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Characterization and treatment of artisanal gold mine tailings

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Abstract

The solid waste generated by artisanal gold mining, with high mercury and gold contents, can be found in several areas in the South America. The present study focused on the tailings of an artisanal gold mine area located in the Brazilian northeastern. Samples of the mine tailings were taken and used to perform a physical and chemical characterization study using X-ray diffraction, scanning electron microscopy, neutron activation, X-ray fluorescence, induced coupled plasma-mass spectrometry, among others analytical methods. The results indicate that the material is composed mainly by quartz and goethite, the characteristic size of the particles (d_{50}) is about 150 μm, and the density is close of that of quartz. The main constituents are silicon, iron, and aluminum. The tailings gold content is of about 1.8 mg/kg and the mercury content is of about 10 mg/kg. A remarkable feature of this solid waste is that the gold and mercury are both concentrated in both the fine and the coarse particles, but not in particles of intermediary size. Leaching studies indicated that the tailings are stable in weak organic acids, but soluble in alkaline and aired cyanide solutions, in which 89% of gold and 100% of mercury are extracted in 24 h. Electroleaching experiments, performed using sodium chloride as electrolyte, indicated that mercury and gold are extracted simultaneously and the recovery of both metals can be as high as 70% in 4 h. In addition, chromium, nickel, and lead are found in relatively large amounts in the solution, which indicate an effectively action of the electroleaching method to clean up solid wastes contaminated with metals.

Keywords: Characterization; Solid waste; Cleanup process; Mercury removal; Gold mine; Electroleaching

1. Introduction

Several cases of environment degradation due to artisanal gold mine operations in tropical region have been reported [1–3]. After the 1980s, the practice of subsistence gold mining proliferated in several regions in Brazil, including the goldfields located in the Brazilian northeastern, where mercury contamination was also identified. For instance, in a survey performed at the Itapicuru River hydrographic basin, located in the semi-arid region of Bahia State and the main drink water source for millions of inhabitants, important values of heavy metals were present in sediments [4,5]. The high mercury contents (22–123 μg/kg) close to several artisanal gold mine spots in the region are clearly due to anthropogenic activity.

The solid waste of a site from an artisanal gold mine region located in the Serra de Santa Cruz (Bahia, Brazil), at 10°45′13″S, 40°23′04″W, was chosen as a case to perform a characterization and treatment study, because of the vulnerability of the drink water sources in this semi-arid region and taking into account the apparent extension of the degradation by the mining activity in this area.

The studied site is located in the Jacobina basin, which is part of the São Francisco province of Bahia, with a North–South-trending mountain range that is 200 km long by 15–25 km wide containing gold-bearing conglomeratic units and was formed between 2086 and 1883 Ma [6]. Gold is industrially mined in the Jacobina basin from gold-pyrite-bearing conglomerates and artisanaly mined from both quartz veins and gold-pyrite-bearing conglomerates [6]. There are also in the Jacobina basin disseminated gold-bearing mineralization, such as discordant in quartz veins, at the contact basic-ultrabasic rocks, and subconcordant at different statigaphic levels in quartizite and conglomerate [6]. The Serra de Santa Cruz site is close to the Mina Nova site, where the mineralization is contained in a system of Au–As subvertical veins and extension fissures modified by deformation, and the veins are composed of quartz with free gold, pyrite, rare

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arsenopyrite, tourmaline, chlorite, and white mica surrounded by a halo of disseminated pyrite within the quartzite [6]. The major elements of the quartz vains lode in this region are SiO_2 99.09%, Al_2O_3 0.22%, Fe_2O_3 0.77%, loss on ignation (LOI) 0.26%, and it contains gold between 13 mg/t and 1 g/t, chromium up to 5194 g/t, nickel up to 37 g/t, cobalt up to 22 g/t, uranium up to 16.2 g/t, copper between 19 and 167 g/t, zinc between 11 and 27 g/t, arsenic between 40 and 45 g/t, boron up to 4724 g/t and lead about 11 g/t, yttrium about 1.4 g/t, lanthanum about 2.8 g/t, thorium about 1 g/t and zirconium about 17 g/t [6].

