Valorisation of Mining Waste for Gold Recovery

Stefano UBALDINI[1)], Daniela GUGLIETTA[2)], Giorgio UBALDINI[3)], Ida DE MICHELIS[4)]

[1)] Istituto di Geologia Ambientale e Geoingegneria, CNR, Area della Ricerca di Roma RM 1 – Montelibretti, Via Salaria Km 29,300 - 00015 Monterotondo Stazione (ROMA) – Italy; email: stefano.ubaldini@igag.cnr.it
[2)] Istituto di Geologia Ambientale e Geoingegneria, CNR, Area della Ricerca di Roma RM 1 – Montelibretti, Via Salaria Km 29,300 - 00015 Monterotondo Stazione (ROMA) – Italy; email: daniela.guglietta@igag.cnr.it
[3)] Università degli Studi di Roma "La Sapienza", p.le A. Moro 5, 00185 Roma, Italy; giomagica@hotmail.it
[4)] Dipartimento di Chimica, Ingegneria Chimica e Materiali, Universita' degli Studi di L'Aquila, 67040 Monteluco di Roio, L'Aquila, Italy; email: ida.demichelis@ing.univaq.it

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Abstract

Purpose: the metals and industrial minerals contained in the tailings produced by mining and quarrying activities, are cause of environmental damage.
The objective of this experimental work is the application of innovative technologies for the treatment and exploitation of mining tailings in Romania, in order to recover materials high grade raw to be placed on the market, reducing the volume of the wastes.
Further objective, is to optimize some parameters relating to the dissolution of gold and the subsequent recovery from purified solutions, determining beforehand the technical feasibility of the scheme of process developed on a laboratory scale.
Methods: the study is focused on hydrometallurgical process for the recovery of gold. The innovative treatment chosen is the thiosulphate process that, compared to conventional cyanide, has several advantages, first of all the most eco-friendly and non- toxic to humans.
The conventional process shows operating limits in the case of auriferous minerals refractory materials, such as Romanian wastes object of the study. Another fundamental characteristic of the ammoniacal thiosulphate solutions, is the best selectivity towards gold, not attacking the majority of the gangue mineral constituents.
Results: the dissolution rates of gold reached a final value of 70% Au – working at room temperature – with recoveries of the global process of about 65%, in line with the conventional process.
Main conclusions: these results are very encouraging, considering that this is an innovative process, applied to a low content gold ore. The optimization of parameters and operating conditions, and the industrial treatment, continuous and scale greater would certainly permit to reach the best results in terms of process yields and energetic and reagents consumption.

Keywords: mining waste, hydrometallurgical processes, thiosulphate leaching, electrowinning, gold

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Introduction

Mining is one of the most industrial activities that may damage, pollute and alter the territory as it involves the creation of morphologies unstable prone to landslides and collapses, but especially exposure to the environment exogenous vast areas of mineralized rocks and byproducts of treatment, which produce significant changes in the chemical environment (Piga et al., 1995; De Michelis et al., 2013).

The large volumes of wastes produced by mining occupy huge areas; these accumulations come to substantially change the original landscape.

The deposits of mining tailings should not be treated as wastes, inert and treated as such, but as neo-mine be enhanced. Only a short-sighted view it merely neutralize the harmful and toxic waste stored and implement procedures and interventions leading to the restoration of the existing environmental negative conditions. The enhancement of the tailings, in addition to the sale of commodity grade high, allows to implement a complete recovery of the environmental conditions, effective and sustainable because the activity of recycling produces the complete removal of allowing back to the morphological structure of pre-exploitation.

Romania, like many other nations affected by a long history of mining, it is now grappling with environmental and social issues related to tailings produced by the mining activities. On the Romanian territory have been inventoried 300 tailings deposits, all of considerable proportions from the cultivation of different minerals, to be submitted to procedures for the safety (Geological Romanian Institute, 2011; ANMR, 2011). Waste materials come from mining activities that have reached the end of the production cycle to depletion of the reservoir or that were stopped for environmental reasons not related to treatment systems environmentally friendly (such as was put in place at the time of the Convention for Romania's entry into the EU). In Romania, the expertise of these landfills, until their reintegration in the environment in safety conditions, is the Romanian National Agency for Mineral Resources (ANMR). The mining sites considered in this study have been Bălan,

Fig. 1. Tailings of mining site in Romania
Rys. 1. Składowisko odpadów w Rumunii

Tab. 1. Heavy fractions separated from the flow table
Tab. 1. Frakcje ciężkie po wzbogaceniu na stole koncentracyjnym

