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# ОБ ИЗВЛЕЧЕНИИ ЦЕННЫХ КОМПОНЕНТОВ ИЗ ОТХОДОВ ПЕРЕРАБОТКИ ВОЛЬФРАМОВЫХ РУД ВОДНОЙ ЭКСТРАКЦИЕЙ

**С.С. Санжанова[1], Д.И. Жамбалова[1], А.М. Плюснин[1]**

[1] Геологический институт им. Н.Л. Добрецова Сибирского отделения РАН, Улан-Удэ, Россия, e-mail: sanzhanova7@rambler.ru

***Аннотация:*** Лежалые хвосты Джидинского вольфрамо-молибденового комбината содержат остаточную рудную минерализацию. В процессе минералого-петрологического анализа исследуемых хвостов выявлено содержание апатита, шеелита, пирита, магнетита, флюорита, кварца и тенорита. В экстракт в процессе выщелачивания водой из песков переходят сера, фтор, алюминий, железо, цветные металлы и редкоземельные элементы. Для концентрирования ценных компонентов из полученных экстрактов выбран известняк (карбонат кальция), поверхность зерен которого служит основой для их осаждения. При взаимодействии с водными растворами происходит гидролиз верхних слоев известняка и высвобождаются гидроксид-ионы, подщелачивающие кислые экстракты. Отмечено, что концентрация цинка, меди и кальция в водных растворах лабораторной установки возрастает, а редкоземельных элементов, наоборот, уменьшается. Степень извлечения редкоземельных элементов составил 0,8–0,82. Установлено, что за счет повышения pH воды происходит осаждение на поверхности известняка таких ценных компонентов, как цветные металлы и редкоземельные элементы. Проведенный химический анализ известняка показал, что на поверхности известняка также задерживаются кремний, алюминий, фтор и железо. Процесс извлечения компонентов из водных вытяжек отходов переработки вольфрамовых руд известняком сопровождается осаждением гипса, смитсонита и гидроксида железа.

***Ключевые слова:*** лежалые хвосты, Джидинское хвостохранилище, вольфрамовые руды, остаточные минералы, выщелачивание, экстракция, нейтрализация, известняк, цветные металлы и редкоземельные элементы.

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## Recovery of valuable components from aqueous extracts of tungsten ore processing waste

**S.S. Sanzhanova[1], D.I. Zhambalova[1], A.M. Plyusnin[1]**

[1] N.L. Dobretsov Geological Institute, Siberian Branch of the Russian Academy of Sciences, Ulan-Ude, Russia, e-mail: sanzhanova7@rambler.ru

***Abstract:*** Tailings sands of the Dzhida tungsten–molybdenum plant contain residual ore mineralization. Mineralogical and petrological analysis of the studied tailings revealed apatite, scheelite, pyrite, magnetite, fluorite, quartz and tenorite. In water leaching of sands, such chemical elements as sulfur, fluorine, aluminum, iron, non-ferrous metals and rare earth elements pass into the extract. Limestone (calcium carbonate) was selected to concentrate valuable components from the obtained extracts; its grain surface serves as a basis for their precipitation. When interacting with aqueous solutions, hydrolysis of the upper layers of limestone occurs and hydroxide ions are released, alkalizing acidic extracts. It is noticed that the concentration of zinc, copper and calcium in aqueous solutions of the laboratory setup increases, while rare earth elements, on the contrary, decrease. The degree of REE extraction is 0.8–0.82. It is found that due to the increase in water pH, such valuable components as non-ferrous metals and rare earth elements are deposited on the surface of limestone. The chemical analysis of limestone shows that silicon, aluminum, fluorine and iron are also retained on the limestone surface. The process of extracting components from aqueous extracts of tungsten ore processing waste by limestone is accompanied by the deposition of gypsum, smithsonite and iron hydroxide.

***Key words:*** old tailings, Dzhida tailings dump, tungsten ore, residual minerals, leaching, extraction, neutralization, limestone, non-ferrous metals and rare earth elements.

