



## Research on hydrometallurgical processing of titanium-magnesium production sludge with niobium extraction in solution

<sup>1</sup>Ultarakova A.A., <sup>1</sup>Karshyga Z.B., <sup>1</sup>Lokhova N.G., <sup>1\*</sup>Yessengazyev A.M., <sup>2</sup>Burns P.C.

<sup>1</sup>"Institute of Metallurgy and Ore Beneficiation" JSC, Satbayev University, Almaty, Kazakhstan

<sup>2</sup> University of Notre Dame, "Center for Sustainable Energy" USA, South Bend, USA

\*Corresponding author email: a.yessengazyev@satbayev.university

### ABSTRACT

The material composition of sludge deposits from titanium-magnesium production was studied by chemical, X-ray, and microprobe analysis methods. Studies of the phase composition of the collector sludge showed that niobium is mainly bound with aluminum and titanium in oxide compounds. The particles of these compounds are very small and surrounded by the clay and carbonate component of the sludge collector. The chemical and mechanical activation processes of the sludge from the titanium-magnesium production sludge collector were executed based on the data obtained on the material composition. Alkaline leaching of sludge from sludge collector after preliminary activation was executed. The optimum conditions for the niobium extraction from the sludge of the sludge dump from the titanium-magnesium production were determined: sodium hydroxide concentration of 200 g/dm<sup>3</sup>, glycerol 5 g/dm<sup>3</sup>, S:L = 1:10, temperature 95 °C, leaching process duration - 4 hours. The addition of glycerol during alkaline leaching of sludge inhibits the transition of hexaniobate into insoluble sodium metaniobate enabling to increase the niobium extraction by 1.3 times with transfer into the solution of up to 80% of the extracted metal.

**Keywords:** sludge from the sludge collector, niobium, activation, leaching, sodium hydroxide.

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	<b>Information about authors:</b>
<b>Ultarakova Almagul Amirovna</b>	Candidate of Technical Sciences, Leading Researcher, "Institute of Metallurgy and Ore Beneficiation" JSC, Satbayev University, Shevchenko str. 29/133, Almaty, Kazakhstan. E-mail: a.ultarakova@satbayev.university
<b>Karshyga Zaure Baitaskyzy</b>	Ph.D., Leading Researcher, "Institute of Metallurgy and Ore Beneficiation" JSC, Satbayev University, Shevchenko str. 29/133, Almaty, Kazakhstan. E-mail: z.karshigina@satbayev.university
<b>Lokhova Nina Georgievna</b>	Senior Researcher, "Institute of Metallurgy and Ore Beneficiation" JSC, Satbayev University, Shevchenko str. 29/133, Almaty, Kazakhstan. E-mail: n.lokhova@satbayev.university
<b>Yessengazyev Azamat Muratovich</b>	Ph.D., Junior Researcher, "Institute of Metallurgy and Ore Beneficiation" JSC, Satbayev University, Shevchenko str. 29/133, Almaty, Kazakhstan. E-mail: a.yessengazyev@satbayev.university
<b>Peter C Burns</b>	Professor of Civil & Environmental Engineering & Earth Sciences, Director, Center for Sustainable Energy at Notre Dame, South Bend, USA. E-mail: pburns@nd.edu

## Introduction

Niobium (Nb) is a rare metal used in high-quality steel production [1]. It is used in the production of ferroniobium which accounts for more than 90% of niobium production. The largest consumers of niobium are China, North America, and Europe. The substantial economic growth in emerging markets, especially the BRIC's economies, and increased use of niobium in steel production boosted prices for the metal up to US\$32.63/kg in 2007 and further up to the US \$60.00 per kg in 2012. Niobium is expected by analysts to be in demand in the near term, and prices for the metal will remain high.

What sets niobium apart is that it combines a high level of corrosion resistance with a low weight.

The material is used in coin inserts of all colors, corrosion-resistant evaporation bowls for use in coating methods, and mold-resistant crucibles for growing diamonds. Due to its high biocompatibility level, niobium is also used as an implant material. The high transition temperature also makes niobium an ideal material for superconducting cables and magnets [2].

Niobium belongs to the group of refractory metals. Refractory metals are metals whose melting point exceeds the melting point of platinum (1772 °C). The energy that binds individual atoms is extremely high in refractory metals. Refractory

metals are characterized by high melting points combined with low vapor pressure, high elasticity modulus, and high thermal stability. Besides, refractory metals have a low thermal expansion coefficient. Compared to other refractory metals, niobium has a relatively low density of only 8.57 g/cm<sup>3</sup> [3].

