Gold Characterization by MLA and Technological Tests: Discussion of Sample Preparation and Results

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Abstract Gold has been present throughout the history of mankind and used to make jewelry and coins, and recently, it is put into use in industry. The price of gold in international market had a significant increase, surpassing 100 % in the last 5 years. Thereby, deposits with low levels of gold content as well as gold with complex associations or in a very fine particle size became exploitable again, allowing new projects and expansion of existing ones. However, as maximum process efficiency is indispensable and deep knowledge of the characteristics of these minerals and their behavior in face of beneficiation processes. Consequently, an accurate routine for mineralogical and technological characterization is essential. This chapter presents a methodology for characterizing low-grade ores with fine-grained gold. The procedure was conducted in three different samples from different regions, mineralogical assemblages, and grades.

Gold grains and their associations were characterized by SEM-based automated image analysis using the MLA SPL_Lt method, which consists in the initial search of gold, as well as other quite heavy minerals (e.g., platinum, silver, etc.), by atomic number contrast (backscattered electrons) and further identification of these phases and their mineral associations by EDS spectra. The automated routine is set up for allowing the identification of gold grains measuring down to 0.5 μm. Gold extraction assays were accomplished by amalgamation and cyanidation leaching of products from heavy liquid separation. The grades and partition of gold among mineral separation products are discussed in order to evaluate the significance of the gold density preconcentration prior to the characterization by an automated image analysis system. The results attained at mineralogical studies are correlated to the extraction of gold.

Keywords Gold characterization • Image analysis • Sample preparation

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1 Introduction

Gold always has been present throughout the history of mankind. Its brilliance and rarity have made it much sought after for jewelry and coin manufacturing. More recently, gold has been acquired several industrial noblest uses. Moreover, gold is a highly required commodity in times of crisis, and due to the current situation of uncertainty in the global economy, its price had a significant increase in its value, surpassing 100 % in the last 5 years (www.goldprice.com).

Thereby, deposits with low levels of gold content and gold with complex associations or in a very fine particle size became exploitable, allowing new projects and expansion of existing ones. However, maximum process efficiency is indispensable and deep knowledge of the characteristics of these minerals and their behavior in face of beneficiation processes. Therefore, an accurate process mineralogy routine is essential.

Characterization of gold ores differs from the conventional approach since there is a great difficulty to find gold grains for qualitative and quantitative analyses due to the low content of gold (mostly <2 ppm in Brazil) and sparse distribution in mineral deposits. Regarding the high density of gold (19.3 g/cm^3), it is usual to perform a density concentration in order to enrich the heavy products with gold and other heavy minerals to enable a faster analysis on a large number of gold grains or gold-bearing particles without spending much hours searching in multiple polished sections [1].

Density concentration can be carried out by different methods: Wilfrey Table [2], centrifuges, heavy liquid separation, Mozley table, elutriation, and hydro separator [3]. The aim of all these methods is to promote a high-efficient concentration of gold associated with low content of light phases.

The automated methods of image analysis, which are gaining importance in the mining sector due to reliability of the results obtained and the statistical significance afforded by the analysis of large numbers of particles [4], are aiming to obtain the mineralogical composition of the sample, the mineral assemblages, the chemical composition of each phase identified, the partition of elements, the liberation curves of phases of interest, as well as other information.

Automated image analysis by SEM is an efficient and effective mean of identifying visible gold occurrences and distribution, but it is limited on the identification of "invisible" gold [4], which is considered a solid solution or colloidal gold in the range from 0.1 to 0.001 μm [2]. The most sensitive analytical techniques available for the detection of "invisible" gold and other trace elements in minerals are microbeam techniques. These methods can give sensitivities down to the low ppm and even low ppb range, which can detect nanometer-sized micro inclusions and effectively identify "invisible" gold [2].

Although the automated routine through an automated SEM-based mineral liberation analysis system is suitable for the identification of visible gold, due to the difference of spatial resolution magnitude between BSE (0.1–0.02 μm) and

X-ray (2–5 μm) [5], the traditional methodologies are well suited to the determination of particles greater than 5 μm.