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## Introduction

Tailings of tungsten ore mining and processing contain a mixture of minerals [1], which decomposes during long-term storage, enriching pore water with various toxic and industrially valuable components [2]. In early works [3], it was shown that over the period from 1934 to 2013, the old tailings of the Dzhida tailing dump were subjected to leaching, especially sphalerite, galena and chalcopyrite. It is also known that oxidation of sulfides in waste leads to the formation of sulfuric acid [4] and the acidic environment also leads to leaching processes [5]. However, the content of copper, zinc, aluminum in mine waters of tungsten mining falls short of concentrations significant for extraction [6, 7]. Publications on the extraction of valuable components from mine waters mainly concern rare earth elements — yttrium, scandium and lanthanides [8–11], which are in high demand in high-tech industries. The following methods are proposed for the extraction and concentration of rare earth metals from aqueous media: extraction [12], electroflotation using surfactants and coagulants [13], sorption [14], biosorption [15], crystallization and precipitation [16]. For the treatment of large volumes of water, the use of extractants is impractical, since secondary waste appears, which requires additional disposal measures; electrochemical approach requires modern technical equipment; ion exchange and crystallization are labor-intensive and expensive. At the same time, adsorption and precipitation with chemical reagents are most effectively used to extract di- and trivalent metal ions from wastewater [17]. The precipitation method is based on the properties of chemical elements to precipitate under certain acid–base conditions of the environment. According to the reference book [18], trivalent rare earth metals precipitate at pH 6.8, their dilute solutions – at 8.5, $Ce(OH)_4$ in the pH range from 0.8 to 1.5, $Zn(OH)_2$ – 7.0–8.5, $Cd(OH)_2$ and $Co(OH)_2$ – 7.9–9.4. In the work [19], when using MgO as a precipitant, it was possible to extract Cu at pH ⩾ 6.5, Mn, Ni and Zn pH ⩾ 8.0. The objectives of this work were the concentration and complex extraction of non-ferrous and rare earth metals from aqueous extracts obtained from old tailings of a tungsten–molybdenum plant.

## Materials and methods

The work used the old tailings of the Dzhida tailings dump, collected in 2018 and 2019. Mineralogical and petrological analyses of two sand samples were carried out. To obtain aqueous extracts, sands *2* (Fig. 1) were washed with distilled water *1*. Textile and paper filters *3* were provided to retain suspended particles. Resulting extract *4* entered a unit with five sections *5* for metal precipitation, filled with crushed limestone of fraction 1–5 mm. After the interaction of the extracts with limestone, solution *6* was removed through pipe *5*. The rate of the extract passed through limestone was 100 ml/h. The samples of solutions for the analysis were collected using membrane filters with 0.22 μm micropores and acidified with nitric acid of chemically pure grade. Sand samples for electron microscopic examination were washed with distilled water, dried at room temperature and glued to a self-adhesive backing two by three cm. Limestone samples for the physicochemical analysis were taken from each section of the setup at the end of the experiment, dried and crushed manually in an agate mortar.

Physicochemical analytical works were carried out at shared use centers of the Dobretsov Geological Institute and the Limnological Institute SB RAS, and the Sobolev Institute of Geology and Mineralogy. The content of rare earth elements (REE) in water was determined by the ICP-MS method on an Agilent 7500 according to the technique [20], sulfate ions, silicic acid, and pH were analyzed according to generally accepted techniques (Conclusion No. 059-22) in the certified Laboratory of Hydrogeology and Geoecology of the Geological Institute: EXPERT-001 pH meter was used to measure pH of solutions (pH error ± 0.02); sulfates in solutions were determined by the turbidimetric method; silicic acid – by the photometric me-


*Fig. 1. Schematic diagram of obtaining aqueous extracts and recovery of non-ferrous metals and REE*

*Рис. 1. Принципиальная схема получения водных экстрактов и извлечения цветных металлов и РЗЭ* thod. The content of non-ferrous metals and solid samples were determined by X-ray diffraction analysis on ARL Perform'X 4200 crystal diffraction spectrometer and Element I high-resolution mass spectrometer, and electron microscopic analysis was performed on LEO 1430VP microscope.