High-tech production of tantalum and niobium implants is planned in East Kazakhstan. Implants made of tantalum and niobium are considered an advanced technology in modern dentistry and orthopedics because of their high biocompatibility. One of the first enterprises in Kazakhstan that produced tantalum and niobium is Irtysh Chemical and Metallurgical Plant. The production is located in Pervomaisky settlement, Shemonaikha district of East Kazakhstan region. Irtysh Chemical and Metallurgical Plant was built in 1956 and it was the main enterprise in Kazakhstan for the production of niobium, tantalum, and rare-earth metals [4].

In Ust-Kamenogorsk, East Kazakhstan, Ulba Metallurgical Plant JSC produces ingots, rolled products, powders, and niobium pentoxide, as well as NbTi, and NbZr ingots. However, on export sales of rare metals, the volume of sales of niobium products has been decreasing in recent years for 2016 was 52.1 tons, for 2017. - 23.7 tons, for 2018 - 22.9 tons, for 6 months of 2019 - 0.1 tons [5]. It is most likely due to the shortage of niobium concentrates, both foreign and domestically produced.

Chlorination methods for niobium-containing mineral raw materials are known [[6], [7], [8], [9]]. Ore concentrate is chlorinated with chlorine gas in the presence of a reducing agent - charcoal or petcoke. Either briquette charge or ore concentrate is chlorinated in a salt melt. Besides, ore raw materials are chlorinated in autoclaves with liquid carbon tetrachloride or silicon tetrachloride. However, all of them have a number of disadvantages inherent to traditional chlorination; carbon monoxide, phosgene, hydrogen chloride, and chlorohydrocarbons are still produced.

In the modern hydrometallurgical industry extraction and sorption are widely used to improve the quality of raw materials and products and deep purification of technological solutions [[10],[11]].

Niobium is often a companion of titanium along with vanadium, zirconium, and hafnium; therefore, it is present in titanium concentrates obtained from ilmenite placer and ores [12].

Titanium and magnesium production generates thousands of tons of solid waste annually. Reserves

of wastes in sludge collectors at Ust-Kamenogorsk Titanium-Magnesium Plant JSC (UK TMP JSC) alone amount to 1270 thousand tons. These wastes contain 0.4 to 2 % of niobium, which is almost comparable with the content of niobium in industrial ores of pyrochlore carbonatites [13].

The accumulation of niobium occurs in the waste of titanium production during titanium-containing slag chlorination to produce titanium tetrachloride. It is established that 40-45 % of niobium from its content in the initial slag goes to the technical titanium tetrachloride, 50-55 % - into solid chloride sublimations, and 4-5 % into the drained melt of the chlorination unit [14].

The niobium content analysis in solid waste of titanium slag chlorination process for 2005-2007 at UK TMP JSC showed that the niobium content in the pulverized slag of the titanium chlorination unit was from 0.26 to 0.6 %. The subsoil is diluted with water, the resulting chloride sludge is discharged into the acid drain and then neutralized with lime milk under the existing technology.

Analysis of the available scientific and technical and patent literature shows that most of the studies have been performed for conditioned niobium-containing raw materials, while very few studies on niobium extraction from titanium production waste have been performed [[15], [16], [17], and there is no rational technology intended to produce intermediate products containing and concentrating niobium compounds in the amounts suitable for further use for titanium-magnesium production. Niobium extraction from the collector sludge into marketable concentrate will enable to extract the valuable component and dispose multitonnage waste.

## Materials and methods

*Materials and equipment* are sodium hydrogen carbonate, purity grade "chemically pure (C.P.)"; sodium hydroxide, purity grade "chemically pure (C.P.)"; glycerol, laboratory grade; sludge from the sludge collector of titanium-magnesium production provided by UK TMP JSC. Contents of the main sludge components of the titanium production sludge collector, mass in %: 12.70 TiO<sub>2</sub>; 0.73 Nb<sub>2</sub>O<sub>5</sub>; 32.90 CaO; 2.00 MgO; 3.40 Al<sub>2</sub>O<sub>3</sub>; 4.10 SiO<sub>2</sub>; 2.70 Fe<sub>2</sub>O<sub>3</sub>; 0.26 MnO; 0.29 V<sub>2</sub>O<sub>5</sub>.

IV-6 Vibro distiller (Russia), reactor equipped with a reflux condenser, ES VELP stirrer (Velp Scientifica), IKA RW16 stirrer (Germany), Shimadzu

scales (Japan), SNOL drying cabinet (Lithuania), distiller (Russia).

**Experimental method.** The necessary amount of leaching solution was put in the reactor, heated up to 95 °C, the specified quantity of sludge was brought in, and stirring was started up. Duration of pulp activation was 4 hours. The sludge was filtered at the end of the experiment. The sludge was washed with hot water to remove sodium hydroxide, glycerol, and soluble salts. The products were analyzed for the content of niobium, vanadium, aluminum, and silicon, the solubility of which in alkaline solutions is high enough.

