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APPLICATION OF INNOVATIVE PROCESSES FOR GOLD RECOVERY FROM ROMANIAN MINING WASTES

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Abstract. The application of a new hydrometallurgical process for gold extraction by thiosulphate leaching from Romanian mining wastes, coming from Balan and Deva deposits, was studied. Another objective of this work was to develop an integrated flow-sheet including the recycling of process solutions and of the coconut activated carbon used for gold purification. There was obtained 85% of Au extraction after leaching; moreover, an integrated flow-sheet, including recycling of process solution and carbon, was outlined, based on results obtained at a laboratory scale, using a schematic chemical circuit of treatment. Global recovery of the process (leaching-adsorption-desorption-electrodeposition) of about 75-80% of Au was achieved. The developed integrated flow-sheet, allows to recycle the reagents during the process, with a loss of only 5-10%, in particular thiosulphate and alcohol, for each complete circuit of treatment.

Keywords: mining waste, gold, thiosulphate leaching, activated carbon, adsorption, desorption.

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Introduction

Raw materials (RMs) are essential for the industrial, technological and social development of European and non-European countries. The supply of certain RMs is becoming a growing concern in the EU and the rest of the world. European countries depend almost completely on the external countries for supplies of RMs, mainly the USA, Brazil and China [1,2]. The progressive depletion of primary deposits makes indispensable the exploitation of secondary resources such as mining and industrial waste, and waste electronic electrical equipment (WEEE), due to their high content of valuable and strategic metals. Extraction activities, recycling and reuse of wastes, must be pursued with appropriate management strategies, in order to reduce the environmental impacts [1,2].

Metals - such as Au, Ag, Cu, Zn, Mn, Ni and critical raw materials (CRMs) – such as Pt, In, Co, Vn, Mg, Sb, Nb, rare earth metals are indispensable for many industrial activities, in particular for the application of new emerging technologies and for the protection of environment [2,3]. It is therefore important to develop environmentally friendly processes, to obtain RMs and CRMs of economic interest through the exploitation of secondary RMs [2,4,5].

The application of innovative bio-hydro-electro-metallurgical integrated circuits allows advancement that consists in low environmental impact and energy consumption, together with a high degree of purity of the obtained valuable metals, if compared with the conventional processes; these sustainable technologies have been applied to the treatment and exploitation of mining tailings [6-8]. Gold extraction by thiosulphate leaching represents an example of the application of these new processes: in the presence of ammonia and copper(II) ions, the oxidation reaction of metallic gold as the ionic form in ammonium thiosulphate solution, where cupric tetraamine complex ion is the oxidant, can be represented by the following Eq.(1) [9]:

$$Au + 5S_2O_3^{2-} + Cu(NH_3)_4^{2+} \rightarrow$$

$$\rightarrow Au(S_2O_3)_2^{3-} + 4NH_3 + Cu(S_2O_3)_3^{5-} \quad (1)$$

The application of the conventional process (cyanidation) to the treatment of auriferous refractory minerals and mining wastes, such as the Romanian samples - object of the present study, does not allow a sustainable Au recovery [10,11]. Another fundamental characteristic of the ammoniacal thiosulphate solutions is the best selectivity towards Au, without attacking the majority of the gangue mineral constituents [12-15]. The applied innovative treatment has technical advantages over conventional cyanide, is non-toxic to humans and has a lower environmental impact than cyanidation [12,16-21]. In addition, a series of social benefits can be generated through the large-scale application of these new technologies, contributing thus to the development of repressed areas, improving the competitiveness and creating added value and new jobs in the field of RMs processing, refining, equipment manufacturing and downstream industries [1,2].

The main aim of this experimental study is the application of a new hydrometallurgical process to valorise Romanian mining wastes from Balan and Deva deposits through Au extraction with thiosulphate leaching. Based on obtained results at laboratory scale, utilising a schematic chemical circuit of treatment, the final purpose of this work was set to achieve an integrated flow-sheet, including the recycling of process solution and coconut activated carbon used for Au purification.

Experimental

Starting from previous experiments conducted by Brad Ribita and Brad Criscior samples [18], a complete integrated circuit was defined, aimed to minimize the consumption of the reagents. In the present study, samples from Balan and Deva deposits were used, which have different mineralogical characteristics in comparison with those collected from Brad Ribita and Brad Criscior and different gold content. The coconut activated carbon used in the purification phase has been reused in several treatment cycles. The consumption of the reagents was calculated aiming to recirculate the process solutions several times.