**Results and discussion**

Early studies of the Dzhida plant showed that the sands consisted of quartz with admixtures of feldspar, apatite, albite, amphibole, epidote, fluorite, hubnerite, scheelite, beryl, magnetite, goethite, chlorite, pyrite, molybdenite, zircon, siderite and scheelite [3]. Two sand samples used in the work were found to contain apatite, scheelite, pyrite, magnetite, fluorite and quartz (Fig. 2, *a*). In the first sand sample, the predominant mineral was pyrite. REEs are found as inclusions in scheelite (Fig. 2, *b*). Tenorite was found in the second sample (Fig. 2, *c*).

The sands were poured one by one into the container shown in Fig. 1. By extracting two sand samples with distilled water, two samples of aqueous extract were obtained, respectively. The content of chemical elements in the obtained extracts A and B were as follows (mg/l): sulfur — 4700 and 280, sulfate ions — 4000 and 700, silicic acid — 9 and 4, calcium — 673 and 360, aluminum — 2200 and 161, zinc — 300 and 0.450, iron — 66 and 0.23, copper — 3.9 and 0.32, cobalt 9.5 and 0.008, nickel — 5.3 and 0.007 each, cadmium — 4.6 and 0.01. High concentrations of lanthanides were recorded at the beginning of the extraction process in the first liter (μg/l): Y — 2000, La — 990, Ce — 2100, Pr — 230, Nd — 860, Sm — 250, Eu — 95, Gd — 360, Tb — 68, Dy — 430, Ho — 90, Er — 260, Tm — 39, Yb — 50, Lu — 36.

The extract A pH at the beginning of the extraction process was 3.2, after 35 L it increased to 5.6; the extract B pH was 4.0, after 12 L — 6.5. The change in pH of the extracts depending on the duration of the process indicates that the pores of the sands contain a limited amount of acids, which are washed out of the sands by 9–35 L of water depending on the original concentration. When the extracts passed through the sections of the experimental setup with limestone for 8 hours, pH of the extract A increased by 1.6, and that of extract B by 1.1. The content of calcium, zinc, cadmium, nickel, cobalt and copper in the solutions of the setup sections increased compared to the original extracts. Figures 3, *a* and 3, *b* show the curves of the change in metal concentrations in the extracts A and B, where the points related to the initial concentrations are indicated

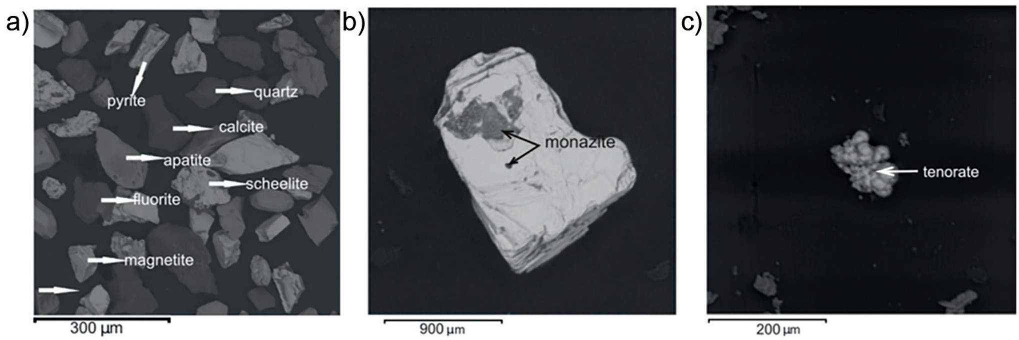
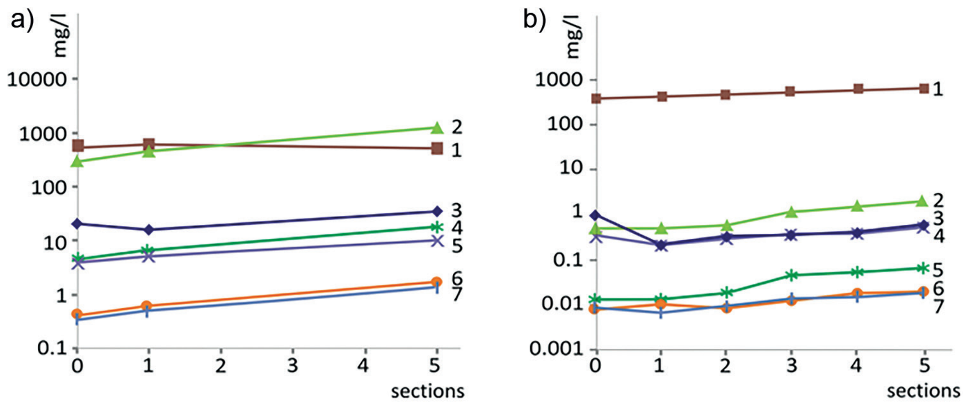