**Analysis methods:** X-ray experimental data were obtained with the BRUKER D8 ADVANCE device with copper radiation at an accelerating voltage of 36 kV, and a current of 25 mA. X-ray fluorescence analysis was performed with a Venus 200 PANalytical B.V. (PANalytical B.V., Holland) spectrometer with wave dispersion. Chemical analysis of the samples was performed with an optical emission spectrometer with inductively coupled plasma Optima 2000 DV (USA, Perkin Elmer). Mapping of elemental and phase composition of the samples was executed on a JXA-8230 electron-probe microanalyzer by JEOL (Japan). Thermal analysis of the provided sample was executed using the device of synchronous thermal analysis - STA 449 F3 Jupiter (Germany).

## Results and discussion

The titanium production sludge was crushed, averaged, and dried before the physical and chemical studies. The result of the X-ray phase analysis is presented in Table 1 and Figure 1, where it is seen that the basis of the sludge is calcium carbonate, niobium is bound to calcium and embedded along with titanium in the lattice of aluminum oxide.

The main feature of thermal analysis is the possibility to determine small impurities on the background of the thermal inertness of the main components. It is seen on the derivatogram of the sludge sample from the sludge collector (Fig. 2) that endothermic effects of varying intensity are manifested on a rather long temperature segment of 100 - 900 °C on the DTA curve. They developed against the background of reducing the mass of the sample. Most likely, the manifestation of decomposition of various carbonates takes place here. Pyroaurite ( $Mg_6Fe_2(OH)_{16}CO_3 \times 4H_2O$ ), chantite

$CaMg_3(CO_3)_4$ , and calcite. For example, the combination of endothermic effects with extremes at 203.3 °C, 427 °C, and 820 °C is characteristic of the manifestation of lamellar pyroaurite with calcite.  $Mg_6Fe_2[CO_3](OH)_{16} \times 4H_2O$  is pyroaurite.

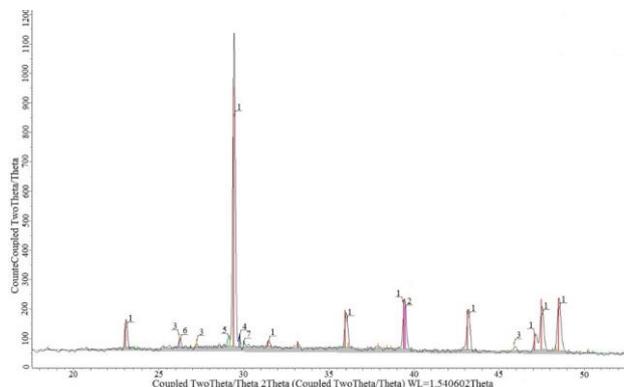


Figure 1 - Sludge diffractogram

Table 1 - Results of X-ray phase analysis of sludge

Phase number in Figure 1	Compound name	Formula	Content, %, relative.
1	Calcite	$Ca(CO_3)$	79.7
2	Aluminum Niobium Titanium	$Al_{0.12}Nb_{0.38}Ti_{0.5}O$	6.5
3	Aragonite	$Ca(CO_3)$	3.9
4	Calcite, magnesian	$(Mg_{0.129}Ca_{0.871})(CO_3)$	3.1
5	Fersmite	$CaNb_2O_6$	2.4
6	Iron(III) aluminium titanium oxide Iron Aluminum Titanium Oxide	$FeAlTiO_5$	2.3
7	Wollastonite-2M	$CaSiO_3$	2.1

The weak endothermic effect with the extremum at 307 °C on the DDTA curve shows melting of the  $FeCl_3$  impurity or sublimation of the  $HfCl_4$  impurity. The endothermic effect with extremum at 674.3 °C on the DTA curve shows the enantiotropic polymorphic transformation of  $2CaOSiO_2$ . It is mentioned in [18] that "peritectic equilibrium" is observed in the Al - Nb system at 661.4 °C. The endothermic effect with extremum at 765.8 °C characterizes the melting of  $CaCl_2$ , KCl,  $NdCl_3$  impurities.

Endothermic effect with extremum at 918.7 °C shows polymorphic transformation of strontium carbonate. The weak endothermic effect with extremum at 1219.4 °C shows the phase transition of Nb<sub>2</sub>O<sub>5</sub>Al<sub>2</sub>O<sub>3</sub> impurity. The kink at 1270.4 °C may show the melting of the sample.