Fraction	%
Heavy Brad Ribita	8.00
Heavy Brad Criscior	12.00

Tab. 2. Gold recovery obtained with a solution of ammoniacal leaching having the following composition: 2 M $S_2O_3^{2-}$, 0.1 M $CuSO_4$ and 0.1 M NH3
Tab. 2. Uzysk złota w procesie ługowania w roztworze 2 M $S_2O_3^{2-}$, 0.1 M $CuSO_4$ and 0.1 M NH3

Time (h)	**mg Au**	**Au recovery (%)**
15 (min)	0.23	16.23
30 (min)	0.23	16.59
1	0.23	16.59
90 (min)	0.24	17.31
2	0.26	18.76
3	0.36	25.61
4	0.47	33.55
Washing	0.06	4.55

Deva deposit 1, Deva deposit 2, Brad Ribita and Brad Criscior.

Mine Bălan, for example, which ceased its operations in 2006, under the Convention for the entry of Romania into the EU, is a tabular deposit tectonised in metamorphic rocks. The main mineral is chalcopyrite. The reserves remaining amounted to 500,000 tonnes at 0.8% copper content. These reserves are substantially unavailable. The cost to re-mining and to make modifications to the enrichment of mineral are too high compared to the value of resources and the costs of extraction (Ubaldini et al., 1995; Ubaldini et al., 1996; ANMR, 2011; Geological Romanian Institute, 2011).

The deposits of tailings, left on the land by mining during his exercise, containing 24.5 million tonnes of slag resulting from the treatment of ore copper, or more than 10 million cubic meters of materials that constitute a refusal toxic and harmful. These deposits are located at a distance of few kilometers from the mining center, in the neighborhood of the town of Bălan (ANMR, 2011; Geological Romanian Institute, 2011).

The raw materials, high-grade, removable from the tailings Bălan, like those of the other sites mentioned are:

- industrial minerals, such as quartz, pyrite and chlorite, recoverable through minerallurgical processes;
- base metals, including copper and zinc, and precious metals such as gold and silver, extracted with hydrometallurgical processes.

The aim of this work is to apply innovative technologies for the treatment and exploitation of mining tailings Romanians, in order to recover high grade raw materials to be placed on the market. Specifically, the study focuses on the hydrometallurgical process for the recovery gold. The innovative treatment chosen is the thiosulfate process, that present several advantages in comparison with the conventional cyanide and is non-toxic to humans. The thiosulphate process shows some characteristics that distinguish it from process to conventional cyanide for gold recovery, in fact, it's environmental impact is lower than the cyanidation (Abbruzzese et al., 1995; Aylmore et al., 2001; Grosse et al., 2003; Rath et al., 2003; Ubaldini et al., 2003a; Bas et al., 2011).

The main advantages of this innovative treatment for gold recovery, can be summarized in the following points:

1. solubilization of gold with an appreciable rate of dissolution;

2. ammonia solutions not attack most of the gangue mineral constituents;

Tab. 3. Gold recovery obtained with a solution of ammoniacal leaching having the following composition: 2 M $S_2O_3^{2-}$, 0.1 M $CuSO_4$ and 4 M NH_3 (with addition of H_2SO_4 to maintain the pH constant at 10.58)

Tab. 3. Uzysk złota w procesie ługowania w roztworze : 2 M $S_2O_3^{2-}$, 0.1 M $CuSO_4$ oraz 4 M NH3 (dodatek H_2SO_4 do uzyskania pH =10.58)

Time (h)	**mg Au**	**Au recovery (%)**
15 (min)	0.91	73.38
30 (min)	0.90	72.31
1	0.86	69.63
90 (min)	0.82	66.95
2	0.74	60.52
3	0.73	59.99
4	0.74	60.52
Washing	0.033	2.33

Tab. 4. Kinetics of Au adsorption at different concentrations of carbon in solution

Tab. 4. Kinetyka adsorpcji złota dla różnych koncentracji węgla

Time (min)	**5 g/L carbon**	**10 g/L carbon**	**15 g/L carbon**
	% Au		
15	53.59	50.00	82.05
30	61.54	70.52	98.72
45	76.65	87.18	98.72
60	85.90	98.72	98.72

Tab. 5. Kinetics of Au desorption at different concentrations of carbon in solution