Samples preparation

The experimental work was carried out on samples of Romanian mining wastes coming from Balan and Deva deposits with a Au content between 1 and 4.5 g/t. The sampling campaigns took place in 2017 and 2018. Once transferred to the laboratory, the samples were dried in the oven for 2 days at 60°C [8].

In the subsequent steps, homogeneous and representative samples were prepared for characterization and leaching experiments, using a RETSCH rotary splitter; therefore, these were subjected to comminution using a bar mill model Sepor. Then, the samples were ground using a planetary ball mill agate model FRITSCH pulverisette, until reaching particle size of 80 μm (80%) [8]. The used jars and grinding balls were made of agate, to prevent sample contamination [1,8]. At the end of the comminution, the samples were dried in the oven at 80°C.

Samples for leaching tests were submitted to gravimetric separation by flow table with the goal to concentrate pyrite and the gold associated with it in the heavy fraction [8].

Samples characterization

The heavy fraction reached about 12% in weight and contained mineralogical species detected by X-ray diffraction [8], including:

- quartz (SiO_2) (45.3%);
- pyrite (FeS_2) (23.8%);
- muscovite $(K,Ba,Na)_{0.75}(Al,Mg,Cr,V)_2(Si,Al,V)_4O_{10}(OH,O)_2$ (14.9%);
- albite ($Na_{0.75}Ca_{0.25}$)($Al_{1.26}Si_{2.74}O_8$) (8.2%);
- chamosite $(Mg_{5.036}Fe_{4.964})Al_{2.724}(Si_{5.70}Al_{2.30}O_{20})(OH)_{16}$ (5.2%);
- calcite—$CaCO_3$ (2.4%);
- chalcopyrite—$CuFeS_2$ (0.2%).

The main elements determined in the samples using a Perkin Elmer 400 optical plasma spectrometer (ICP-AES) were: Si (25.0%), S (13.0%), Fe (12.6%), Al (3.2%), V (2.9%), Ca (1.1%), Ba (1.1%), Cr (0.9%), Mg (0.8%), Na (0.7%), K (0.4%), Cu (0.2%).

The Au content was determined using an atomic absorption spectrometer (AAS Perkin Elmer 460), after the chemical dissolution of homogenized representative samples of approximately 15 g, of the particle size was less than 80 μm (80%) [8,22]. With the aim to study the process of Au recovery, were considered and chosen samples with the greatest gold content (4.5 g/t Au).

Schematic chemical circuit of treatment

Selected samples of mining wastes have been leached by thiosulphate solutions. The leached Au was purified through adsorption onto activated carbon; gold stripping from the carbon was carried out by hydroalcoholic solutions, while the final recovery of the gold from the solutions was carried out by means of electrowinning. The schematic chemical circuit of treatment on laboratory scale is shown in Figure 1.

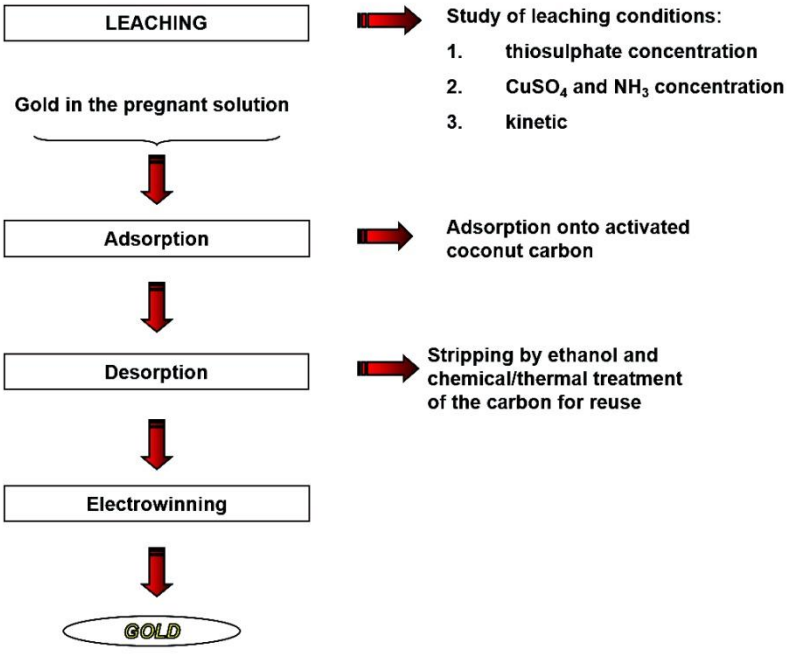

Figure 1. Schematic chemical circuit of treatment of gold mining wastes on laboratory scale.