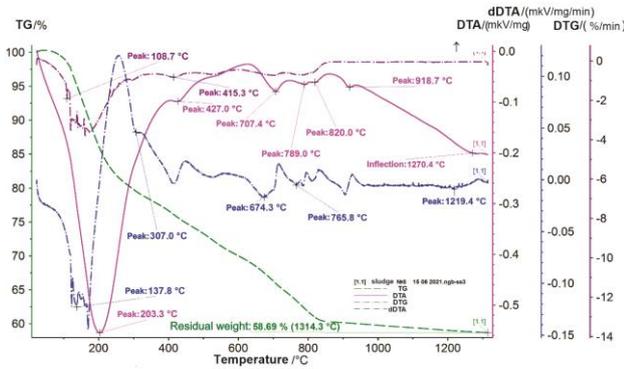


Figure 2 - Derivatogram of the sludge sample from the sludge collector (sample size of 0.2 g)

Endothermic effect with extremum at 918.7 °C manifests the polymorphic transformation of strontium carbonate. The weak endothermic effect with extremum at 1219.4 °C shows the phase transition of Nb<sub>2</sub>O<sub>5</sub>Al<sub>2</sub>O<sub>3</sub> impurity. The kink at 1270.4 °C may show the melting of the sample. Thus, the thermal analysis data confirm the presence of a niobium-aluminum bond.

Next, the sludge deposit was studied on an electron-probe microanalyzer. The electron-probe study was executed in COMPO mode - backscattered electron imaging of minerals. A peculiarity of the COMPO image is an increased resolution enabling to register images of particles with small sizes. Thus, bright small particles including niobium compounds became visible at image magnification of 1000 times (Fig. 3).

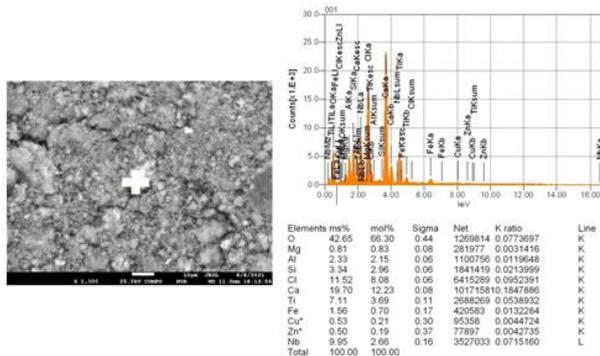


Figure 3 - COMPO image of the sludge from the sludge collector and its particle spectrum

Thus, the physicochemical studies of the phase composition of the sludge collector have shown that niobium is mainly bound to aluminum and titanium in oxide compounds. The particles of these compounds are very small and surrounded by clay and carbonate component of the sludge collector.

**Activation of the sludge from sludge collector before leaching.** The main task of sludge activation is to release the surface of niobium-containing particles. It will ensure effective access of the leaching agent and increase the niobium extraction degree into the solution.

Aragonite turns into calcite as a result of sludge activation with sodium hydrogen carbonate solution at elevated temperature. Chloride ion passes into the solution and calcite with magnesium forms a solid solution of (Mg<sub>0.03</sub>Ca<sub>0.97</sub>)CO<sub>3</sub> (Figure 4, Table 2). The duration of the experiments was 0.5 h, 2 h, 4 h, and 6 h at 120 °C. The disadvantages of this method shall be referred to the accumulation of chlorine ion in the sodium bicarbonate solution during solution turnover.

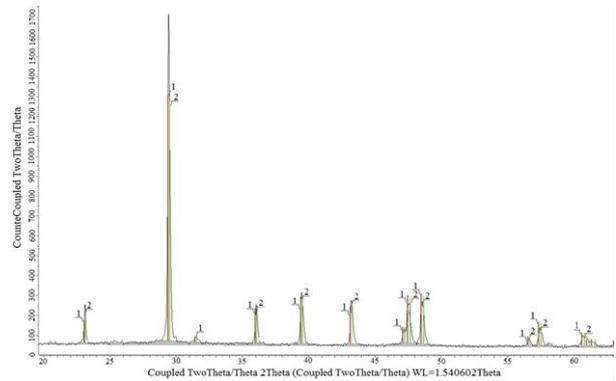


Figure 4 - Diffractogram of activated sludge from the sludge collector (6 h, 120 °C, 60 g/dm<sup>3</sup> NaHCO<sub>3</sub>)

Table 2 - Results of X-ray phase analysis of activated sludge from sludge collector

Phase number in the figure 4	Compound name	Formula	Contents content, %, t.r.f.
1	Calcite, magnesian, syn	(Mg <sub>0.03</sub> Ca <sub>0.97</sub> )CO <sub>3</sub>	50.3
2	Calcite, syn	Ca(CO <sub>3</sub> )	49.7

After activation of sludge from the sludge collector with sodium bicarbonate solution, the test object was mechanically activated on a vibrating scrubber. The effect of the mechanical activation

duration on the disperse composition of the sludge from the sludge collector is shown in Table 3. Studies were executed during 0,5 - 5 min [19].

The crystal lattice deformation of the main components of the sludge from the sludge collector was estimated by means of X-ray diffraction analysis. Diffractograms of the initial and 5 min activated sludge from the sludge collector in the superposition are shown in Figure 5. A comparison of diffractograms showed distortion of the crystal lattice of calcite and aragonite in the activated sludge sample from the sludge collector.

