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Study of the efficiency of using pelletizing poor scheelite ore for leaching tungsten

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ABSTRACT

Heap leaching technology has many advantages, the most important of which are the minimum operating and capital costs. The research aims to intensify tungsten extraction by heap leaching from a pelletized sample of scheelite ore with solutions of hydrochloric and oxalic acids. The Kappes percolation test was used to control the quality of agglomeration operations and determine the requirements for ore agglomeration. Building dependencies were used to study leaching and recovery rates. A scheme of heap leaching of pelletized scheelite ore using a solution of hydrochloric and oxalic acids as a solvent of scheelite has been developed. Technology provides conditions that minimize the loss of oxalic acid with oxalate precipitation and the use of productive solutions in circulation. The behavior of granules in the percolation leaching method indicates the effectiveness of pre-pelletizing a sample of scheelite ore with the Alcotac®CB6 reagent. During the leaching process, the pelletized material has sufficient porosity and permeability while maintaining the integrity of the granules. It provides access to leaching solutions to the surface of valuable metals. During leaching, the operation of the initial ore with a solution of hydrochloric acid is provided. During this operation, rock minerals leach, which helps reduce oxalic acid consumption during subsequent tungsten leaching.

Keywords: ore, reagent, pellets, percolation leaching, extraction, tungsten, oxalic acid solution.

INTRODUCTION

Tungsten is included in strategic reserves in many countries and occupies an indispensable position due to its low global reserves and high demand [1-7]. Tungsten forms several minerals, which are, as a rule, compounds formed by its trioxide with oxides of other metals, mainly iron, manganese, calcium, copper, and lead.

Hydrometallurgical methods for processing ores and concentrates are based on decomposing raw materials by mineral acids or alkalis at normal or elevated pressure. Of the acidic processing methods, the method of processing concentrates with hydrochloric, sulfuric and nitric acids to obtain industrial tungstic acid deserves attention [8, 9]. However, this method has several disadvantages associated with increased acid consumption, which is necessary for the dissolution of not only tungsten-bearing minerals but also components of waste rock. The solutions obtained in this case are characterized by high corrosion activity and complex composition. Acidic decomposition methods of tungsten-containing raw materials are used in the industry to process rich scheelite concentrates. Hydrometallurgical methods are the most suitable for extracting tungsten from the weathering crust. In 2016-2018, Dala Mining conducted a series of hydrometallurgical tests focused on underground leaching.

Heap leaching technology has many advantages, such as minimal operating and capital costs. In the heap leaching method, crushed and/or pelletized ore is stacked (pile), and the initial solution is fed from above the pile, passing through the ore layer. The productive solution enriched with the extracted metal is collected in storage ponds at the base of the pile.

However, due to their culmination, the leaching of clay and slurry ores dramatically reduces the seepage of solution through the ore layer and sometimes completely stops the process. To improve the filtration properties of the stack during heap leaching, technologies for pelletizing clay ores are used [10-16]. These technologies are widely used in the industrial mining of gold-bearing ores of the weathering crust gold mining dumps with a high content of clay fractions. They aim to create a durable and porous material that retains its properties during pile formation and leaching of raw materials.

A method known [14] involves pelletizing various types of ores with an average consumption of cement per ton of ore – 5-6 kg, bleach – 3.5-4 kg. As a result, the strength of the pelletized ore with such a binder content is 73.5-78.4 kPa. A further increase in the binder content leads to a decrease in the permeability of the pelletized ore, an increase in the leaching time, and an increase in the cost of the ore preparation process. In addition, when pelletizing clay ores and enrichment tailings, an increase in cement can lead to the opposite effect, manifested in a decrease in the permeability of pellets [15].

In [17], a laboratory test was performed to study the permeability of a pile of rare earth elements with adsorbed ions under conditions of lateral liquid injection.

In [18], laboratory tests were carried out on percolation leaching of stale copper tailings pelletized with a granulator using an organic binder Alcotac® CB6 (BASF, Germany).