*Fig. 2. Electronic images: minerals contained in the tailings sands (a); scheelite with an admixture of monazites (b), tenorite (c)*

*Рис. 2. Электронные снимки: минералов, входящих в состав песков хвостохранилища (а); шеелита с примесью монацитов (б); тенорита (в)*




1 – calcium, 2 – zinc, 3 – iron, 4 – copper, 5 – cadmium, 6 – nickel, 7 – cobalt

1 – кальций, 2 – цинк, 3 – железо, 4 – медь, 5 – кадмий, 6 – никель, 7 – кобальт

*Fig. 3. Content of non-ferrous metals in obtained extracts A and B*

*Рис. 3. Содержание цветных металлов в полученных экстрактах A и B*

in the OY axis. The increase in calcium concentration in the solutions after contact with limestone (up to 3%) occurs due to the acid decomposition of limestone. The greatest increase in the sections was observed for the content of zinc, copper, cadmium, nickel, cobalt and cadmium (up to 8%) due to the formation of hydroxides. Unlike the listed metals, the content of REE in the solutions of the sections decreased (Fig. 4). But the content of REE decreased from the first section to the fifth, except for thulium and ytterbium.

The percentage of REE extraction by limestone reaches 82% in the first section and decreases to 80% by the fifth section. Accordingly, the chemical composition of limestone after the experiment shows that most REE are more concentrated in limestone of the first section (Table 1). The highest extraction was obtained for yttrium – ten times. Gypsum (Fig. 5, *b*) and smithsonite (Fig. 5, *c*) were recorded on the surface of limestone grains using an electron microscope. The numbers indicate the points of the areas of chemical