Tab. 5. Kinetyka desorpcji złota dla różnych koncentracji złota

Time (h)	mg Au	% Au yield
1	0.20	18.00
2	0.34	30.00
4	0.46	41.00
6	0.88	79.00
Washing	0.30	20.00

Tab. 6. Best process parameters of gold desorption from the activated carbon

Tab. 6. Optymalne parametry desorpcji złota z węgla aktywnego

Parameters	**Value**
Temperature	80°C
Speed of agitation	300 rpm
Time of extraction	6 hours
Mass ratio of coal and stripping solution	75 g/L

Tab. 7. Experimental electrowinning results during gold deposition

Tab. 7. Wyniki elektrolizy

Time (min)	**cathodic voltage (V)**	**cell voltage (V)**	**current intensity (mA)**	**electric charge (C)**
15	1.4	2.25	100	161
30	1.4	2.51	210	254
45	1.4	2.47	210	352
60	1.4	2.52	210	448
75	1.4	2.51	210	564

Tab. 8. Best process parameters of the phase of gold electrodeposition

Tab. 8. Optymalne warunki elektrolizy

Parameters	**Value**
Temperature	40°C
Time of electrolysis	75 min
Voltage at the cathode	- 1.4 V
Voltage of the cell	2.50 V

3. production of leached solutions, practically free from metallic elements that hinder the subsequent recovery operations;

4. ability to recirculate the leaching ammonia solutions;

5. non-toxicity of the reagents used in the leaching process;

6. possibility of recovery the dissolved gold by known techniques, such as carbon adsorption and electrodeposition.

Final purpose of this work is to develop a process scheme on the basis of results obtained in laboratory scale, in order to provide a preliminary study of the technical feasibility of process. The following photograph shows an example of tailings of mining site in Romania.

Materials and Methods

Sampling and characterization

The mining sites under study were: Bălan, Deva deposit 1, Deva deposit 2, Brad Ribita and Brad Criscior.

For each mining site were prepared homogeneous and representative samples for subsequent characterizations. To this end, after being dried in oven at 80°C for one day and sieved with a sieve of 4 mm, the non-reusable fractions were subjected to a first sampling using rotary splitter. For each site, eight samples have been obtained.

For the characterization of the particle size, the prepared samples were quartered with the manual sampler and submitted to wet sieving using sieves of following size: 0.5 mm – 0.351mm – 0.250 mm – 0.125 mm.

The granulometric fractions obtained were filtered, dried in a laboratory oven at 80 ° C for one day and weighed. On the basis of the weights obtained, distribution curves were constructed (data not showed here). For each mineral deposit, one of the eight samples prepared was submitted to gravimetric separation by flow table. The four fractions obtained (light, intermediate, mixed and heavy) were filtered, dried in a stove at 80° C for a day and weighed.

With the main aim to obtain homogeneous and representative samples, a rotary splitter RETSCH has been utilized, to divide the sample into the various fractions whose composition corresponds exactly to that of the initial sample, because only a representative sample of the initial rate can provide significant analytical results.

This procedure ensures a high degree of accuracy and reproducibility. It is used in combination with the vibrating feeder RETSCH DR 100, utilized for the assay homogeneous and uniform for the conveying of the material, it has thus a process automatic allocation, without interruptions and loss of material. The speed was monitored and kept constant.

A planetary ball mill agate mod. FRITSCH pulverisette, was used for the fine grinding of the samples. The jars and grinding balls made of agate were useful to avoid metallic contamination.

The mineralogical characterization was carried out by the technique of X-ray diffraction, with the X-ray diffractometer Bruker, mod. D8 Advance.

The analytical determination of metals and gold content of the fractions obtained from the table, was carried out by optical plasma spectrometer (ICP–AES) Perkin Elmer, mod. 400 with data station, after chemical dissolution.

Homogeneous and representative samples of approximately 10 g, submitted to chemical attack, were prepared by rotary and manual splitters and subsequently milled in a planetary mill with agate jars – equipped with agate balls – to avoid any metal pollution of the samples, up to obtaining products with a particle size less than 80 μm, appropriate to the subsequent leaching tests (Abbruzzese et al., 1995). On the basis of the results obtained from the chemical analysis, relatively to the content of gold minerals examined, the sample on which to implement the process for gold recovery has been chosen.

The experimental work was carried out on the heavy fraction of a mixed sample, whit an average gold content was of about 3 g/t.