This study involves the characterization of the forms of occurrence of gold and its association by quantitative mineralogy by electron beam at an MLA system, and technological tests are to evaluate the recovery of gold by amalgamation and cyanidation in three gold ore samples. The sizes of gold grains are also highlighted since they occur mostly below 5 μm. The grades and partition of gold among mineral separation products (light, intermediate, and heavy products) are discussed in order to evaluate the significance of the gold density preconcentration prior to the characterization by an automated image analysis system. The results attained by cyanide leaching and MLA are compared.

2 Materials and Methods

2.1 Sample Preparation and Gold Technological Tests

The study was carried out in three samples: one from oxidized ore and two from sulfide ore, from two different Brazilian deposits and composed by drill core fragments (approximately 400 kg of each sample). The samples were identified, according to the main composition and origin, as high-grade samples from the same deposit—OXI-HG and SULF-HG and low-grade samples from another deposit—SULF-LG.

The sample preparation comprised sequential steps of comminution, homogenization, and sampling to obtain representative aliquots for this study, comminuted below 0.30 mm. The importance of the sampling is particularly relevant for low-grade ores because of the great care taken on splitting—it was always conducted with rotary sample dividers, both for sample preparation and for the chemical analysis.

The samples were screened in narrow sieve fractions down to 0.037 mm (0.21, 0.15, 0.074, and 0.037 mm) and each sieve fraction was separated by heavy liquid (bromoform) at 2.8 g/cm^3. The sink product of each fraction was then elutriated under controlled conditions to simulate a concentration at higher density and generated the intermediate and the heavy products. So, at the end of mineral separations, there were three products: light (float at 2.8 g/cm^3), intermediate (sink at 2.8 g/cm^3 and light from elutriation), and heavy (sink at 2.8 g/cm^3 and heavy from elutriation). One representative aliquot of the heavy product was taken for SEM-MLA analyses.

Gold extraction assays comprised cyanidation leaching of the light and intermediate products and amalgamation plus cyanidation for the heavy products (coarse gold is recovered at the amalgam and the remaining gold can be accessible to the cyanide solution or not). All cyanide solutions containing gold, as well as the

amalgam and the solid residues, were assessed for the determination of the gold content.

The amalgamation was conducted in the weight proportion of 1:20 (Hg/sample) in a 50 % w/w solids suspension for 15 h; after that, the amalgam (Hg + Au + Ag) was separated from the ore by elutriation (density separation). The cyanidation was performed with a sodium cyanide (NaCN) solution and a concentration of 2,000 ppm of NaCN in a 50 % w/w solid suspension, whose pH was balanced between 10 and 11 with sodium hydroxide (NaOH) under agitation for 48 h. The final solution and the residue were recovered by filtration.

The content of gold in each product, amalgam, cyanide solution, and solid residue was evaluated by fire assay at an international certified laboratory (gold assessed by fire assay with lower limit of 0.01 ppm or 0.01 mg/l).

2.2 SEM-MLA Settings

Due to the low grades of gold, a density concentration previous to SEM analysis was performed by heavy liquid separation and elutriation in order to enrich the sink products and increase the concentration of gold grains in the products to be characterized. To provide statistical robust data on the quantification of gold associations, 26 polished sections were analyzed by MLA.

The gold characterization was carried out by systematic analysis on scanning electron microscopy (SEM) with a field emission source (FEG) coupled with an energy-dispersive spectrometer (EDS) in order to identify gold bearing particles and determine the gold grain composition. The search for gold grains was done automatically by MLA software (Mineral Liberation Analyser—FEI), considering the atomic number (gray levels contrast—backscattered electrons) and chemical composition (EDS microanalysis). It was determined by the gold mineral association, gold liberation-exposed perimeter, and gold grains' size distribution.

The gold grains and their associations were characterized by MLA-SPL_Lt method, which consists in the initial identification of gold by atomic number contrast (backscattered electrons) and EDS spectrum and further identification of the associated phases (perimeter of contact). The operating conditions permitted the identification of gold grains with sizes down to 0.5 μm.

3 Results

3.1 Technological Characterization and Gold Extraction Tests

The weight distribution by sieve fraction, the grades, and the distribution of gold by fraction are shown in Table 1. The grade of gold is around 3 ppm for samples from the high-grade deposit (tendency to enrichment towards the coarse faction) and 0.51 ppm for the low-grade sample (increasing at the finest fraction).