Table 3 - Effect of mechanical activation duration on the disperse composition of sludge

Activation time, min	Fraction, mm				Losses, %
	+0.315	-0.315 +0.1	-0.1 +0.064	-0.064 +0.056	
0	26.7	50.5	18.2	3.6	1.0
0.5	17.7	69.1	11.5	0.8	0.9
1.0	17.7	72.1	8.8	0.9	0.5
2.0	16.0	76.2	6.0	0.9	0.9
3.0	13.5	80.1	49.0	0.5	1.0
5.0	7.0	89.3	2.2	0.5	1.0

As follows from the data in Table 3, the share of large +0.315 and small - 0.1+0.056 mm particles significantly decrease with an increase in mechanical activation duration. Probably, the fine particles are mutually attracted to the formation of agglomerates during the abrasion of sludge from the sludge collector. From Fig. 6, it can be seen that there are also larger particles in the image of the sludge activated for 5 min besides the fine particles. Note that the presence of small particles in the image of activated sludge is a reflection of particles containing niobium absent in the image of the original product. Thus, mechanoactivation of sludge from the sludge collector provides a contact of the reagent with the surface of niobium-containing particles which will increase the leaching process efficiency.

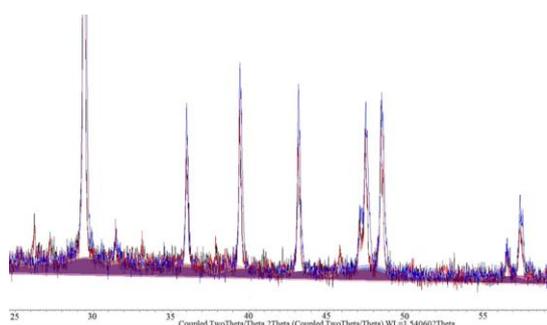


Figure 5 - Diffractograms of the initial and activated sludge from the sludge collector in overlay (red - original, blue - activated)

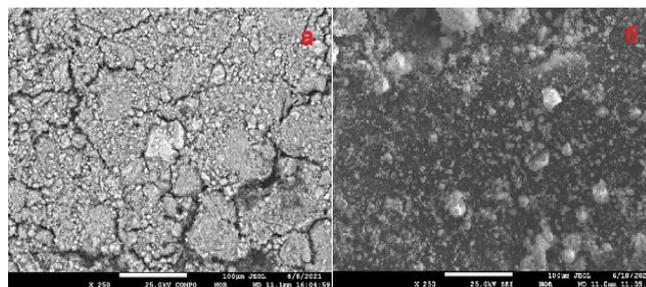


Figure 6 - Images of the initial (a) and activated (b) sludge from the sludge collector

**Study of the sludge leaching process with an alkali solution.** It is known [20] that niobium compounds are soluble not only in strong acids but also in strong alkali solutions indicating the presence of marked signs of amphotericity. The niobium extraction in solution occurs in solutions of caustic alkalis, due to the formation of water-soluble niobates with a  $\text{Na}_2\text{O}/\text{Nb}_2\text{O}_5$  ratio greater than one [21].

It was found in [22] that the solubility of niobium oxide increases tens of times in alkaline solutions containing the five-atom alcohol xylite. It was noted that the solubility was  $3.9 \text{ mg/dm}^3 \text{ Nb}_2\text{O}_5$  in a pure alkaline solution with a sodium hydroxide concentration of  $160 \text{ g/dm}^3$ .

It should be noted that preliminary experiments showed that the use of alkaline solution with sodium hydroxide content of more than  $200 \text{ g/dm}^3$  in the presence of glycerol results in the formation of poorly filterable pulp and requires a large amount of water to wash the sludge. In this regard, studies on the niobium extraction in solution were executed with solutions of 100 and  $200 \text{ g/dm}^3 \text{ NaOH}$ .

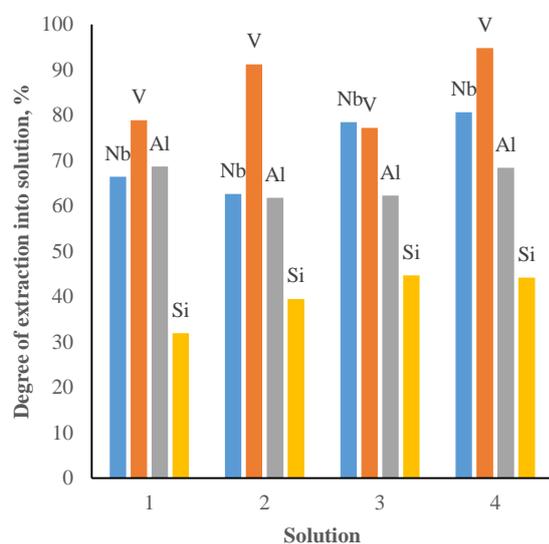
From the data in Table 4, it can be seen that increase in the concentration of alkali in the solution containing  $2 \text{ g/dm}^3$  of glycerol from 100 to  $200 \text{ g/dm}^3$  has practically no effect on the concentration of soluble aluminum compounds. At the same time, the aluminum extraction degree into the solution is 68.7 and 62.4 rel. %, respectively. An increase in the glycerol content up to  $5 \text{ g/dm}^3$  has little effect on the extraction degree of aluminum compounds in the solution (Figure 7).