These technologies have widely applied in the industrial mining of weathering crust ores, placers, and indigenous gold mining dumps with a high clay fraction content [19, 20].

Our research aims to increase the filtration properties of a pile of heap leaching of clay, oxidized, and scheelite ores, which are prone to culmination. As a result, the seepage of solution through the ore layer is sharply reduced. Solutions of hydrochloric and oxalic acids are used as a solvent for scheelite. Hydrochloric acid is necessary for the acidification of rocks, leaching of rock-forming acid-soluble minerals, and opening iron-manganese hydroxides with tungsten. Oxalic acid is needed to form water-soluble oxalate complexes of tungsten in an acidic environment.

Thus, the dissolution of synthetic scheelite in solutions of oxalic acid ($H_2C_2O_4$) was studied in [21-23]. When examining the mixing rate, temperature, oxalic acid concentration and particle size for the dissolution of scheelite, it was found that the dissolution reaction proceeds in two stages. First, an intermediate chelated compound of calcium aqua oxalate tungstate ($Ca[WO_3(C_2O_4)H_2O]$) is formed. Secondly, the resulting compound reacts with $H_2C_2O_4$ to form water-soluble hydrogen aquaoxalate tungstate ($H_2[WO_3(C_2O_4)H_2O]$) and solid $CaC_2O_4H_2O$. The resulting $CaC_2O_4H_2O$ covered the surfaces of unreacted $CaWO_4$ particles as a protective layer, and the dissolution reaction was interrupted after a certain transformation at high temperatures and concentrations of oxalic acid [22].

That is, the dissolution of scheelite with oxalic acid solutions proceeds with the formation of a solid product – calcium oxalate, which inhibits the diffusion of the tungsten oxalate complex (reaction product) from the surface of the scheelite grain into a film of an aqueous solution wetting a piece of ore, as a result of which the leaching of tungsten can completely stop.

Thus, using oxalic acid solutions without additives of other mineral acids is associated with incomplete extraction of tungsten and high consumption.

For a mixture of hydrochloric and oxalic acids, leaching is described by the reaction:

$$CaWO_4 + 2HCl + H_2C_2O_4 = CaCl_2 + H_2WO_3\ C_2O_4 + H_2O \qquad (1)$$

In sufficiently acidic solutions, oxalate complexes of iron (II), aluminum, and manganese are formed in small quantities, since their stability decreases with increasing acidity, and calcium and magnesium oxalates dissolve in excess of hydrochloric acid.

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At the same time, the main minerals (calcite, siderite, chlorite, muscovite are leached by reaction:

$$CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2 \quad (3)$$

$$FeCO_3 + 2HCl = FeCl_2 + H_2O + CO_2 \quad (4)$$

$$(Mg,Fe)_3Si_4O_{10}(OH)_2{\cdot}(Mg,Fe)_3(OH)_6 + 12HCl = \\ =3MgCl_2 + 3FeCl_2 + 4H_4SiO_4 + 2H_2O \quad (5)$$

$$KAl_2(AlSi_3O_{10})(OH)_2 + 10HCl = KCl + 3AlCl_3 + 3H_4SiO_4 \quad (6)$$

The course of these reactions depends on the acidity of the solution used. Carbonates and anorthite are dissolved first, followed by muscovite and chlorite.

When using sulfuric acid solutions instead of HCl, one product of the dissolution of calcium minerals is gypsum, which forms a dense shell around ore particles, blocks the surface of mineral grains and prevents their dissolution. As a result, sulfuric acid does not allow for a sufficient degree of tungsten extraction with minimal oxalic acid consumption.

As can be seen from the reaction equations, the reaction products are water-soluble compounds, except for silicic acid. As a result, surface films that inhibit scheelite leaching are not formed or are formed in small quantities. With a high content of calcium in the productive solution, precipitation of its oxalate may occur:

$$CaCl_2 + H_2C_2O_4 = CaC_2O_4 + 2HCl \quad (7)$$

In addition, the highest local calcium concentrations are formed on the surface of calcium minerals that dissolve in acidic chloride-oxalate solutions. In this case, calcium oxalate can precipitate on the surface of calcite, anorthite, apatite and scheelite, slowing down the leaching of tungsten and rock minerals. The degree of these reactions depends on the initial contents in the ore, on the ratio "ore/leaching solution" (S:L) and concentrations of reagents, on which, in turn, the concentrations of reaction products depend during the leaching process.