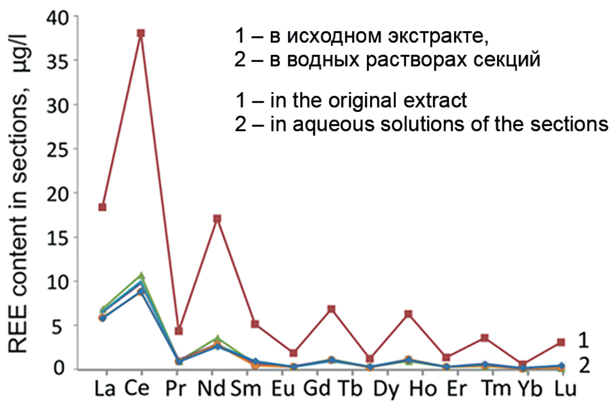



*Fig. 4. Content of rare earth elements in extract A*

*Рис. 4. Содержание редкоземельных элементов в экстракте A*

Table 1

***Microelement composition of limestone, g/t***
***Микроэлементный состав известняка, г/т***

| | Zn | Cu | Cd | Co | Ni | Y | Sc | La | Ce | Pr | Nd | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Initial limestone | 16 | 14 | < | < | < | 1.3 | < | 1.18 | 1.72 | 0.20 | 0.86 | 0.18 | 0.046 | 0.19 | 0.03 | 0.19 | 0.04 | 0.12 | 0,02 | 0.13 | 0.02 |
| 1st section | 143 | 48 | 10.5 | 3.4 | 3 | 13.8 | 1.5 | 4.2 | 7.9 | 0.86 | 4.2 | 1.3 | 0.43 | 1.38 | 0.25 | 1.56 | 0.32 | 0.94 | 0.15 | 1.0 | 0.15 |
| 2nd section | 105 | 41 | < | < | < | 7 | 1 | 2.7 | 4.7 | 0.5 | 2.2 | 0.59 | 0.19 | 0.74 | 0.13 | 0.81 | 0.17 | 0.49 | 0.08 | 0.5 | 0.07 |
| 3nd section | 104 | 36 | < | < | < | 5.7 | 0.8 | 1.62 | 2.6 | 0.27 | 1.1 | 0.29 | 0.1 | 0.39 | 0.07 | 0.44 | 0.093 | 0.27 | 0.04 | 0.27 | 0.04 |
| 4th section | 66 | 33 | < | < | < | 3.8 | 0.6 | 1.3 | 1.97 | 0.22 | 0.9 | 0.22 | 0.075 | 0.27 | 0.05 | 0.31 | 0.27 | 0.19 | 0.03 | 0.18 | 0.03 |
| 5th section | 91 | 34 | < | < | < | 4.5 | 0.5 | 1.58 | 2.6 | 0.27 | 1.13 | 0.28 | 0.08 | 0.3 | 0.05 | 0.35 | 0.04 | 0.24 | 0.03 | 0.23 | 0.03 |

Comment: < – below the detection limit of the XRF method; metals, yttrium and scandium were analyzed by the XRF method, rare earth.
Примечание: < – ниже предела обнаружения метода РФА; металлы, иттрий, скандий проанализированы методом РФА, редкоземельные элементы – МС.

Table 2

***Limestone surface composition (electron microscopy data), %***
***Состав поверхности известняка (по данным электронной микроскопии), %***

| Figure | Sampling locations | $SiO_2$ | $Al_2O_3$ | FeO | CaO | $Na_2O$ | CoO | NiO | CuO | ZnO | CdO | $SO_3$ | F |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A | 1 | 0 | 0 | 0 | 72.48 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| B | 1 | 7.83 | 33.69 | 2.29 | 7.49 | 0 | 0 | 0 | 1,41 | 3.62 | 0 | 12.61 | 0 |
| | 2 | 2.78 | 40.66 | 2.73 | 19.69 | 0 | 0 | 0 | 0 | 5.03 | 0 | 11.21 | 0 |
| C | 1 | 0 | 3.55 | 0 | 10.45 | 14.76 | 0 | 0.61 | 0 | 75.62 | 0 | 22.1 | 5.66 |
| | 2 | 0 | 5.54 | 0 | 14.41 | 15.5 | 0 | 0 | 0 | 75.36 | 0 | 21.03 | 5.73 |

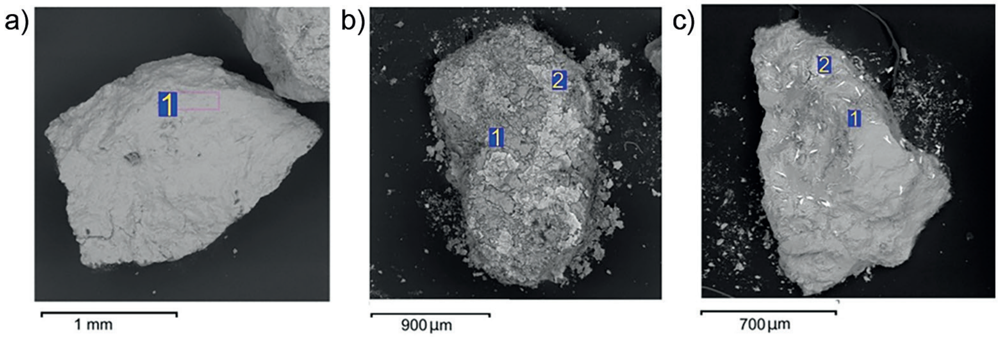



*Fig. 5. Electron microscopic photographs of limestone: original limestone (a); after extraction of non-ferrous metals and REE (b, c) (numbers indicate locations of material collection for chemical analysis by electron microscopy)*