Thiosulphate leaching

The experimental work was carried out on the heavy fraction of mixed samples with an average gold content of 4.5 g/t. Gold has been recovered by chemical leaching combined with an ammoniacal solution, where the sodium thiosulphate was the active leaching agent.

Thiosulphate leaching was conducted in mechanical Pyrex glass stirred reactors, with the capacity of 3000 mL. The composition of the leaching solutions was [9,23,24]:

- sodium thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$), used as leaching agent;
- ammonia (NH_4OH 30%), utilised to adjust the pH value;
- copper(II) sulphate ($CuSO_4 \cdot 5H_2O$), used as a gold oxidizing agent.

The concentration of the used reagents (from 1 to 2 M $Na_2S_2O_3$, from 0.06 to 0.1 M $CuSO_4$ and from 0.1 to 4 M NH_4OH), of analytical grade, was determined according to previous investigations [8,12,18]. Distilled water has been used throughout this study.

Experiments were conducted under mechanical stirring (350 rev/min) at a temperature of 25°C and atmospheric pressure for a total leaching time of 5 h. The weight of the homogeneous and representative samples was of 1000 g, the particle size 80 µm (80%), pH between 10.0 and 10.5, redox potential +0.1 V. At set time intervals, small volumes (5 mL) of slurry samples were taken from the reactor and filtered by pressure filters to study the kinetics of Au dissolution. The liquids were analysed directly and the solid residue was washed with distilled water and ammonia and submitted to chemical dissolution for Au analysis [12,25].

A combined glass electrode and a platinum combination electrode, connected to a digital pH meter, were used to measure the pH and the redox potential.

Gold adsorption onto activated carbon

The Au content has been recovered from the slurry and purified by adsorption onto granular coconut activated carbon (CECA Italiana, Milan, Italy), with particle size distribution between 1 and 3 mm [8,12,26]. Coconut activated carbon has been chosen owing to its high Au loading, excellent adsorption kinetics, and good attrition resistance [8,12,26].

During this investigation, the Au adsorption at different concentrations of activated carbon was studied [31-35]. Activated carbon has been dried for several days at room temperature before being weighed for adsorption tests. Experiments have been conducted using deionised water.

The tests were conducted in 3000 mL Pyrex glass reactors, under mechanical stirring (350 rev/min), at a temperature of 25°C and pH 10.5, for a total contact time of 1 h [36], by placing in contact the pulp (1000 mL) with different concentrations of activated carbon

(10, 15, 20 g/L) [33]. Small volume withdrawals at set intervals permitted to study the influence of the contact time. After each experiment, the activated carbon was recovered from the pulp and left air drying. Representative samples of carbon were collected and submitted to quantitative chemical analysis [36]; moreover, with the aim to separate the solid from the liquid, the filtration of the slurry has been performed by using pressure filters. At the end of the process, the solid has been washed by ammonia solution.

Gold desorption from activated carbon

The adsorbed Au was stripped from the carbon by a hydroalcoholic solution prepared using absolute ethanol. The desorption tests have been carried out in a Pyrex glass reactor, with a volume of 500 mL [8,33]. The experiments were conducted at temperatures varying from 40 to 80°C, at pH 10.5, during 1-8 h and mechanical stirring at 350 rev/min.

The kinetics of Au desorption process was monitored by withdrawing small volumes of samples at prefixed time intervals and analysing for Au content. The activated carbon was used in repeated adsorption-desorption cycles after a regeneration treatment by chemical washing with diluted hydrochloric acid and heat treatment at 500-600°C in oven [8,17,32,33].

Gold electrowinning

The metallic Au was recovered from the hydroalcoholic solution by electrowinning process [8,36]. Tests were conducted in a jacketed Pyrex glass electrolytic cell, with a total capacity of 100 mL. The temperature of the cell was regulated by a Julabo 5B thermostat. The cell was equipped with a cathode used as a working electrode consisting of platinum wire with a surface area of 100 cm^2, an anode used as counter-electrode (spiral platinum) and a saturated calomel reference electrode. The cell has been connected to the potentiostat – galvanostat AMEL model 555 B [8,18]. The mixing of the solution placed inside the cell was guaranteed by the magnetic stirrer bar. Each electrowinning experiment has been carried out with the solution coming from the desorption step.