The concentration of sodium hydroxide and glycerol has no noticeable effect on the content of sodium metasilicate in the solution under the studied conditions of sludge leaching (Table 4).

Table 4 - Effect of glycerol on the transition of niobium, vanadium, aluminum, and silicon in alkaline solution under hydrothermal conditions (S: L = 1:10, 95 °C, 240 min)

Concentration in solution, g/dm <sup>3</sup>		Cake output, %	Content in the cake, wt. %				Concentration in solution, mg/dm <sup>3</sup>			
NaOH	glycerin		Nb <sub>2</sub> O <sub>5</sub>	V <sub>2</sub> O <sub>5</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Nb <sub>2</sub> O <sub>5</sub>	V <sub>2</sub> O <sub>5</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>
100	2	64.6	0.375	0.126	1.626	2.880	282	179	1.353	1.094
100	5	71.0	0.380	0.048	2.275	2.230	301	235	1.157	1.039
200	2	61.2	0.254	0.144	2.065	3.567	378	199	1.391	1.177
200	5	63.0	0.222	0.032	2.069	3.180	386	244	1.187	1.164

It is known [23] that the solubility of iron oxide (III) in sodium hydroxide solution (200-240 g/dm<sup>3</sup>) even at 120-220 °C is ~ 0.1 g/dm<sup>3</sup>. Therefore, the behavior of iron was not controlled.



1 - NaOH 100, glycerin 2; 2 - NaOH 100, glycerin 5; 3 - NaOH 200, glycerin 2; 4 - NaOH 200, glycerin 5 (95 °C, 4 h, S: L = 1:10)

Figure 7 - Dependence of niobium, vanadium, aluminum, and silicon extraction rates on leaching solution composition, g/dm<sup>3</sup>

The degree of niobium extraction in the solution is greatly influenced by the concentration of alkali - by increasing the concentration of caustic soda from 100 to 200 g/dm<sup>3</sup> the degree of extraction increases by 1.2-1.3 times. The maximum concentration of niobium in the solution is 380 mg/dm<sup>3</sup>. Glycerol in this case restrains the transition of hexaniobate formed during the interaction of alkali with sludge into insoluble sodium metaniobate.

It is of interest to study the behavior of another valuable component of sludge - vanadium. Since the acid properties of vanadium are higher than those of

niobium, the extraction degree into the solution under the studied conditions reaches 91-95%. The effect of the solid-to-liquid ratio was studied in the range 1:(4÷12).

Figure 8 shows that an increase in the S: L ratio and, accordingly, the alkali consumption rate results in an increase in the degree of niobium transfer into the solution up to S: L = 1: 10, and with a further increase in the ratio that becomes practically constant. Vanadium extraction into the alkaline solution also increases with the increase of the S: L ratio. At the same time the vanadium transfer degree into the solution is higher than niobium.

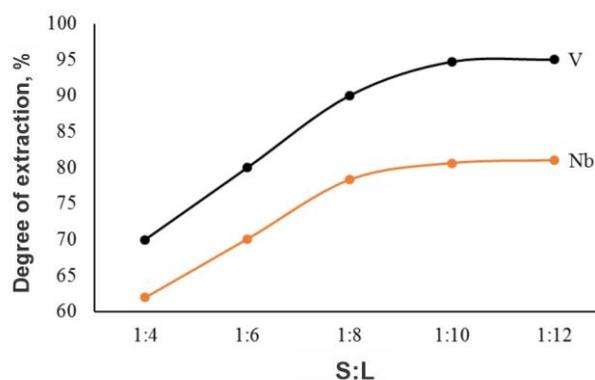


Figure 8 - The dependence of the niobium and vanadium extraction degree on the ratio S: L (NaOH 100 g/dm<sup>3</sup>, glycerol 2 g/dm<sup>3</sup>, 95 °C, 4 h)

Thus, experimentally it was found that the optimum conditions for niobium extraction in the solution of sludge from the sludge collector of titanium-magnesium production - sodium hydroxide concentration of 200 g/dm<sup>3</sup>, glycerol 5 g/dm<sup>3</sup>, S: L = 1:10, temperature 95 °C, the duration of the leaching process 4 h.