The particle size distribution demonstrates that oxidized sample presents a significant higher amount of fine fraction (below 0.037 mm) than sulfide samples; despite the origin of sulfide samples, the total content of gold associated with the fines represents around 26 % of the total gold from the sample, while this value is 37 % for the oxidized sample. This result demonstrates once more the importance of characterizing gold in fine grains and also the development of technologies to improve their recovery.

The summary of the results of technological characterization studies (total composed from fractions below 0.30 and above 0.037 mm) considering the grades of gold, weight, and gold distribution in each product is shown in Table 2.

The preconcentration done in three steps demonstrates that the heavy products (submitted to MLA analysis) represent different mass recovery and depend on the composition; for sulfide samples, this product represents around 10 and 11 % in mass; however, for oxidized samples the heavy products account for less than 3 % in mass.

The content of gold in heavy products exceeds 30 ppm (mass recovery of 2.8 % in assay) for the oxidized samples and 30 % of gold in assay. For sulfide samples, despite the content of gold in the sample (low- or high-grade ore), the content of gold is around 50 ppm in heavy products (mass recovery around 10 % in assay).

The content of gold in intermediate products is relatively low for sulfide sample, but relatively high for oxidized sample; so, mineralogical analysis carried out in

Table 1 Particle size distribution and gold content and distribution (samples comminuted below 0.30 mm)

Sieve fraction (mm)	OXI-HG			SULF-HG			SUL-LG		
	wt (%)	Au (ppm)	Dist (%)	wt (%)	Au (ppm)	Dist (%)	wt (%)	Au (ppm)	Dist (%)
−0.30 + 0.21	15.2	3.66	19.8	18.2	4.57	23.2	19.0	0.51	19.0
−0.21 + 0.15	11.8	3.19	13.3	14.8	3.35	13.9	17.5	0.48	16.7
−0.15 + 0.074	16.5	3.03	17.8	22.8	3.16	20.2	24.6	0.44	21.3
−0.074 + 0.037	13.5	2.56	12.3	17.3	3.38	16.4	19.7	0.45	17.6
−0.037	43.0	2.40	36.8	26.9	3.50	26.3	19.1	0.68	25.5
Total −0.30	100.0	2.81	100.0	100.0	3.57	100.0	100.0	0.51	100.0

Table 2 Results of density separation for the fraction size above 0.037 mm (samples comminuted below 0.30 mm)

Sample −0.30 +0.037 mm	Products	Weight (%)		Grades	Distribution Au (%)	
		Assay	Sample	Au (ppm)	Assay	Sample
OXI-HG	Light	84.6	48.20	0.88	23.80	15.00
	Intermediate	12.6	7.20	11.50	46.50	29.40
	Heavy	2.8	1.60	32.90	29.70	18.80
	Total calc.	100.0	57.00	3.12	100.00	63.30
SULF-HG	Light	48.3	35.30	0.73	9.83	7.24
	Intermediate	40.4	29.50	3.21	36.10	26.60
	Heavy	11.3	8.30	17.20	54.10	39.80
	Total calc.	100.0	73.10	3.60	100.00	73.70
SULF-LG	Light	77.7	62.9	0.16	26.60	19.80
	Intermediate	12.1	9.83	0.90	23.40	17.40
	Heavy	10.2	8.21	2.31	50.00	37.30
	Total calc.	100.0	80.90	0.47	100.00	74.50

oxidized sample can also consider a preconcentration step, but it has to be lower than the density established for sulfide samples, taking into account the density of the separation (gold associated with other phases besides sulfides).

The content of gold in light product is at least three times lower than that in the fraction and represents the largest amount of sample; the analysis of this product by MLA will bring additional difficulties in terms of gold identification and number of polished sections to be analyzed.

The extraction of gold by cyanidation for light and intermediate products and by amalgamation and cyanidation for the heavy products is presented in Table 3.

The extraction of gold by cyanide leaching (liquor) in the light and intermediate products is similar to each sample, suggesting that the association of gold in both products is similar, though the gold recovery varies significantly among the samples. The high losses of gold in the solid residue are related to inaccessible gold or refractory gold.