The research aims to intensify tungsten extraction by heap leaching from a pelletized sample of scheelite ore with solutions of hydrochloric and oxalic acids.

MATERIAL AND METHODS

Oxidized poor scheelite ore (Republic of Kazakhstan) was used as the object of the study. The results of the chemical and granulometric compositions of the ore sample are shown in **Table 1** and **Table 2**.

Table 1 Chemical composition of the initial ore sample, %

W	Mo,g/t	Cu	Bi,g/t	Ca	Mg	K	Na	Al
0.25	114	0.40	849	14.7	0.46	0.60	0.32	4.62
SiO_2	Fe_{total}	Pb	Zn	Mn	Ti	S	P	As,g/t
36.70	6.5	0.03	0.09	1.33	0.12	0.11	0.13	40

Table 2 Granulometric composition of the sample with the distribution of the size of the controlled components by class

Class, mm	Recove-ry, %	Components, %					Distribution, %			
		W	Mo, g/t	Cu	Ca	Fe_{total}	W	Mo	Cu	Ca
+ 20	3.11	0.35	125.5	0.13	20.71	7.04	4.37	3.43	1.05	4.39
- 20 + 10	4.75	0.24	119.8	0.31	14.34	6.80	4.50	5.00	3.75	4.64
- 10 + 5	17.48	0.23	97.0	0.42	13.05	6.64	16.22	14.90	18.65	15.55
- 5 + 2.5	17.66	0.22	93.8	0.40	14.81	6.83	15.75	14.55	17.99	17.82
- 2.5 + 1.25	7.74	0.20	89.5	0.38	16.71	7.00	6.31	6.08	7.43	8.81
- 1.25 + 0.5	18.10	0.21	92.0	0.40	16.80	6.61	15.42	14.62	18.67	20.72
- 0.5 + 0.315	7.70	0.23	99.5	0.38	12.50	6.42	6.99	6.72	7.39	6.56
-0.315 + 0.1	9.65	0.28	137.9	0.36	15.68	5.81	10.78	11.6	8.97	10.31
- 0.16 + 0	13.81	0.35	190.0	0.46	11.89	5.40	19.64	23.0	16.11	11.19
Initial ore	100.0	0.25	114	0.40	14.7	6.5	100.0	100.	100.0	100.0

The data in **Table 2** show that class -5 +0 mm ore content is 74.66%, of which 13.81% is in class -0.16+0. The highest tungsten content – 0.35 %—is observed in grades +20 mm and -0.16+0 mm.

The results of the phase analysis of the ore sample according to the degree of copper oxidation show that the sample contains 93.25% oxidized minerals and 6.75% sulfide.

For ores with a high clay or fines content, agglomeration is often the most important step in the ore processing process to achieve suitable blade permeability and maximum metal recovery.

The material of the scheelite ore sample was crushed to a class -20 + 0 mm, after which it is subject to pelletizing. Additives were used as a binding material:

- cement grade PC-400 (with a consumption of 6 kg per ton of ore);
- BASF organic binder - Alcotac CB6 (with a consumption of 1 kg/t);
- without adding a binder (on water).

The Kappes percolation test (Kappes A/P) Kappes, Cassiday [24] was used to control the quality of ore agglomeration operations.

Before the research started, the pelletized ore (granules) loaded into the column was kept in water for 2 hours. Then, the initial height of the ore layer (H1), the height after settling in water (H2), the height after shaking the column (H3), and the height after draining the water (H4) were fixed.