## Conclusions

Physical and chemical properties of sludge collector wastes were studied. Chemical and X-ray fluorescence analyses determined the content of the main components, wt. %: 12.7 TiO<sub>2</sub>; 0.73 Nb<sub>2</sub>O<sub>5</sub>; 32.9 CaO; 2.0 MgO; 3.4 Al<sub>2</sub>O<sub>3</sub>; 4.1 SiO<sub>2</sub>; 2.7 Fe<sub>2</sub>O<sub>3</sub>; 0.26 MnO; 0.29 V<sub>2</sub>O<sub>5</sub>. X-ray phase analysis showed the following phase composition: calcite 79.7 %, aluminum-niobium-biotite-titanium oxide 6.5 %, aragonite 3.9 %, magnesian calcite 3.1 %, fersmite 2.4 %, iron-aluminum-titanium oxide 2.3 %, wollastonite 2.1 %. The base of the sludge deposit is calcium carbonate, with niobium bound to calcium and embedded along with titanium in the aluminum oxide lattice.

Thermal analysis confirmed the presence of calcite, pyroaurite, calcium, potassium chlorides, and niobium bond with aluminum.

It was found that aragonite transforms into calcite when the sludge is activated with sodium hydrocarbonate solution at elevated temperatures. Chloride ion passes into the solution, and calcite with magnesium forms a solid solution of  $(Mg_{0.03}Ca_{0.97})CO_3$ . It was determined that with an increase in mechanical activation duration the proportion of large  $+0.315$  and small  $-0.1+0.056$  mm particles significantly decreases. Probably, the fine particles are mutually attracted to the formation of agglomerates during sludge abrasion. The process of sludge leaching by alkali solution was studied. If the caustic soda concentration is increased from

100 to 200  $g/dm^3$ , the extraction degree of niobium increases 1.2-1.3 times, and the maximum concentration of niobium in the solution reaches 380  $mg/dm^3$ . Glycerol in this case restrains the transition of hexaniobate formed during the interaction of alkali with sludge into insoluble sodium metaniobate. The extraction of vanadium in the alkaline solution also increases with the increase in the S: L ratio. At the same time, the degree of vanadium transition into solution is higher than that of niobium.

The optimum conditions for the niobium extraction from the titanium-magnesium production sludge - sodium hydroxide concentration 200  $g/dm^3$ , glycerol 5  $g/dm^3$ , S: L = 1:10, temperature 95 °C, leaching process duration 4 h.

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## Титан-магний өндірісінің шламжинағыш шөгіндісінен ниобийді ерітіндіге шығара отырып, гидрометаллургиялық өңдеу бойынша зерттеулер

<sup>1</sup> Ультаракова А.А., <sup>1</sup>Қаршыға З.Б., <sup>1</sup>Лохова Н.Г., <sup>1</sup>Есенгазиев А.М., <sup>2</sup>Бернс П.С.

<sup>1</sup> «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Алматы, Қазақстан

<sup>2</sup> Нотр-Дам университеті, «Тұрақты энергия орталығы» АҚШ, Саут-Бенд, АҚШ

<b>ТҮЙІНДЕМЕ</b>	
<p>Мақала келді: 29 сәуір 2022 Сараптамадан өтті: 31 тамыз 2022 Қабылданды: 06 қазан 2022</p>	<p>Химиялық, рентгендік және микронзондты талдау әдістерімен титан-магний өндірісінің шламжинағыш шөгінділерінің заттық құрамы зерттелді. Шламжинағыш шөгіндісінің фазалық құрамын зерттеу ниобий негізінен алюминий және титанның оксидті қосылыстарына қосылатындығын көрсетті. Бұл қосылыстардың бөлшектері өте кішкентай және шламжинағыш шөгіндісінің сазды және карбонатты компонентімен қоршалған. Заттық құрамы бойынша алынған деректер негізінде титаномагний өндірісінің шламжинағыш шөгінділерінің химиялық және механикалық белсендіру процестері жүзеге асырылды. Алдын ала белсендіруден кейін шламжинағыш шөгіндісін сілтілі шаймалау жүргізілді. Титаномагний өндірісінің шламжинағыш шламынан ниобийді алудың оңтайлы шарттары белгіленді: натрий гидроксидінің концентрациясы 200 <math>г/дм^3</math>, глицерин 5 <math>г/дм^3</math>, Т:Ж = 1:10, температура 95 °C, шаймалау процесінің ұзақтығы 4 сағат. Шламды сілтімен шаймалау кезінде глицерин қосу гексаниобаттың ерімейтін натрий метаниобатына ауысуын тежейді, бұл ниобийді алу дәрежесін 1,3 есеге арттырып, алынатын металды 80% - ға дейін ерітіндіге ауыстыруға мүмкіндік береді.</p> <p><b>Түйін сөздер:</b> шламжинағыш шөгіндісі, ниобий, белсендіру, шаймалау, натрий гидроксиді.</p>
<b>Авторлар туралы ақпарат:</b>	
<b>Ультаракова Алмагуль Амировна</b>	Техника ғылымдарының кандидаты, жетекші ғылыми қызметкер, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Шевченко көш., 29/133, Алматы, Қазақстан. E-mail: <a href="mailto:a.ultarakova@satbayev.university">a.ultarakova@satbayev.university</a>
<b>Қаршыға Зәуре Байтасқызы</b>	PhD докторы, жетекші ғылыми қызметкер, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Шевченко көш., 29/133, Алматы, Қазақстан. E-mail: <a href="mailto:z.karshigina@satbayev.university">z.karshigina@satbayev.university</a>
<b>Лохова Нина Георгиевна</b>	Аға ғылыми қызметкер, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Шевченко көш., 29/133, Алматы, Қазақстан. E-mail: <a href="mailto:n.lokhova@satbayev.university">n.lokhova@satbayev.university</a>