The content of gold associated with the amalgam varies markedly among the samples and is related to the gold grains in particle's surface (exposed gold); the much higher grades of gold recovered by amalgamation of OXI-HG sample are probably associated with the presence of a gold nugget than to higher proportion of fine gold grains in exposed surface.

3.2 Gold Association by SEM-MLA for the Heavy Products

The total amount of gold-bearing particles and the number of gold grains identified by MLA routine are exposed in Table 4. For OXI-HG sample, it is notable the high

Table 3 Results of amalgamation and leaching tests for the density separation products (samples comminuted below 0.30 mm)

Samples, total −0.30 +0.037 mm		OXI-HG			SULF-HG			SULF-LG		
			Dist. Au (%)			Dist. Au (%)			Dist. Au (%)	
Products from amalgamation/cyanidation		Au (ppm)	Assay	Sample	Au (ppm)	Assay	Sample	Au (ppm)	Assay	Sample
Float	Liquor	0.61	69.6	10.50	0.42	57.1	4.14	0.12	76.30	15.10
	Residue	0.27	30.4	4.57	0.32	42.9	3.11	0.04	23.70	4.70
	Total calc.	0.88	100.0	15.00	0.73	100.0	7.24	0.16	100.00	19.80
Intermediate	Liquor	7.29	63.2	18.60	1.59	49.5	13.20	0.61	72.70	12.70
	Residue	4.25	36.8	10.80	1.62	50.5	13.40	0.25	27.30	4.74
	Total calc.	11.50	100.0	29.40	3.21	100.0	26.60	0.90	100.00	17.40
Sink	Amalgam	25.40	77.2	14.50	10.60	61.6	24.60	0.02	0.69	0.26
	Liquor	1.55	4.7	0.88	2.18	12.7	5.07	1.64	70.90	26.40
	Residue	5.96	18.1	3.40	4.40	25.7	10.20	0.66	28.40	10.60
	Total calc.	32.90	100.0	18.80	17.20	100.0	39.80	2.31	100.00	37.30

Table 4 Gold-bearing particles and grains (samples comminuted below 0.30 mm)

Fraction (mm)	OXI-HG		SULF-HG		SULF-LG	
	Part.	Grains	Part.	Grains	Part.	Grains
−0.3 +0.21	95	139	50	54	23	43
−0.21 +0.15	73	109	37	38	34	41
−0.15 +0.074	46	85	20	21	56	68
−0.074 +0.037	62	159	10	13	14	16
Total +0.037	276	492	117	126	127	168

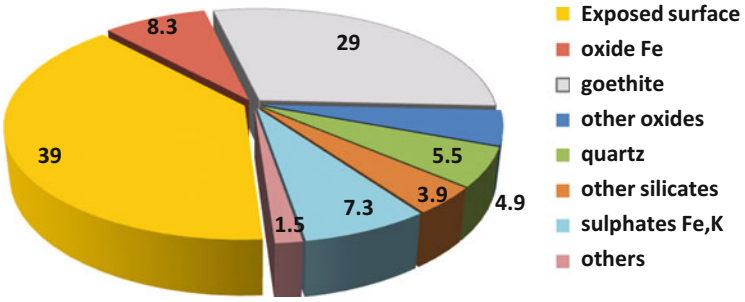


Fig. 1 Gold associations (% of perimeter of contact), sample OXI-HG

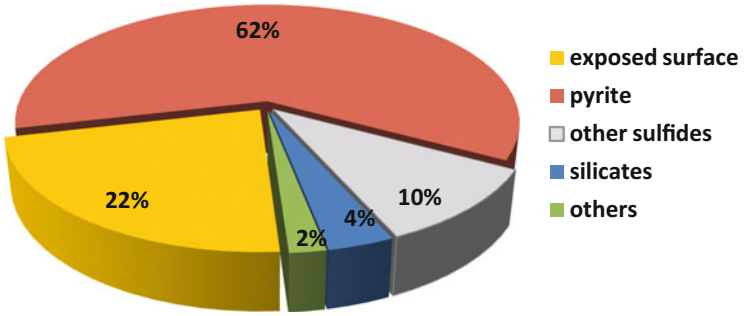


Fig. 2 Gold associations (% of perimeter of contact), sample SULF-HG

quantity of gold grains identified at the microscope (492 grains and 276 bearing particles—most particles contain more than one gold grain) is due to the very high grade of gold in heavy product in this sample (>30 ppm). On sulfide samples, the quantity of gold grains identified is also considerable, for both samples, with 17 and 2.3 ppm of gold in heavy products, respectively.