In this study, the gold mine tailings are described, including their mineralogy, grain size, density, and chemical composition, after an electrochemical clean up technique is applied. The methodology presented in the present study can be used to characterize other gold mine tailings, and the study of the nature of solid wastes found in artisanal gold mines is relevant because it can aid the development of specific clean up or immobilization techniques.

2. Experimental techniques

2.1. Tailings sampling

Sampling campaigns were performed at the gold mine tailings of the Serra da Santa Cruz to provide information about the site, the extraction and processing method and the extension of the degradation. The tailings, which are disposed in heaps, were sampled in several points and the solid material dried, homogenized, re-sampled and used for granulometric, mineralogical and other characterization studies. The first sampling campaign took place in 1995, and samples were used to preliminary gold and mercury analysis and to evaluate the efficiency of the amalgamation process to gold concentration; this sample is referred here as sample A, and the results are summarized in Section 3.1. The second sampling campaign, was performed in 2002, and provided samples from a region of the heap related to the material ground in the first stage of the operation (named here as sample B) and another related to the material recently ground (named here as sample C). These three samples (especially sample B) were further used in a more detailed characterization study that included chemical and physical analysis and are presented in this work; samples B and C were also used in the clean up studies using electroleaching.

2.2. Physical characterization

The X-ray diffraction analysis, used to identify the main mineralogical composition of the solid waste, was performed on a Philips PW1710 diffratometer with Cu Kα radiation at the Department of Mining and Materials Engineering of McGill University (Canada). The diffractogram interpretation was made with the assistance of the X'Pert Quantify search match software by PANalytical. The scanning electron microscope JEOL 840-A equipped with a X-ray dispersion energy spectrometry system (EDS) from the Department of Mining and Materials Engineering of McGill University (Canada) was used to evaluate the composition and the texture of the particles of the tailings. The samples were directly mounted in glass lames with an epoxy resin and carefully polished.

The tailings size distribution was evaluated using the conventional screening test with a ro-tap and a set of screens from 20 to 400 mesh Tyler and the density was measured using the classical pycnometer method.

2.3. Inorganic assays

Several analytical methods were used to evaluate the tailing elements content in order to produce representative results. These methods included neutron activation analysis (INAA), induced coupled plasma (ICP), induced coupled plasma and mass spectroscopy (ICP-MS), fire-assay (FA), infrared (IR), atomic absorption spectroscopy with cold vapor generation (FIMS), and atomic absorption spectroscopy (AAS). The neutron activation analyses were performed at the Department of Physical Engineering of the Polytechnic School (University of Montreal, Canada), and the other analyses were performed at the Activation Laboratories Ltd. (Canada). One remarks that the accuracy of the INAA is about 5%, and the detection limit of FA is about 0.03 mg/kg and FIMS and FA-AAS is about 5 μg/kg.

2.4. Solubilization experiments

The toxicity characteristic leaching procedure (TCLP) developed by U.S. EPA [7,8] was used in this study for a preliminary evaluation of the hazard potential of the tailings. The TCLP experiments use buffered acetic acid with pH 5, to simulate the action of organic acids, after, the solution metals content were analyzed.

The current industrial process to gold extraction from ores uses cyanide solutions. Gold cyanidation is an electrochemical process that occurs according to the parallel reactions [9–11]:

$$2Au + O_2 + 4CN^- + 2H_2O \rightarrow 2[Au(CN)_2]^- + 2OH^- + H_2O_2 \quad (1)$$

$$2Au + 4CN^- + H_2O_2 \rightarrow 2[Au(CN)_2]^- + 2OH^- \quad (2)$$

Silver cyanidation follows reactions analogous to that given by Eqs. (1) and (2). Copper can form several complexes with cyanide, which depends of the cyanide concentration and the pH. In this study, the bulk leach extractable gold (BLEG) test was used to evaluate the recoverability of mercury and precious metals from the tailing, without additional grinding, by cyanidation. This test corresponds to a 24 h roller bottle alkaline cyanidation followed by filtration and analyses of the metals in solution by induced coupled plasma and mass spectrometry (ICP-MS).