<b>Есенгазиев Азамат Муратович</b>	PhD докторы, кіші ғылыми қызметкер, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Шевченко көш., 29/133, Алматы, Қазақстан. E-mail: a.yessengaziyev@satbayev.university
<b>Питер С Бернс</b>	Азаматтық және экологиялық инженерия және жер туралы ғылымдар профессоры, Нотр - Дамдағы Тұрақты энергия орталығының директоры, Саут-Бенд, АҚШ. E-mail: pburns@nd.edu; O

## Исследования по гидрOMETаллургической переработке осадка шламонакопителя титаномагниевого производства с извлечением ниобия в раствор

<sup>1</sup> Ультаракова А.А., <sup>1</sup>Каршыға З.Б., <sup>1</sup>Лохова Н.Г., <sup>1</sup>Есенгазиев А.М., <sup>2</sup>Бернс П.С.

<sup>1</sup>АО «Институт металлургии и обогащения», Satbayev University, Алматы, Казахстан

<sup>2</sup>Университет Нотр-Дам, «Центр устойчивой энергетики» США, Саут-Бенд, США

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### АННОТАЦИЯ

Изучен вещественный состав осадков шламонакопителей титаномагниевого производства методами химического, рентгеновского и микрозондового анализа. Исследования фазового состава осадка шламонакопителя показало, что ниобий связан в основном с алюминием и титаном в оксидные соединения. Частицы этих соединений очень мелкие и окружены глинистой и карбонатной составляющей осадка шламонакопителя. На основании полученных данных по вещественному составу, осуществлены процессы химической и механической активации осадка шламонакопителя титаномагниевого производства. Проведен щелочное выщелачивание осадка шламонакопителя после предварительной активации. Установлены оптимальные условия извлечения ниобия из шлама шламоотвала титаномагниевого производства: концентрация гидроксида натрия 200 г/дм<sup>3</sup>, глицерина 5 г/дм<sup>3</sup>, Т:Ж = 1:10, температура 95 °С, продолжительность процесса выщелачивания 4 ч. Добавление глицерина при щелочном выщелачивании шлама ингибирует переход гексаниобата в нерастворимый метаниобат натрия, что позволяет увеличить степень извлечения ниобия в 1,3 раза с переводом в раствор до 80 % извлекаемого металла.

**Ключевые слова:** осадок шламонакопителя, ниобий, активация, выщелачивания, гидроксида натрия.

### Информация об авторах:

<b>Ультаракова Алмагуль Амировна</b>	Кандидат технических наук, ведущий научный сотрудник, АО «Институт металлургии и обогащения», Satbayev University, ул. Шевченко, 29/133, Алматы, Казахстан. E-mail: a.ultarakova@satbayev.university
<b>Қаршыға Зәуре Байтасқызы</b>	Доктор PhD, ведущий научный сотрудник, АО «Институт металлургии и обогащения», Satbayev University, ул. Шевченко, 29/133, Алматы, Казахстан. E-mail: z.karshigina@satbayev.university
<b>Лохова Нина Георгиевна</b>	Старший научный сотрудник, АО «Институт металлургии и обогащения», Satbayev University, ул. Шевченко, 29/133, Алматы, Казахстан. E-mail: n.lokhova@satbayev.university
<b>Есенгазиев Азамат Муратович</b>	Доктор PhD, младший научный сотрудник, АО «Институт металлургии и обогащения», Satbayev University, ул. Шевченко, 29/133, Алматы, Казахстан. E-mail: a.yessengaziyev@satbayev.university
<b>Питер С Бернс</b>	Профессор гражданской и экологической инженерии и наук о Земле, директор Центра устойчивой энергетики в Нотр-Даме, Саут-Бенд, США. E-mail: pburns@nd.edu

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