The main gold associations determined by the perimeter of contact with other minerals (gold carrier) are shown in Figs. 1, 2, and 3. An accurate ore characterization is essential in determining how an ore behaves in a traditional recovery circuit and this way to know the amenability of an ore to different process options

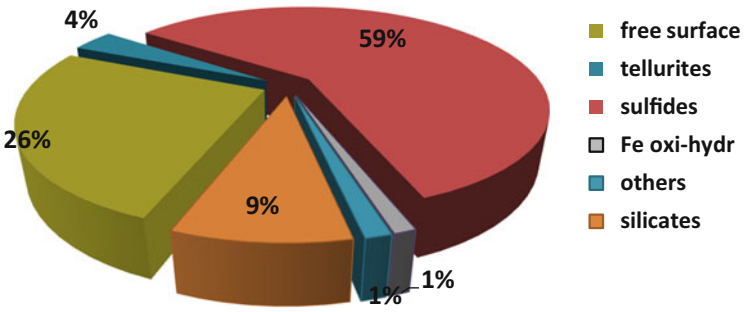


Fig. 3 Gold associations (% of perimeter of contact), sample SULF-LG

[2]. For example, flotation could be indicated if gold is exposed in particle's surface or locked in sulfide particles; cyanidation, if gold grains are very fine or associated with silicates; density concentration, if gold occurs in coarse grains; and so on.

For sample OXI-HG, the average gold association for the heavy products (−0.30 +0.037 mm) indicates that 39 % of the perimeter of gold grains is exposed in particle's surface. Gold grains in locked particles are essentially associated with iron hydroxides (29 %) and oxides (8.3 %) and secondarily to silicates and other oxides.

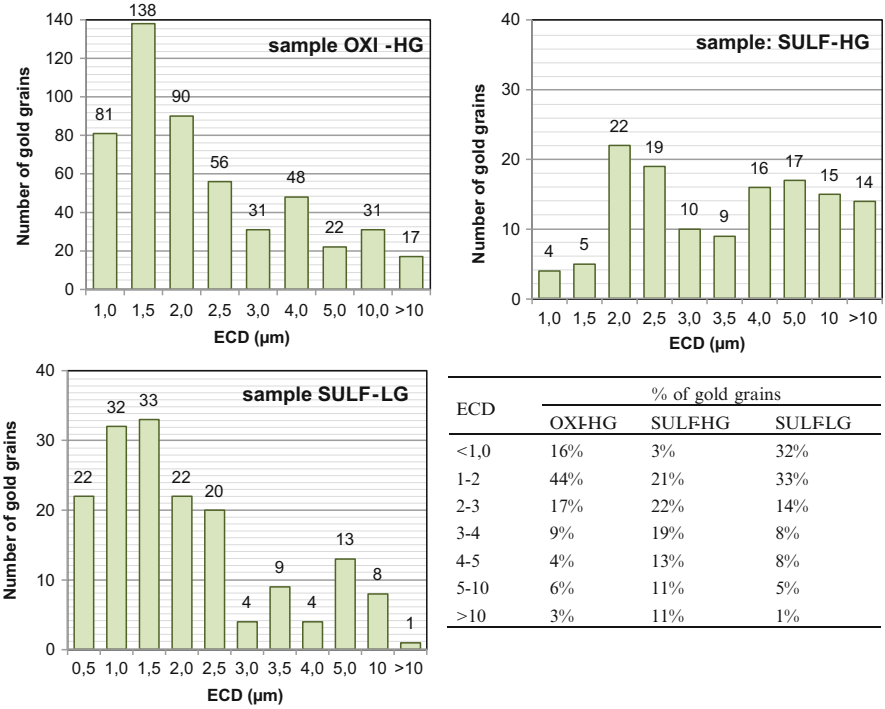
For sample SULF-HG, the average gold association for the heavy products (−0.30+0.037 mm) indicates that 22 % of the perimeter of gold grains is exposed in particle's surface. Gold in locked particles is essentially associated with pyrite (62 %) and secondarily to other sulfides (chalcopyrite—4 %, CuS—3 % and others—4 %), followed by silicates (4 %) and other minerals (2 %).