During the percolation leaching of the sample, a transparent plastic column 1 m high with an internal diameter of 140 mm was used. The pelletized ore weighing 14 kg was loaded into columns and acidified with a hydrochloric acid solution with an initial concentration of 20 g/l, then leached with oxalic acid. The solution released from the column was reinforced with hydrochloric acid and returned to the column. The initial solution was supplied by a Qdos 60 peristaltic pump from a collecting tank to a distribution device installed in the upper part of the column, which evenly distributed the solution over the section of the column. The solution seeped through the ore layer in the column and was collected in a collecting tank. From the collection, the solution was either put into circulation or sent for processing. The process was controlled by taking average daily samples at the inlet and outlet of the column, with fixation on the volume of the solution passed per day. The samples were analyzed using ICP-OES or X-ray fluorescence according to certified methods.

The solution's acidity was analyzed using titrimetric methods, which are known methods.

After leaching, the solutions were adjusted according to the concentration of hydrochloric and oxalic acids and fed into the column following the technological scheme. The productive solutions were returned to circulation or removed from the cycle.

Such an organization of tests made it possible to fully reproduce the technological scheme under test, control the behavior of useful and impurity components, track tungsten extraction (from ore into solution) at each operation, and obtain consumption indicators for reagents.

The irrigation density during the tests was set to 240 ml/hour and varied, if necessary, by adjusting the solution's flow rate. This feed rate is usually used in copper production for low-permeability ores and was chosen specifically to simulate a pessimistic scenario in the case of severe culmination of the material in the column.

In real conditions, as the test results later showed, the irrigation density can be significantly increased without the danger of creating conditions of saturated hydraulic conductivity, fraught with loss of stability of the ore stack.

After leaching, the ore (cake) was washed and unloaded into parts (the upper and lower parts of the column).

RESULTS AND DISCUSSION

In experiments using cement and without a binder, the destruction of ore in the column occurs due to the static pressure of the overlying upper part of the granules exceeding the strength of the pelletized ore. After complete solution saturation, the content of coarse-grained fractions decreases, and the slurry fraction present before pelletizing reappears in the ore composition (**Fig. 1**).

When using Alcotac CB6, the granules retain their integrity and shape. **Fig. 1** shows granules using water, cement, and Alcotac CB6 as a binder before and after percolation.

Based on the data obtained, a drop in the height of the ore layer level was calculated. The results are presented in **Table 3**.

Table 3 Kappes test results

Binder name	Layer height, mm			
	H1, init.	H2	H3	decline, %
w/o binder	225	195	195	13.33
cement PC-400	210	175	175	16.67
Alcotac, 1000 g/t	245	245	245	0.0

The experiments carried out found that using Alcotac CB6 binder for pelletizing ensures the maximum safety of granules.

To reduce the consumption of the Alcotac CB6 binder during the pelletizing of scheelite ore, tests were carried out to select its consumption in the range from 200 - 1000 g/t. The tests were performed according to the previously described Kappes—Cassiday method; the results are presented in **Table 4**.

The data obtained show that when using the Alcotac CB6 binder with a flow rate between 200 and 1000 g/t, the height of the granule layer practically does not decrease.

When studying the permeability of ore or determining the critical degree of flushing (the rate at which the material is flooded in the column), the intensity of water supply to the column was defined as 1769.28 dm^3/(m^2*hour) in 90 minutes for pellets pelletized with a binder consumption of 400 g/t.

Table 4 Kappes test results

Binder consumption (Alcotac), g/t	Layer height, mm			decline, %
	H1, init.	H2	H3	
200	213	200	200	6,103
400	210	205	205	2.381
600	250	249	249	0.400
1000	250	250	250	0.000

For pellets with a binder consumption of 600 g/t, the critical degree of flushing was achieved in 18 minutes at a water supply rate of 1769.28 dm^3/(m^2*hour).

Studies show that pellets using Alcotac CB6 as a binder are characterized by high permeability, which is a favorable factor for processing the ore under study by heap leaching. At the same time, the binder consumption can be varied between 600-1000 g/t.

For further studies on percolation leaching in columns 1 meter high, ore pelletizing was carried out using the Alcotac CB6 reagent, at a flow rate of 900 g/t.