In the present study, both TCLP and BLEG leach experiments and solution analysis were performed at the Activation Laboratories Ltd. (Ontario, Canada).

2.5. Electroleaching experiments

The electroleaching process has several analogies with the electrodyalitic remediation [12]; however, the main objective of

Table 1
Electroleaching test conditions

Test #	Sample	Sample weight (g)	Pulp density (%)	Initial chloride concentration (M)	Current (A)	Potential (V)	Electrolysis time (h)
1	B	100	16.7	1.0	1.0	4.3	4.0
2	B	70	16.7	2.0	1.0	3.9	4.0
3	C	100	16.7	1.0	1.0	4.1	1.0
4	C	100	16.7	1.0	2.0	4.9	5.0

this process is the readily dissolution and recovery of the metals, thus it takes place in stirred tanks and often uses sodium chloride as electrolyte [13–19]. In the electroleaching process, at the anodic surface, the chloride ion (Cl^-) is oxidized to free chlorine (Cl_2) while in the cathodic surface, the water reduces with the production of hydrogen (H_2) and hydroxyl ions (OH^-). Then, the free chlorine reacts with the hydroxyl ions to produce chloride and hypochlorite ions (ClO^-). Depending of the solution pH, the hypochlorous acid (HClO) can dissociate in hypochlorite ions and hydrogen that reacts with the hydroxyl ions to form water. Later, the chlorate ions (ClO_3^-) are generated by chemical reaction of hypochlorite ions and hypochlorous acid and by oxidation of the hypochlorite at the anodic surface. The hypochlorite ions (ClO^-) or the hypochlorous acid will oxidize the elemental mercury as follows [16,17,19]:

$$Hg + 2ClO^- + 4Cl^- + 2H_2O \rightarrow Cl_2 + HgCl_4^{2-} + 4OH^- \tag{3}$$

Complex ions of mercury with one to four chlorine atoms will be present in the solution depending of the chloride concentration, temperature, and pH. However, the $HgCl_4^{2-}$ is the predominant specie in the current experiment condition.

The chlorate ions (ClO_3^-) dissolve the elemental gold according to the following reaction [16,17,19]:

$$Au + ClO_3^- + 7Cl^- + 3H_2O \rightarrow 2AuCl_4^- + 6OH^- \tag{4}$$

The soluble mercury and gold species are reduced at the cathode surface, and due to the susceptibility of gold to form amalgams with mercury, this will be the form of the deposits of this metal on the electrode surface [15–17,19].

In the present study, the electroleaching experiments were performed at the Laboratory of Extractive Metallurgy of the Federal University of Bahia (Brazil), using a potential-current source, a magnetic stirrer, a stainless steel cathode, a graphite anode, and pulp inside a beaker of two liters that acts as a simple electrolytic cell.

In the electroleaching process, several variables can play important roles and have to be taking into account in the process optimization [13,15–17,19]. The most important variables are current density, initial chloride concentration, distance between the electrodes, pH, ore particles size distribution, temperature, solid concentration in the pulp (or pulp density), and experiment duration. Table 1 summarizes the conditions that were used to perform the electroleaching experiments with samples B and C. One remarks that the initial pH was 7.0 and the initial temperature was between 29 and 30 °C, and the chloride concentration, pH, and temperature evolved without strict control during the experiments. The electrodes were a 3.7 cm × 5.3 cm × 0.1 cm stainless steel cathode and a 5.2 cm × 2.9 cm × 0.3 cm graphite anode. The current density were 2.5 and 5 A/dm^2, and the distance between the electrodes was 25.4 mm. The leaching residual and the evaporated solution content where evaluated by X-ray fluorescence (XRF). The metals solution content were then evaluated by atomic absorption spectroscopy (AAS).

3. Results and discussion

3.1. Physical and chemical characterization

The diffratogram scan for sample B of the artisanal gold mine tailings is shown in Fig. 1. This diffratogram clearly shows that the tailings are predominantly composed by quartz (SiO_2) with small amount of goethite (FeO(OH)), which is in agreement with the site geology [6].