Physical process

The samples from mining sites Brad Ribita and Brad Criscior were sieved with a sieve of 0.5 mm, the fractions <0.5 mm were subjected to gravimetric separation by flow table. The table was set with the aim to obtain a heavy fraction richer in those heavy that they could instead collected in the mixed fraction. The heavy fractions recovered were filtered and dried in a stove at 80°C for one day.

Grinding

The heavy fractions, obtained by gravimetric separation, were subjected to comminution (<80 μm), through the mill bar, to make them suitable to determination of the content of gold and for the subsequent tests leaching. The grain sizes obtained were analyzed with the laser granulometer SYMPATEC.

At the end of the comminution, the drum was emptied and the slurry after being filtered was dried in a stove at 80°C for one day. The heavy fractions, after comminution, were mixed homogeneously by rotary splitter to prepare samples of the mix Brad Ribita - Brad Criscior, and submitted to leaching tests. The gold content of the mixture of minerals was determined after chemical dissolution, with an Atomic Absorption Spectrometer (AAS Perkin Elmer mod. 460).

Chemical process

The study of the thiosulphate process, was conducted in Pyrex glass mechanical stirred reactors, of capacity of 2000 mL.

Leaching experiments were carried out – after comminution (<80 μm) – to study the influence of the concentration of ammonia and the concentration of thiosulfate on gold dissolution, using reagent of analytical degrees and distilled water.

The leaching solutions consist of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$), used as active leaching agent, ammonia (NH_4OH 30%) – for the control of pH – and copper (II) sulphate ($CuSO_4 \cdot 5H_2O$) – which acts as an oxidant of gold.

The tests were carried out at atmospheric pressure and room temperature, while the speed of mechanical agitation was kept constant at 400 rev/min. The time of attack was of 4 hours. Samples of ore from attack, 500 g each, had a particle size of less than 80 μm, a pH of 6 and a redox potential +0.1 V. At set intervals, small volumes of leaching solution were taken from the reactor, for the kinetic study of the gold dissolution (Ubaldini et al., 1998; Ubaldini et al., 2003b).

The pH and the oxidation-reduction potential of the slurry, were measured using a combined glass electrode and a platinum combination electrode, respectively, both connected to a digital pH meter.

At the end of each test, the reactor was emptied, while the filtration of the slurry was realized through pressure filters. The sterile was submitted to washing with distilled water and ammonia.

After leaching, the gold was purified by selective adsorption onto granular activated coconut carbon. The influence of the carbon concentration has been studied.

Also this stage of the process was conducted in Pyrex glass reactors of capacity of 2000 mL under mechanical stirring (400 rev/min), at room temperature, for a total contact time of 1 hour (Jeffrey et al., 2010; Ubaldini et al., 2003; Ubaldini et al., 2006).

The tests were carried out by placing in contact leached solution with the coal coconut 500 mL. The influence of the contact time by performing withdrawals at set intervals was investigated. The concentration of activated carbon was varied from 5 g/L to 15 g/L. After each experiment, carbon was recovered from the solution and left air dry. Representative samples of carbon were collected and submitted to quantitative chemical analysis.

The adsorbed gold has then recovered in the desorption phase, by elution with an aqueous solution of ethyl alcohol (absolute ethanol C_2H_5OH) and subsequent cathodic electrodeposition in electrolytic cell, for final recovery of metallic purified gold.

The desorption of gold from carbon was carried out through re-extraction with water-alcohol solution. The stripping test were conducted in a glass reactor Pyrex, of capacity of 250 ml. The reactor was fitted with three necks: in the first was placed a reflux condenser for the removal of vapors, in the second was made to pass the probe, connected to the plate shaking-heating, for stabilize the temperature and into the last was inserted the thermometer for temperature control. Tests have been conducted varying the temperature from 40 to 85°C. The stripping solution used was a hydro-alcoholic solution containing ethanol.

Samples of the solution were taken, at predetermined intervals, for the study of the kinetics extraction during the stripping phase.

The final recovery of metallic gold purified from the water-alcohol solution, was carried out by electrochemical process (Ubaldini et al., 1998; Ubaldini et al., 2000; Ubaldini et al., 2003b).

Tests were conducted in an electrolytic cell of capacity of 200 ml in glass Pyrex with shirt, connected to a thermostat Julabo, mod. 5B (control from -20 to +100° C). The cell was fitted with a saturated calomel reference electrode, an electrode of work (cathode) constituted by a net of platinum wire, having a surface area of 100 cm^2, and a counter-electrode (anode) consisting of a spiral platinum.

