. Redkozemel'nyye elementy v chernoslantsevyykh rudakh, XVIII Mezhdunarodnaya nauchno-prakticheskoy konferentsii studentov i molodykh uchenykh imeni professora L.P. Kulova «Khimiya i khimicheskaya tekhnologiya v KHKHI veke». - Tomsk, 2017. P 397-398: scientific and theoretical justification, preparation and direct participation in research, analysis and generalization of the results obtained, writing the text of the report;

11 Patent RK №33153 Yulusov S.B.; Dulenin A. P.; Surkova T. YU.; Kenzhaliyev B. K.; Pirmatov E. A., Sposob izvlecheniya redkozemel'nykh elementov iz kremnistogo syr'ya, Opubl. № byul.38. 15.10.2018.: scientific and theoretical justification and direct participation in the writing of the application;

12 Patent of the Republic of Kazakhstan No. 34620 Kozlov V.A., Yulusov S.B., Baigenzhenov O.S., Omar G., Zhumakynbay N. Method for processing carbon-silica ores, Publ. No. bul. 46 - 20.11.2020: scientific and theoretical justification and direct participation in the writing of the application;