Percolation leaching of the pelletized ore was carried out with acidification with hydrochloric acid and further leaching with oxalic acid. Technology provides conditions that minimize the loss of oxalic acid with oxalate precipitation and the use of productive solutions in circulation. For this purpose, acidification of the initial ore with hydrochloric acid solutions is provided. During this operation, rock minerals are leached by reactions (3-6). The $CaWO_4$ mineral is relatively stable in diluted hydrochloric acid solutions, achieving selective rock minerals leaching and reducing oxalic acid consumption during subsequent tungsten leaching.

After the ore was acidified, the tungsten leaching operation was carried out by reaction (1). The results of acidifying the ore sample with hydrochloric acid are indicated in Figure 2 in the range from 0 to 15 of the specific daily flow rate of the solution volume ($V^{/}$) (up to the vertical line).

Fig. 3 shows the results of tungsten extraction into solution, depending on the stage and duration of leaching in % (rel.).

As seen from the data in **Fig. 2**, during ore leaching, tungsten and molybdenum, unlike other components, are extracted into solution only with oxalic acid.

Calcium extraction slows down somewhat due to the precipitation of its sulfate, but an increase in magnesium extraction compensates.

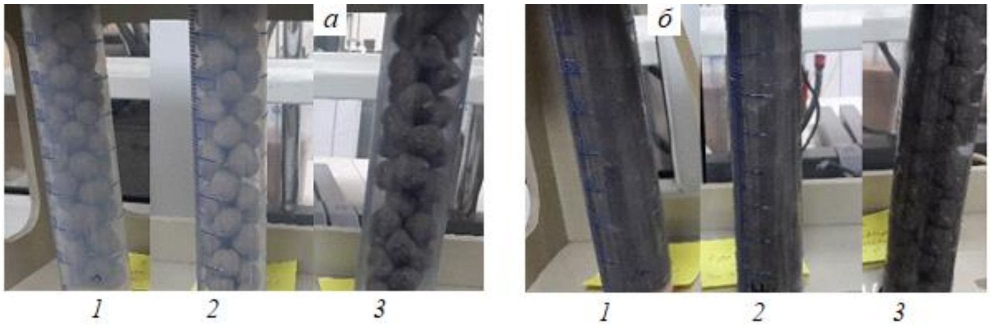
The sample was tested in 96 days. When acidifying the ore with a hydrochloric acid solution, its consumption was 44.61 kg/t, and when leaching with oxalic acid, it was 43.1 kg/t. Tungsten extraction was 73.22%, and the cake yield was 82.5%.

Table 5 shows the chemical composition of the cake (upper and lower part of the column) after leaching.

Productive leaching solutions were processed according to the sorption scheme (sorption/desorption). The daily volume of the productive leaching solution from the column was sent to sorption columns filled with ion exchange Seplite MA 900 OH resin. The solutions obtained after tungsten sorption isolation were reinforced to the required concentration with oxalic acid and sent into circulation for leaching.

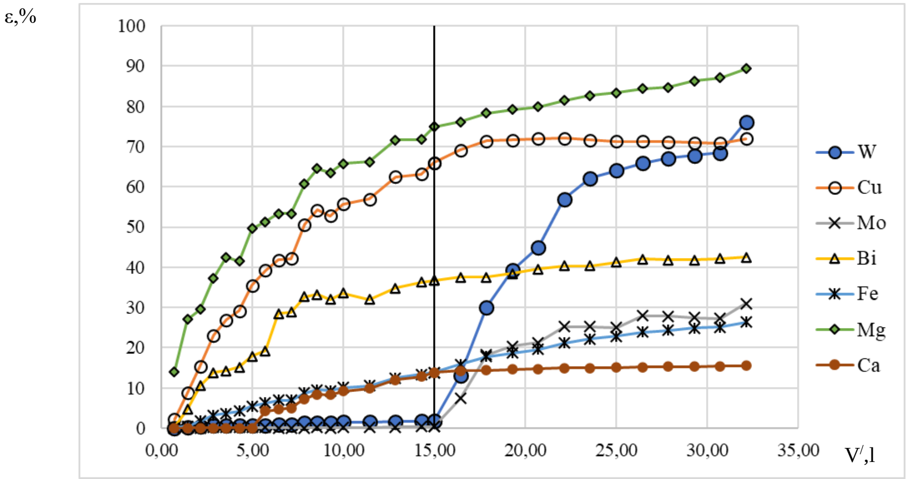
The study of the chemical composition of the cake after the ore is percolated and leached shows a slight decrease in the content of tungsten and copper in the lower part of the column. A decrease in the copper content in the bottom layer of the cake from 0.116 to 0.107% indicates a positive leaching process.