Results and discussion

The novelty and originality of the paper consist in the first experimentation, on the laboratory scale, of a schematic chemical circuit of treatment including thiosulphate leaching, for Au extraction from mining wastes samples coming from Balan and Deva deposits, taking account of the results achieved from experiments conducted with samples from Brad Ribita and Brad Criscior [18]. This phase of the study represents a basic step towards the first scale-up of the process for industrial application.

Chemical circuit of treatment

Thiosulphate leaching

By preliminary tests, conducted according to the main bibliographic information [8,12,22,37,38], 41.55% Au recovery has been achieved, considering the gold concentration of the ammonia washing solution (37.10% Au leached + 4.45% Au washed) (Table 1).

Table 1

Gold recovery with a solution of thiosulphate leaching (composition: 2 M $Na_2S_2O_3$, 0.1 M $CuSO_4$ and 0.1 M NH_4OH).

Time (h)	*Au mass (mg)*	*Au recovery (%)*
0.25	0.80	17.85
0.5	0.82	18.25
1	0.82	18.25
1.5	0.86	19.04
2	0.93	20.64
3	1.27	28.17
4	1.66	36.90
5	1.67	37.10
Washing	0.20	4.45

During the investigation different parameters on Au recovery have been studied, including the influence of $Na_2S_2O_3$, $CuSO_4$ and NH_4OH solutions concentration on Au recovery. According to the experimental data (Table 2), higher gold recoveries (about 80% Au after 15 min, without washing) have been achieved during the best kinetic. The trend of the extraction is decreasing; in fact, after 5 h, about 70% Au, including washing, was recovered (66.35% Au leached + 2.56% Au washed) [21].

The obtained experimental results show good gold dissolution kinetics in the aqueous ammoniacal solution of thiosulphate, at room temperature (25°C), while the trend of Au extraction demonstrates that the thermodynamic parameters have not been optimized; in fact, during the best experiment, reported in Table 2, the trend of the gold recovery is decreasing. The explanation can be linked to the thermodynamic instability of the oxidizing agents (copper ions) and complexing ligands (ammonia and thiosulphate) [9,15,24]; consequently, during the next step of investigation the experimental activity will continue with further experiments, finalised to the optimization of the process. It is very interesting to observe that the extraction of gold is already very high in the first 15 min; this is due to the gravimetric enrichment and means that a part of the gold is free and is not incorporated into the mineral matrix (Table 2).

Table 2

Gold recovery with a thiosulphate leaching solution (composition: 2 M $Na_2S_2O_3$, 0.1 M $CuSO_4$ and 4 M NH_4OH).

Time (h)	*Au mass (mg)*	*Au recovery (%)*
0.25	3.63	80.72
0.5	3.56	79.54
1	3.45	76.59
1.5	3.31	73.64
2	3.00	66.68
3	2.97	66.00
4	2.99	66.57
5	2.98	66.35
Washing	0.11	2.56

It is necessary to consider that samples of mining wastes were leached after comminution until an average particle size of 80 μm (80%): it is reasonable to assume that a finer grinding would result in an increase of Au extraction yields, because the thiosulphate is very selective towards Au, unlike cyanide, which forms stable complexes with different elements, such as Cu, Zn, Fe *etc*., therefore the process is less affected by the other metals which are released [6,7,29].

In all cases, only a careful cost analysis of the process can lead to the determination of the most suitable particle size [12,16].

Gold adsorption onto activated carbon

The use of activated carbon significantly enhances gold dissolution and recovery in a thiosulphate-oxygen-copper leaching system and the gold leach kinetics could be significantly improved by its use [9]. As a result, the activated carbon assisted thiosulphate-oxygen-copper leaching system is expected to attract industry interest and potentially more commercial uptake. Studies have shown that the addition of fine activated carbon can ensure good Au recovery from leaching solutions, assuming that the activated carbon forms galvanic couples with gold; consequently, the rate of oxygen reduction is directly linked to the increase of the total surface area available on the carbon [26]. Other studies demonstrate that the adsorption of Au onto activated carbon obtained from vegetal raw material [16,27-31,34,35].