The cell was connected to a potentiostat – galvanostat AMEL, model 555 B. The current flowing through the cell was converted into a numerical value by an integrator AMEL, model 721. The potential difference between the cathode and the anode was measured with an electrometer differential AMEL, model 631.

The cell was placed on a plate shaking, that thanks to the presence of a stir bar magnetic allows stirring of the solution. The electrolysis tests were conducted using 200 mL of stripping solution.

Results and Discussion

Physical process

The homogeneous and representative samples object of the study, after the step of screening that has allowed to retain particles with a diameter greater than 0.5 mm, were submitted to gravimetric separation by flow table. The goal of the physical process was to concentrate pyrite, which is associated with the gold present in the tailings in heavy fraction, while quartz in that light.

The heavy fraction in no deposit reaches 10%, but we must consider that most of the pyrite could be concentrated on the mixed fraction. In all sites light fraction exceeds 40%, while over 61% in Balan.

The chemical analysis, has allowed to determine the gold content of the mixed fractions and heavy minerals studied. The mining sites of greatest interest from the point of view of the gold content are: Brad Criscior and Brad Ribita. These latter have been chosen for the study of the process of recovery of the precious metal.

The X-ray diffraction performed on the heavy fractions and mixed by Brad Criscior and Brad Ribita has allowed to determine the mineralogical composition by the interpretation of the X-ray diffraction spectra.

The pyrite (FeS_2) concentration reaches the 20.4% of the heavy fraction of Brad Ribita while most part (48.3%) is constituted by quartz (SiO_2). FeS_2 in the mixed fraction of Brad Ribita is 6.3%, while was up 10% on both the fractions of Brad Criscior.

The tailings from Brad Criscior and Brad Ribita Criscior, chosen for their higher gold content, after being suitably sampled, have been submitted to physical separation by flow table. The purpose of this gravimetric separation was to concentrate FeS_2, and then the gold associated with it, in the heavy fraction. The results obtained are shown in the following Table 1.

The heavy fractions recovered represent about 10% of the mineral feeding table.

Chemical process

Gold Leaching

The experimental work was carried out on the heavy fraction, mix between Brad Criscior and Brad Ribita, with an average gold content of 3 g/t. Gold was recovered by leaching with sodium thiosulfate.

After preliminary experiment – carried out starting from literature information (Abbruzzese et al., 1995; Rath et al., 2003), leaching permitted to reach gold recovery of 38.10% Au, when consider also those of the sterile washing, carried out with distilled water and ammonia (Table 2).

The complete study permits to study the influence and the interactions between $S_2O_3^{2-}$, $CuSO_4$ and NH_3 concentration (data not shown here).

The table 3 shows higher gold recoveries (about 65% Au after 15 minutes), but it was observed that the kinetics of extraction decreases by approximately 20% (Abbruzzese et al., 1995).

The goals of the further work will be to optimize the leaching process conditions, with the aim to increase gold extraction yields, and to reduce the reagents consumption, decreasing the concentration of thiosulphate used.

Gold Adsorption

The leaching solution containing gold in the form of soluble complex, is placed in contact with the activated carbon to selectively separate the gold by adsorption. The experimental conditions described in Materials and Methods chapter, have been applied to study the influence of the mass ratio of carbon and solution of the gold recovery process. The purification of the solutions leached has allowed to obtain recoveries shown in the following Table 4.

The results show an almost complete recovery of gold present in solution.

From the trend it is clear that the increase of the concentration of carbon in solution promotes the recovery. In particular, after 1 hour, with a concentration of carbon of 5 g/L is adsorbed about 86% Au, but the recoveries reach 99% Au when the concentration increases to 10 g/L. Also it is possible to observe that, with a concentration of 15 g/L after only 30 min, 99% Au adsorption was achieved.

Gold desorption

The purpose of the test was to re-extract the gold adsorbed and concentrate it.

The duration of the kinetics of recovery was fixed at 6 hours, by making withdrawals at set intervals.

From the experimental results, shown in Table 5, it can be observed that the final recoveries have been of 79.00% Au: preliminary study on the type of alcohol use may improve the efficiency of the stripping process.

The parameters – that have permitted to achieve 99.00 % Au recovery – including washing – after 6 hours of that loaded onto activated carbon – are shown in Table 6.

Electrowinning

The last step of the process is the electrowinning, by gold deposition in the electrolytic cell. The goal of this step was the recovery of gold from the stripping solution, for cathodic deposition of the metal. Table 7 shows the experimental results obtained during the process.