In the process of percolation leaching, it was found that a stack of tungsten ore pelletized under selected conditions has favorable characteristics for processing by heap leaching.



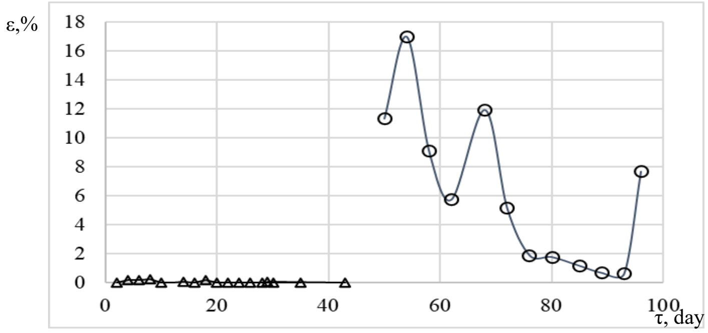
a – initial pellets, *b* – pellets after percolation;
1 – without binder (water); 2 – cement; 3 – Alcotac CB6

Fig. 1 Filtration properties of granulated ore using various binding materials



from 0 to 15 – acidification (HCl), from 15 to 32 V/ – leaching ($H_2C_2O_4$)

Fig. 2 Dependence of the extraction of scheelite ore components on the stage and duration of leaching, expressed in the specific daily consumption of the solution volume



Δ - acidification; o– leaching of tungsten

Fig. 3 Results on the extraction (rel.) of tungsten by acidification of the sample with hydrochloric acid followed by leaching with oxalic acid

Table 5 Chemical composition of cake after leaching, %

Cake weight, kg	W	Mo, g/t	Cu	Bi, g/t	Mn	Ca	Mg	K	Al	Fe
5.62 (top of the column)	0.073	50.96	0.116	597.5	0.563	16.23	0.149	0.578	3.312	6.591
5.93 (column bottom)	0.088	77.74	0.107	443.2	0.57	16.41	0.17	0.589	3.475	6.408

CONCLUSION

The behavior of granules in the percolation leaching method indicates the effectiveness of pre-pelletizing a sample of scheelite ore with the Alcotac® CB6 reagent. During the leaching process, the pelletized material has sufficient strength, porosity, and permeability and provides access to leaching solutions on the surface of valuable metals.

Percolation leaching of the pelletized ore was carried out with acidification with hydrochloric acid and further leaching with oxalic acid. Rock minerals leached during acidification, and scheelite was relatively stable in dilute hydrochloric acid solutions. This achieves selective leaching of rock minerals and reduces oxalic acid consumption during subsequent tungsten leaching.

The analysis of the research conducted shows:

The conditions for selectively extracting the ore's rock components have been selected. After acidification, tungsten and molybdenum pass into an oxalic acid solution with minimal losses.

Tungsten enters the solution sequentially after almost completely leaching bismuth and calcium. These extremely favourable circumstances greatly facilitate the selective isolation of commercial tungsten and bismuth products.

The main components were extracted at %: W—73.22; Cu—76.56; Mo—53.13; Bi—50.0.

Cake yield was 82.5 %;

The specific consumption of hydrochloric and oxalic acids amounted to 44.61 kg/t and 43.1 kg/t, respectively.

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