*Рис. 5. Электронно-микроскопические фотографии известняка: исходного известняка (а); после извлечения цветных металлов и РЗЭ (б, в) (цифрами обозначены места отбора материала для химического анализа методом электронной микроскопии)*

analysis by electron microscopy and are given in Table 2. The presence of impurity elements Si, Al, F and Fe indicate the co-precipitation of aluminosilicates, fluorite and iron hydroxide.

## Conclusions

Sands of the Dzhida tailings contain apatite, scheelite, pyrite, magnetite, fluorite, quartz and tenorite.

Leaching the sands with 12–35 liters of distilled water allows obtaining extracts with a pH of 5–6 and almost completely displaces non-ferrous and rare earth metals from the pore space of the sands.

Two samples of extracts obtained by washing contained the following chemical elements (mg/l): sulfur — 4700 and 280, sulfate ions — 4000 and 700, silicic acid — 9 and 4, calcium — 673 and 360, aluminum — 2200 and 161, zinc — 300 and 0.450, iron — 66 and 0.23, copper — 3.9 and 0.32, cobalt — 9.5 and 0.008, nickel — 5.3 and 0.007, cadmium — 4.6 and 0.01; of the rare earth elements, cerium was found in the largest amount — 38 µg/l; yttrium — 40 µg/l.

Laboratory experiments have shown that the interaction of the acid extracts and limestone for 8 hours increases pH by 1.1–1.6 units. At the same time, the content of non-ferrous metals and calcium in the aqueous solutions increases. Based on the results of the work, the conclusion is drawn about the ability of limestone to concentrate zinc and REE. The maximum zinc content was found in the first section and amounted to 143 g/t. The total extraction of REE reaches 40 µg/g, which is 80–82% of the total REE content in the aqueous extract.

Thus, the first stage of the experimental extraction and concentration of valuable components from tungsten ore processing waste has been carried out. The further experiments will be aimed at the extraction of non-ferrous metals and REE from limestone.

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## ИНФОРМАЦИЯ ОБ АВТОРАХ

*Санжанова Сэсэг Сергеевна*[1] — канд. техн. наук,
научный сотрудник, e-mail: sanzhanova7@rambler.ru,
ORCID ID: 0000-0001-7584-0344,
*Жамбалова Дашима Ивановна*[1] — канд. геол.-минерал. наук,
научный сотрудник, e-mail: re.ly.re@yandex.ru,
ORCID ID: 0000-0002-0230-6973,
*Плюснин Алексей Максимович*[1] — д-р геол.-минерал. наук,
зав. лабораторией e-mail: otchernye@yandex.ru,
ORCID ID: 0000-0001-9380-7281,
[1] Геологический институт им. Н.Л. Добрецова Сибирского отделения РАН.
**Для контактов**: Санжанова С.С., e-mail: sanzhanova7@rambler.ru.

## INFORMATION ABOUT THE AUTHORS

*S.S. Sanzhanova*[1], Cand. Sci. (Eng.), Researcher,
e-mail: sanzhanova7@rambler.ru,
ORCID ID: 0000-0001-7584-0344,
*D.I. Zhambalova*[1], Cand. Sci. (Geol. Mineral.),
Researcher, e-mail: re.ly.re@yandex.ru,
ORCID ID: 0000-0002-0230-6973,
*A.M. Plyusnin*[1], Dr. Sci. (Geol. Mineral.),
Head of Laboratory, e-mail: otchernye@yandex.ru,
ORCID ID: 0000-0001-9380-7281,
[1] N.L. Dobretsov Geological Institute, Siberian Branch
of the Russian Academy of Sciences, 670047, Ulan-Ude, Russia.
**Corresponding author**: S.S. Sanzhanova, e-mail: sanzhanova7@rambler.ru.

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