To carry out the study of gold adsorption, the pulp containing gold in the form of the soluble complex has been placed in contact with the activated carbon. Applying the experimental conditions reported in the experimental section, the kinetics of Au adsorption has been studied as a function of the activated carbon concentration. In conclusion, high Au recoveries onto the activated carbon were achieved (Table 3).

It is possible to observe a practically complete gold recovery at the end of the kinetics. The trend indicates that the concentration of gold increases with the increase of the recovery of activated carbon. For example, after 60 min, when the concentration of carbon is of 10 g/L, about 89% Au is adsorbed, but the recovery reaches 99% when the concentration of the adsorbent increases to 15 g/L. In addition, when the concentration is of 20 g/L, 99.99% Au is adsorbed after 30 min. However, the results are not in line with those of previous experimental investigation [18]: it can be assumed that there is a close relationship between Au adsorption, the composition of the process solutions and the difference in Au concentrations, due to both the different composition and gold content in each sample.

The Au adsorption onto activated carbon presented quick kinetics of adsorption with a high Au uptake. The used coconut activated carbon demonstrated a good attrition resistance; the loss of activated carbon was of 0.1 g/kg for each cycle, with a total reuse in 5-6 cycles after chemical-thermal regeneration treatment.

Gold desorption from activated carbon

The aim of the desorption phase has been the stripping of the gold adsorbed onto the activated carbon and its subsequent concentration in hydroalcoholic solution.

The results reported in Table 4 show the best kinetics that permitted to achieve 99.0% Au recovery - including washing - after 8 h, at a temperature of 80°C.

Table 3

Gold adsorption (%) onto activated carbon.

Time	*Activated carbon concentration*		
(min)	*10 g/L*	*15 g/L*	*20 g/L*
15	54.63	60.00	92.20
30	63.74	75.53	99.99
45	78.65	91.19	99.99
60	88.90	95.71	99.99

Table 4

Best results of gold desorption kinetics.

Time (h)	*Au mass (mg)*	*Au yield (%)*
1	0.22	19.80
2	0.37	33.00
4	0.51	45.10
6	0.97	86.90
8	0.98	87.00
Washing	0.33	12.00