Backscattering scanning electron microscope images of sample B of the tailings are presented in Fig. 2. These images show the predominance of quartz and the presence of some goethite regions. The SEM images also shows the presence of some scarce elements, such as a titanium, zirconium, cerium, bismuth, and lanthanum characteristic of the Jacobina basin geologic region [6]. One also remarks a liberated gold particle of about 3 μm, which is compatible with the gold occurrence in this region [6].

The density value of the artisanal gold mine tailings is 2.76 g/cm^3 that is close to the quartz density (2.65 g/cm^3), the major mineralogical constituent of the tailings. The enhance-

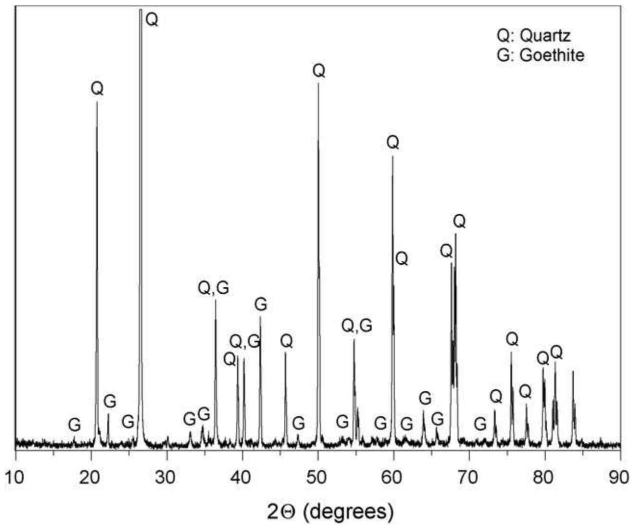

Fig. 1. X-ray diffractogram for the ore tailings (sample B).