Table 8 shows the optimized process parameters of the phase of gold electrodeposition.

The kinetics of electrodeposition in cell of laboratory is quick and final gold recovery very high (98% Au). The concentration of gold in the sterile solution is nothing (below the detectability of the instrument) already after the first 30 min. A dark deposit, is uniformly distributed on the surface of the cathode.

From the reported data it can be observed that the intensity of the measured current is around to around 210 mA, which corresponds to a current density of about 2.1 mA/cm², knowing that the cathode surface is 100 cm². As can be seen from the experimental results, in the cell are passed, after 30 min, 254 Coulombs, the efficiency of the current is so low (5%) due to parasitic reactions. Consumption energy is of about 20 kWh/kg of gold deposited. The faradic efficiency and energy consumption are relatively low.

Conclusions

The preliminary study of the parameters and operating conditions for the various phases of the process thiosulphate shows, for the sample of mining wastes from Brad Ribita and Brad Criscior, the technical viability of the process; in fact, the experimental results obtained have confirmed the good kinetics of dissolution of gold in aqueous solution of thiosulfate, which can be used without special precautions and restrictions.

The dissolution rates of gold reached a final value of 70% Au working at room temperature, with recover-

ies global process (leaching – adsorption - desorption – electrodeposition) of about 65%.

These results are very encouraging, considering that it is an innovative process, applied to a gold ore with a low content. The optimization of parameters and operating conditions, and the industrial treatment, continuous and scale greater would certainly permit to reach the best results in terms of process yields and energetic and reagents consumption.

Considering both the alarming estimates of reserves of gold, that the inability of the gold production to quickly react to the prospect of a change in prices and to changes in demand, it is providential gold recovery from tailings mining. The high price of the precious metal now allows, as shown preliminary economic analysis conducted, the feasibility of these processes despite the low levels of gold, the high ratio between sterile and mineral and the high costs of extraction.

The enhancement of the tailings, in addition to the sale of raw materials of high grade, allows to implement a complete recovery of the environmental conditions, effective and sustainable. It is thus able to ensure over time the use of two essential resources and of primary importance: the environment as a whole from one side and the raw materials mining the other. In addition to the economic and environmental aspects, we must consider the social benefits that such projects can be made, providing many jobs and contributing thus to the development of areas, for various reasons, repressed.

In the next step of investigation, complete process analysis, including detailed description of the process scheme together economic analysis will be outlined.

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Odzysk złota z odpadów górniczych

Cel: metale i minerały zawarte w odpadach powstających w wyniku działalności górniczej i wydobywczej są przyczyną szkód w środowisku. Celem prac eksperymentalnych jest zastosowanie innowacyjnych technologii do obróbki i eksploatacji odpadów wydobywczych w Rumunii, w celu odzyskania wysokiej jakości surowców, które zostaną wprowadzone do obrotu, zmniejszając ilość deponowanych odpadów. Dalszym celem jest optymalizacja niektórych parametrów związanych z rozpuszczaniem złota i późniejsze odzyskiwanie z oczyszczonych roztworów, z określeniem technicznej wykonalności schematu procesu opracowanego w skali laboratoryjnej.

Metoda: badanie koncentruje się na procesie hydrometalurgicznym odzyskiwania złota. Innowacyjnym sposobem jest zastosowanie do ługowania tiosiarczanu, który w porównaniu z tradycyjnym cyjankiem ma wiele zalet, przede wszystkim jest bardziej przyjazny dla środowiska i nietoksyczny dla ludzi. Konwencjonalny proces daje ograniczone wyniki dla badanych odpadów złotonośnych. Podstawową zaletą amoniakalnych roztworów tiosiarczanu jest lepsza selektywność w stosunku do złota, a nie ługowanie wszystkich składników mineralnych skały płonnej.

Wyniki: wskaźniki rozpuszczania złota osiągnęły wartość 70% Au - w temperaturze pokojowej - w porównaniu do odzysku z procesu konwencjonalnego około 65%.

Główne wnioski: wyniki są bardzo zachęcające, proces jest innowacyjny, zastosowany do rudy o niskiej zawartości złota. Optymalizacja parametrów i warunków pracy oraz wdrożenie w skali przemysłowej pozwoli osiągnąć lepsze wyniki pod względem wydajności procesu oraz zużycia energii i odczynników.

Słowa kluczowe: odpady górnicze, proces hydrometalurgiczny, ługowanie tiosiarczanem, elektroliza, złoto