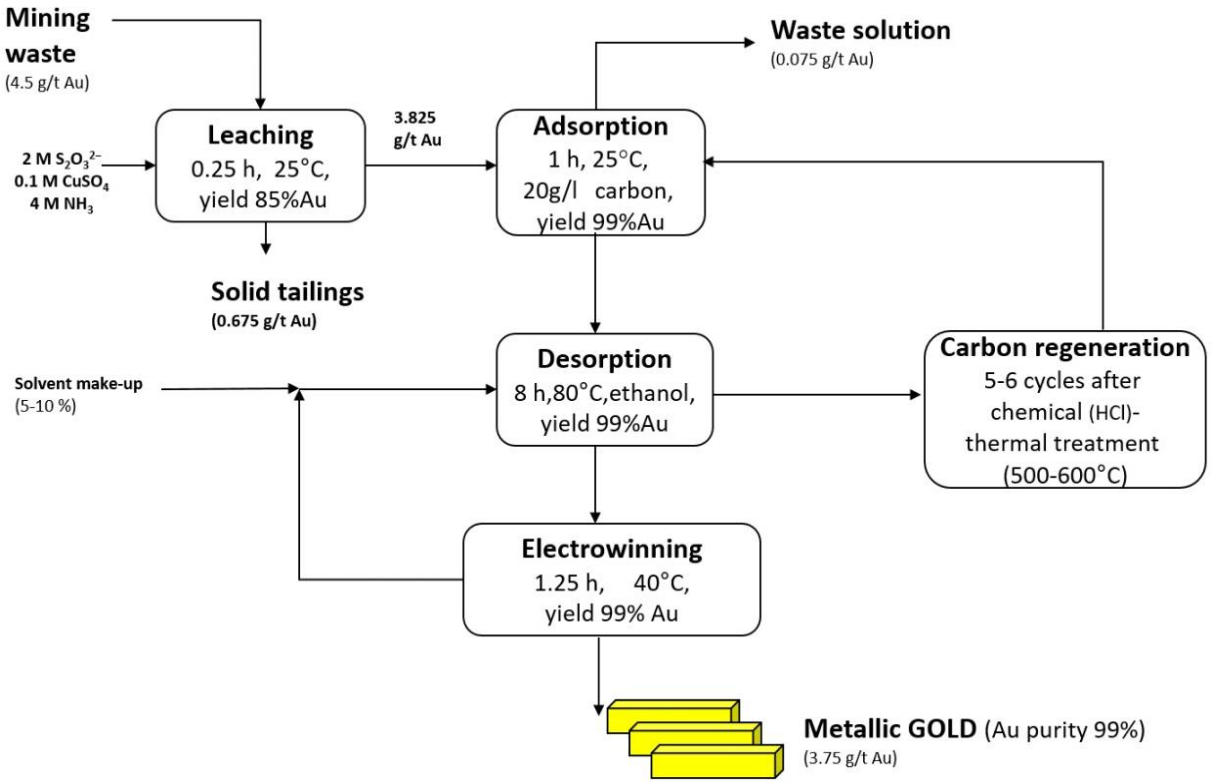

Figure 2. Integrated flow-sheet, including recycling of process solutions and activated carbon, with main parameters applied and gold recovery yields.

Gold electrowinning

The goal of this step was the recovery of Au from the stripping solution by cathodic deposition of the metal in laboratory electrolytic cell. The main results were in line with those obtained in our previous works [8,18].

Schematic integrated flow-sheet

An 85% of Au extraction was experimentally achieved after leaching at a temperature of 25°C, while from adsorption-desorption–electrodeposition cycle about 98% Au has been recovered, purified and concentrated: consequently, a global recovery of the process including leaching–adsorption–desorption–electrodeposition of about 75-80% Au (considering washing) was achieved, in line with the conventional cyanidation process [16-18]. These results are very encouraging, considering that this is a commercially innovative process applied to a low gold content ore (from 1 to 4.5 g/t). The best process yields can be achieved after the optimization of the parameters and operating conditions. Following this investigation, the experimental activity will continue with the main aim to apply the integrated flow-sheet outlined (Figure 2) to the treatment of secondary RMs of other origins, such as WEEE, coming from Romania and from others European and non-European countries.

The integrated flow-sheet allows to recycle the reagents during the complete process, permitting their low consumption: in particular, a loss of 5-10% of thiosulphate has been calculated during leaching process and alcohol usage in the desorption phase, due to evaporation and spilling.

The loss of activated carbon was only 0.1 g/kg during the treatment of mining wastes sample for each cycle; the activated carbon has been regenerated and reused for 5–6 adsorption–desorption cycles (stripping), after chemical-thermal regeneration [27].

The preliminary cost estimation, including both capital and operating costs was prepared, considering a processing plant treating 1000 t per day of gold ore. Capital costs are of about €4 million and annual operating costs are about €3.5 million [8].

During the next step of investigation, the experimental activity will continue with further experiments, finalised to the optimization of the process, to identify the best levels of the parameters and operating conditions. In a

subsequent phase, the outlined integrated flow-sheet will be applied to the treatment of secondary RMs of other origin, such as WEEE, coming from Romania and from others European and non-European countries, in accordance with the application of the principles of the circular economy.

Conclusions

A new hydrometallurgical process has been applied to valorise Romanian mining wastes coming from Balan and Deva deposits by gold extraction with thiosulphate leaching.

A schematic chemical circuit of treatment has been used, taking into account the results achieved from previous experiments conducted with samples from Brad Ribita and Brad Criscior. This represents a step towards the first scale-up of the process for industrial application.

The dissolution rates of gold reached a final value of 85% Au at room temperature (25°C), after 15 min (also considering the gold recovered from the washing solutions), with global recoveries of the process, including leaching–adsorption desorption-electrodeposition, of about 75-80% Au.

Starting from the schematic circuit of treatment on laboratory scale, an integrated flow-sheet has been developed in which the used reagents and coconut activated carbon are recycled: only a 5-10% loss, in particular thiosulphate and alcohol, due to spillage and evaporation has been calculated. The loss of carbon was of 0.1 g/kg waste sample for each cycle; moreover, the carbon was regenerated (by chemical/thermal treatment) and reused for 5–6 cycles of adsorption–desorption.

These results are very good because this new process has never been applied on a commercial scale. Considering the progressive depletion of the reserves of the gold mines it is of great economic importance to recover it from the mining tailings.

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