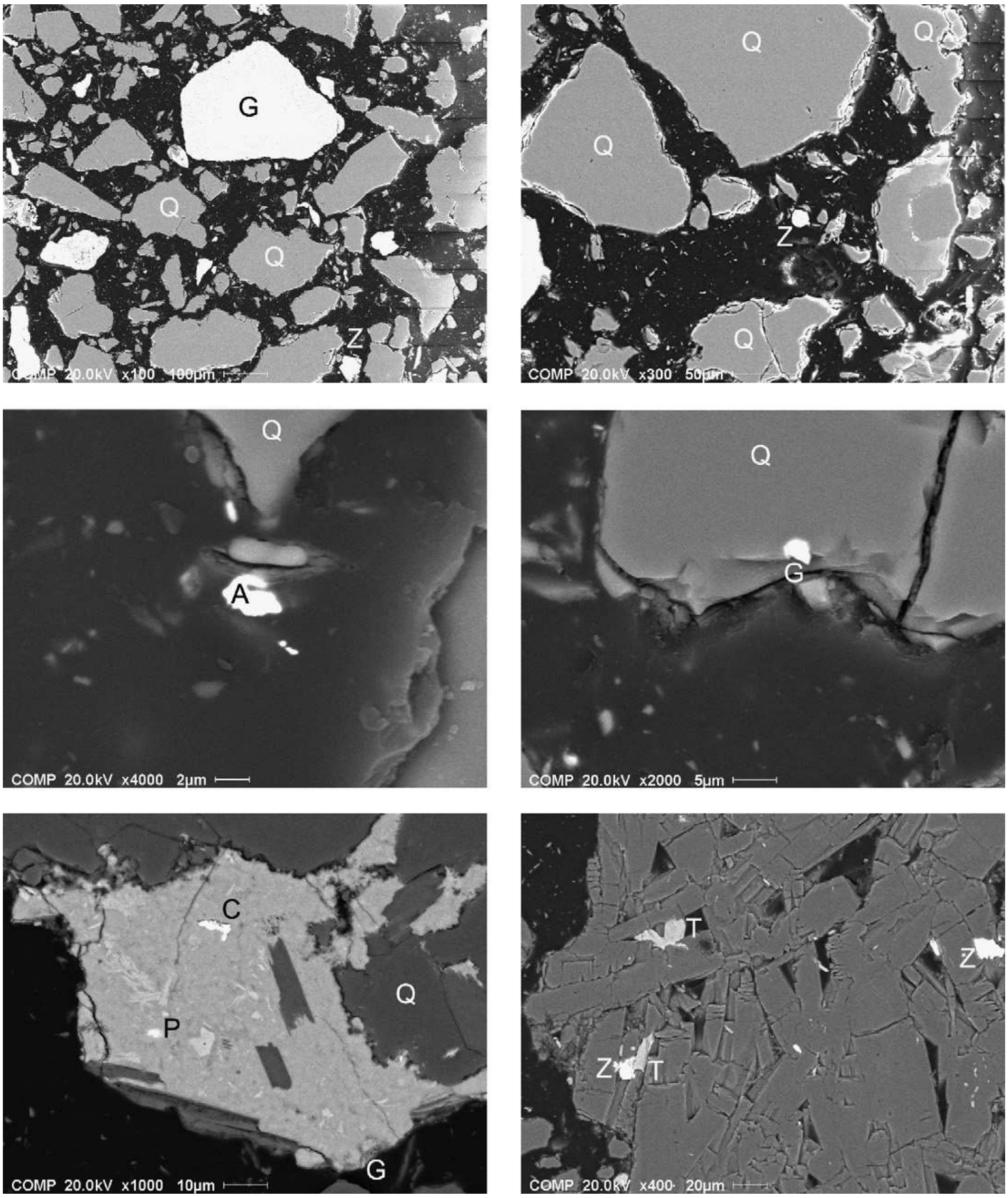

Fig. 2. Backscattering scanning electron microscope images of the ore tailing particles (sample B). In this figure, A stands for gold (Au), Q for quartz (SiO_2), G for goethite (FeO(OH)), Z for zircon ($ZrSiO_4$), C for cerium (Ce) and lanthanum (La) mineral, P for gadolinium (Gd) phosphate mineral, T for a titanium mineral (TiO_2).

ment in this value is due to the presence of goethite in the tailings.

The size distribution of the particles of the artisanal gold mine tailings, for samples A and B, are presented in Fig. 3. One remarks that the characteristic diameter that retains half of the particles population (d_{50}) in both cases is about 150 μm, which characterizes a relatively coarse tailings for a gold ore [9]. It is likely that a larger amount of gold is still locked inside the tailing particles, in other words, the gold particles were not completely liberated, due to the inadequate grinding procedure used in this artisanal gold mine operation.

Table 2 and Fig. 4a and b show the gold and mercury content and the distribution in the particles from sample B. As expected, that gold is very concentrated in the coarse particles. An interesting feature of these tailings is the large amount of mercury in both finer and coarse particles. One remarks also that mercury occurs associated with gold, which seems to indicate the formation of an amalgam with this metal. The metal distribution clearly shows that the gravity concentration procedure, using a simple table, collected particles in the region of 200–400 μm, but not the finer ones. The high values for coarse particles are due to the non-liberation of the gold particles mentioned above. The cumulative gold and mercury distribution shows that gold is more heterogeneously distributed in the particles due to the concentration of particles with intermediate size and gold location of this metal in the coarse particles.

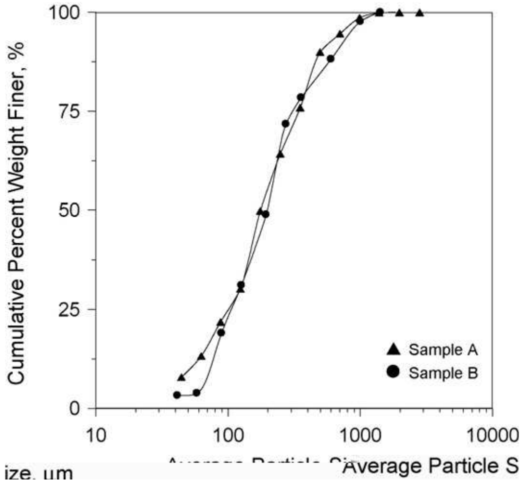

Fig. 3. Size distribution of the particles from two places of the tailings heap (samples A and B).