For sample SULF-LG, the average gold association for the heavy products (−0.30+0.037 mm) demonstrates the exposed gold grains correspond to 26 % of the total gold perimeter; the main association of the gold is with sulfides (59 %, mainly pyrite and more rarely pyrrhotite and chalcopyrite), followed by silicates (9 %), telluride (4 %), and other minerals (2 %). In this sample, a gold nugget is identified on fraction −0.074+0.037 mm, influencing the gold grain size distribution and exposed surface proportion.

3.3 Gold Grain Size

The size distribution of gold grains (absolute number of grains and frequency) is shown in Fig. 4, in terms of ECD (equivalent circle diameter).

For sample OXI-HG, it was found 466 grains of gold with ECD below 5 μm (91 % of the total grains identified), for sample SULF-HG, this amount corresponds to 78 % (102 gold grains); for sample SULF-LG (159 gold grains), 95 %. Due to the relative amount of fine grains of gold, it is fundamental that the characterization procedure enables the identification of them.



ECD	% of gold grains		
	OXI-HG	SULF-HG	SULF-LG
<1,0	16%	3%	32%
1-2	44%	21%	33%
2-3	17%	22%	14%
3-4	9%	19%	8%
4-5	4%	13%	8%
5-10	6%	11%	5%
>10	3%	11%	1%

Fig. 4 Gold grain size distribution

4 Discussion and Conclusions

This chapter compares the results of mineralogical and technological characterization of three samples with different grades of gold and origin. The density preconcentration step prior to MLA analysis demonstrates that the heavy products (submitted to MLA analysis) obtained for each fraction represent different mass recovery and depend on the composition of the sample.

Considering the enrichment of gold in sink product, a density preconcentration step can be carried out even in oxidized sample for mineralogical analysis, but it has to be lower than the density established for sulfide samples, taking into account the density of the separation, since the occurrence of gold is associated with other phases besides sulfides.

Methods for gold characterization and extraction have to consider the finer particles, since a huge amount of gold can be associated with fractions below 37 µm.

The extraction of gold by cyanide leaching in the light and intermediate product is similar to each sample; though the gold recovery varies significantly among the samples, indicating that the gold associations play a major role in gold recovery, regardless of the content of gold in the sample.

The higher recovery of gold by amalgamation in OXI-HG sample is in agreement with the higher proportion of free surface gold in this sample.

The identification of a large number of gold grains demonstrates the robustness of the established procedure on the search of gold by the MLA and also the effectiveness of the density separation on sample preparation.

Gold grains with ECD below 5 μm correspond to almost all gold in sample (78 %, 91 % and 95 %), so it is fundamental to establish a characterization procedure that enables the identification of them.

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References

1. Nuemann R, Schneider CL, Alcover Neto A (2002) Caracterização tecnológica de minérios. In: Technical communication prepared for the 3rd edition of Tratamento de Minérios, pp 81
2. Goodall WR, Scales PJ, Butcher AR (2005) The use of QEMSCAM and diagnostic leaching in the characterization of visible gold in complex ores. Miner Eng 18:877–886
3. Cabri LJ, Beattie M, Rudashevsky NS, Rudashevsky VN (2005) Process mineralogy of Au, Pd and Pt ores from Skaegaard intrusion, Greenland, using new technology. Miner Eng 18:888–897
4. Goodall WR, Scales PJ (2007) An overview of the advantages and disadvantages of the determination of gold mineralogy by automated mineralogy. Miner Eng 20:506–517
5. Gu Y (2003) Automated scanning electron microscope based mineral liberation analysis. An introduction to JKMRC/FEI Mineral Liberation Analyser. J Miner Mater Charact Eng 2(1):33–41
6. Chryssoulius SL, Cabri LJ (1990) Significance of gold mineralogical balances in mineral processing. Trans Inst Min Metall, Sect C 99(January–April):C1–C10