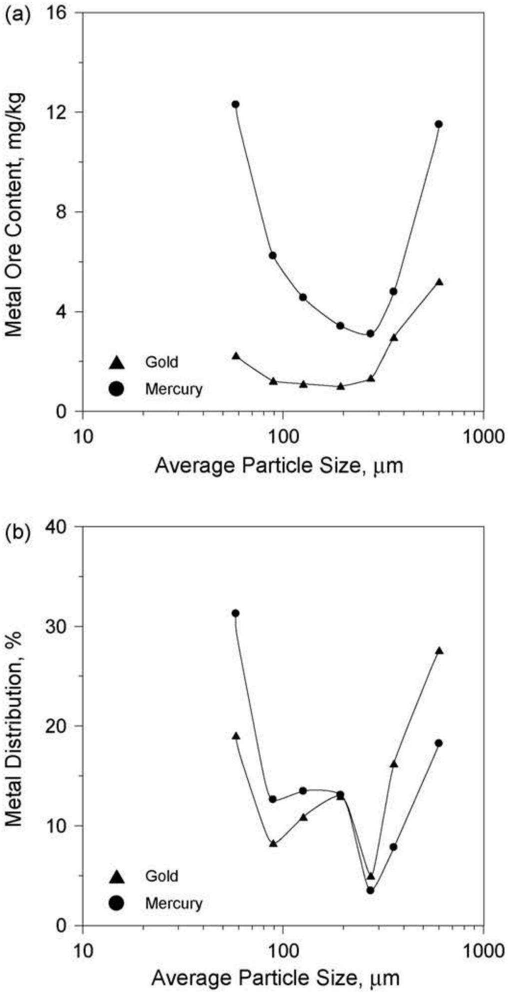

Fig. 4. Gold and mercury in the particles of the tailings pile (sample B): (a) metals ore content, (b) metals distribution.

Table 3 presents the concentration of the major constituents and trace elements from sample B. The analytical method used for the determination of each element is also presented in this table. As indicated by the mineralogic characterization, the main constituents of the tailings are silicon and iron that account for about 50% of the weight. The sample also has some aluminum and magnesium. The total of the major constituents is 108% due to the accuracy of the INAA that is about 5%. Among the trace elements, it can be found arsenic, nickel, copper, zinc, uranium, gold, silver, cadmium, molybdenum, lead, and tungsten characteristic of the rocks from the Jacobina basin geologic region [6].

The tailings gold content is about 1.8 mg/kg and the mercury content is about 10 mg/kg, which are in agreement with the results of metal content in each size fraction, shown in Fig. 4a. The gold content of the tailing is high and has the same order of magnitude of the gold ore treated by a cyanidation plant not far from this region.

3.2. Tailings treatment

The preliminary evaluation of the pollution hazard and the applicability of soil cleanup and valuable metal recovery techniques from the artisanal gold mine tailings were inferred through amalgamation and leach experiments. The measured fraction of the residual gold content that can be re-extracted by amalgamation with mercury for sample A is about 3–13%, which indicates that the gold recovery by the amalgamation process previously performed in the artisanal mining operation is relatively efficient.

Table 4 shows, for sample B, the fraction of some metals from the artisanal gold mine tailings that can be extracted by

Table 2
Gold and mercury content and distribution in the particles of the tailings pile (sample B)

Average size (µm)	Percent weight finer (%)	Gold content (mg/kg)	Mercury content (mg/kg)	Gold distribution (%)	Mercury distribution (%)
1001.50	2.31	–	–	–	–
601.04	9.48	5.20	11.50	27.6	18.3
357.07	9.77	2.97	4.79	16.2	7.8
273.86	6.71	1.33	3.10	5.0	3.5
193.65	22.92	1.01	3.41	13.0	13.1
126.10	17.68	1.10	4.55	10.9	13.5
89.16	12.10	1.22	6.23	8.3	12.6
58.09	15.19	2.24	12.30	19.0	31.3
41.35	0.57	–	–	–	–
31.95	3.26	–	–	–	–

Table 3
Concentrations of the major constituents and trace elements in the gold tailings (sample B)

		Assay method (see Section 2.3)
SiO_2	83%	INAA
Fe_2O_3	19%	INAA
Al_2O_3	4.9%	INAA
MgO	0.46%	INAA
K_2O	0.36%	INAA
Na_2O	0.216%	ICP
Cr_2O_3	0.071%	INAA
S	0.0200%	IR
CaO	0.016%	INAA
MnO	0.0130%	ICP
Cu	87 mg/kg	ICP
Ba	85 mg/kg	INAA
As	71 mg/kg	INAA
Ni	67 mg/kg	INAA
W	62 mg/kg	INAA
Zn	55 mg/kg	ICP
Pb	21 mg/kg	ICP
Hg	9.927 mg/kg	FIMS
Se	3.3 mg/kg	ICP-MS
U	2.9 mg/kg	INAA
Au	1.876 mg/kg	FA
Mo	1.1 mg/kg	INAA
Ag	1.02 mg/kg	ICP-MS
Cd	0.7 mg/kg	ICP
B	<1 mg/kg	ICP-MS

Table 4
Extraction of metals from the tailings by cyanidation (sample B)

Element	Recovery (%)
Ag	12
Au	89
Cu	46
Hg	≈100

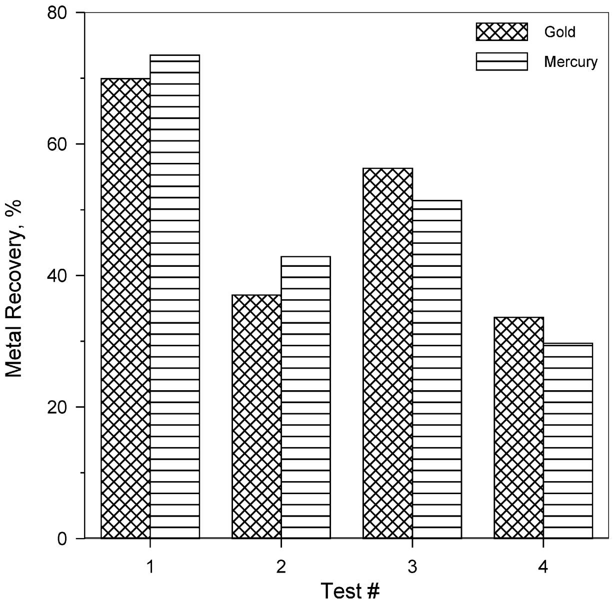

Fig. 5. Gold and mercury recovery for the ore tailings by electroleaching of samples B (Test #1 and 2) and C (Test #3 and 4) (for the test conditions see Table 1).

cyanidation in 24 h. The apparent small recovery of silver is due to its very low content in the ore. For copper, 54% of the metal could not be leached due to a possible lockup and the presence of several copper minerals, especially sulfides, which react slowly with cyanide. One remarks that in the present case a larger amount of gold could be leached but the results indicate that 11% of the gold is locked in the particles of the tailing and are inaccessible to cyanide. Finally, one observes that for the studied tailings, the mercury can be completely extracted by cyanide due to the fact that it is found at the surface of the ore particles, likely associated with the residual gold; thus, it is easily accessible. One remarks that the cyanidation treatment of the tailings was included in the present work, because the results of this study are interesting to demonstrate that the gold and mercury are fully available to leach in the present case, and grinding is not necessary to liberate the gold particles. The TCLP test performed with sample B indicates that for weak acidic environments these tailings seem relatively stable, as shown in Table 5.

The effect of the electrolyses time on the pH, and on the concentrations of ClO^-, $ClO_3{}^-$, Hg in solution and Hg in the tailings during the electroleaching process was discussed by Scheiner et al. [13]. During the electroleaching experiments performed with the artisanal gold mine tailings a temperature enhancement from 29 to 30 and 33.4 to 42.7 °C was observed and an important pH increasing due to the hydroxyl ions generated during the metals solubilization (see Eqs. (3) and (4)) and the water electrolysis on the cathode. The pH should be controlled in a value close to seven to avoid mercury precipitation as HgO [13,15–17,19].

One remarks that the current density and the distance between the electrodes used in the electroleaching experiments were at the same order of magnitude of that previously reported, but the pulp density was slightly lower and the current higher [13,15]. Fig. 5 shows the gold and mercury recovery for samples B and C by electroleaching. One notes that the gold recovery, which ranges from 30 to 70% for a maximum of 5 h of electrolysis, is of the same order as the mercury recovery in all experiments. This fact confirms that the mercury is found associated with the

Table 5
Leach analysis (in mg/L) (toxicity characteristic leaching procedure) (sample B)

	Element									
	Ag	As	B	Ba	Cd	Cr	Hg	Pb	Se	U
Solution content	< 0.1	<0.1	<0.1	<0.1	<0.01	<0.1	<0.01	<0.1	<0.1	<1
Leachate criteria	5	2.5	500	100	0.5	5	0.1	5	1	10

Table 6
Elements content in solution after electroleaching (in mg/kg)

Test #	Cr	Cu	Fe	Ni	Pb
1	2.63	0.18	1.45	1.75	0.94
2	6.40	0.27	2.51	2.63	1.72
3	1.27	0.23	1.35	1.48	1.48
4	5.05	0.22	1.00	1.34	0.64

gold in this particular solid waste, as discussed in Section 3.1. In sample B the mercury solubilizes easier than the gold and the opposite behavior is found for sample C, which is related with the fact that sample C has a gold content smaller than sample B and it seems more liberated.

The semi-quantitative XRF analysis of the evaporated electroleaching solution of Test #1, in addition to the large amount of the expected elements (Na = 42.9%, Cl = 35.4%, and O = 21.2%), indicated a non-negligible amount of several elements (Ca = 130.2 mg/kg, Si = 124.5 mg/kg, S = 57.6 mg/kg, Al = 42.6 mg/kg, K = 26.3 mg/kg, Cr = 25.6 mg/kg, Fe = 13.3 mg/kg, Hg = 6.9 mg/kg, Ni = 2.7 mg/kg, Cu = 2.3 mg/kg, and Au = 1.5 mg/kg). Table 6 shows an AAS quantitative analysis of some solubilized elements by the electroleaching process. One observes that chromium, nickel, and lead are found in relatively large amount, which associated with the fact that some material may be found deposited in the cathode, indicates an effectively action of the electroleaching method to clean up solid waste contaminated with heavy metals.

4. Conclusions

A solid waste from an artisanal gold mine with significant mercury and gold contents was sampled, characterized, and used to clean up studies. The XRD results demonstrated that the tailings are composed by quartz and goethite; the SEM images and EDS spectra revealed the tailings texture and the identity of species that confirmed the presence of gold and indicated the presence of other less common elements.

The chemical analyses, performed with different analytical methods, such as ICP-MS, INAA, XRF, ICP, AAS, among others confirmed the mineralogical results and showed the presence of some less common elements, characteristic of the Jacobina basin geologic region [6]. The tailing gold and mercury contents are about 1.8 and 10 mg/kg, respectively. The gold and mercury distribution in the size fractions shows that these metals are associated and the gold is not completely liberated in the larger fractions.

The leach experiment shows that mercury can be completely recovered by cyanidation and the gold and copper only partially. These results confirm that gold is not completely liberated and indicate that copper occurs as sulfides and oxides that react slowly with cyanide.

The electroleaching experiments demonstrated that, in the present case, mercury and gold are extracted almost simultaneously. In addition, it shows that the recovery of both metals can readily reach more than 70%. The results presented in this work can be further used as a start point for optimization of the cleanup and metal recovery processes.

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