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GOLD PROVENANCE AND CHARACTERIZATION USING MICRO X-RAY FLUORESCENCE (μXRF): PRELIMINARY RESULTS

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Abstract

This work presents some preliminary results that allows to characterize gold samples using Micro X-Ray Fluorescence/μXRF. The first aim of this work is to apply a noninvasive technique, preserving the sample integrity, in order to identify the composition of gold samples and to recognize their possible geographical provenance. Samples have been obtained in geographically distinct gold-digging sites, from three Brazilian and one Colombian areas. These samples were processed only by fusion into a furnace at 1,200 ºC. The proportion of Au, Ag and Cu were measured in gold samples. The results of this work, allowed to characterize and to identify quite well the pure gold provenance, using μXRF instrumentation and related techniques. Further work is in progress to determine the behavior of mixed gold samples from different provenances. Besides that, measurements with different sample preparation will be made, in order to compare the results obtained in this work with those obtained by LA-ICP-MS techniques.

Keywords: Gold characterization. Gold provenance. μXRF. Brazil. Colombia. Gold-digging areas.

1. Introduction

Gold exploration in Brazil was the most important objectives of the ancient settlers, especially in the region of Minas Gerais, in the seventeenth and eighteenth centuries and in the early nineteenth century. After intense exploration, the deposits were almost depleted in that region, so that gold mining activities remained almost dormant until the middle of the 20th century, when the frontiers were enlarged and new regions began to be intensively explored. An emblematic example of this new expansion of mining activities is the mining of Serra Pelada (in the Pará State; Brazil). This region began to be explored in 1979 and since then more than 25 tons of gold have been mined.

The characterization of gold and the analysis of its provenance is a relevant topic because this information can be used to estimate the real production of gold mining in each region, helping to avoid smuggling and tax evasion. For this purpose, there are very precise and refined methods, but they are expensive, and require extended periods of time to perform tasks to get results. So, there is a need to establish a cheap methodology for determining the gold provenance and get results quickly.

Thus, the propose of this work is to develop and apply a low-cost experimental methodology to characterize and trace gold provenances, by measuring the gold, silver and copper contents present in each sample. Three Brazilian and one Colombian samples were tested.

Therefore, the main objective of this research was to verify the potentialities of a simple and economic analytic

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methodology to distinguish the origin of different samples of gold mining based on X-Ray Fluorescence results.

The X-Ray Fluorescence is extensively used to identify different metals and alloys. There are a number of XRF equipment, portable or not, which are largely used for this aim, which are described in detail by Gigante and Cesareo (1998) and Araujo et al. (2004).

1.1 The main goals

The main aim of this work is to develop a methodology is to develop a methodology for characterization and identification of pure gold provenance by applying μXRF techniques. In order to evaluate the potentialities of the proposed methodology, gold samples of well-known provenance were analyzed. These samples were from gold diggings at Cuiabá, Lavrinhas, Peixoto (Brazil) and Buriticá (Colombia).

2 Study Area

2.1 Cuiabá

The samples were extracted from the geological place Cuiabá - Poconé Gold Province, which is known from the 18^{th} century, when the Bandeirantes (flag holders) advanced in the so-called bandeiras (flags) crossed to the west and discovered large gold deposits in the region of Cuiabá (Pinho, 1990; Martinelli, 1999; Geraldes et al., 2008; Fig. 1). This period was known as the gold first cycle in Mato Grosso. After a long period of exploration, it was forgotten by the explorers, until the decade of 1980 when the price of the gold reacted as a commodity and took place a second gold cycle in Mato Grosso.

More than fifty small deposits and occurrences of gold are registered in this province, some of which are abandoned. The best known are the deposits of Casa de Pedra (Minérios Salomão SA), Jardim Italia, CPA, Mineiro, Jatobá and Abdala (Cuiabá and Várzea Grande), deposits of the Salinas and Chaves farms, Conceição and Adolfo Alemão (municipality of Nossa Senhora do Livramento), Pingo de Ouro, Adão, Cutia and São Rafael (District of Cangas), and deposits of Poconé, that add up at least 20 fronts of agriculture in the surroundings of this city.

Gold is associated with a system of quartz veins and disseminations in the Cuiabá Group, internal zone of the Paraguayan Band, in meta-sandstones, phyllite, meta-diamictites, marbles and meta-siltites. The mining areas are located in the hinge and NW flank zones of the Bento Gomes Anticlinal. Figure 1 presents a simplified geological map of the Paraguay Belt as presented by Sial et al. (2016)

Between 1982 and 1995, when a more effective control of the gold production by the municipality took place, this province produced about 26 tons of gold (official data). The data also show that the production peak occurred between 1990 and 1993, reaching 16.3 tons of gold. Production since 1999 has remained ≈2 ton/year, despite the decrease in the number of functioning companies.

2.2 Lavrinha

Lavrinha Gold Deposits is located in the Pontes e Lacerda region, SW of Amazonian Craton, Brazil. More than twenty gold deposits are known in the Pontes e Lacerda region situated around the Guaporé and Jauru rivers in the SW part of Amazonian Craton (Fig. 2), where gneiss and granulite of the basement outcrop, as well as granites of Rondonian mobile belt and rocks deformed during Sunsas-Aguapei mobile belt, the last accretionary event of the Craton. They are distributed along a NW striking, 40 km wide and more than 200 km long belt of deformation activated during the Middle Proterozoic Aguapé-Sunsas tectonic event. Cassiterite deposits, at north of this area, is another important resource of this region (Geraldes et al., 1997, 2004; Fernandes et al., 2003).

2.3 Peixoto de Azevedo

The Alta Floresta Aurífera Province is located at north of Mato Grosso State (Brazil) and includes the Paraíba and Santa Helena deposits, located near the municipalities of Peixoto de Azevedo and Nova Santa Helena, respectively.

This study area is related to the development of successive magmatic arches during the Paleoproterozoic, called Cuiú-Cuiú (2.10 to 1.85 Gyr) and Juruena (1.85 to 1.75 Gyr) (Tassinari et al., 2000; Geraldes et al., 2001). These works performed the petrographic description and isotopic lead studies of mineral areas, aiming to characterize the deposits typology and genesis.

In Santa Helena and Paraíba deposits, the gold occurs in quartz veins. In Santa Helena, the veins are embedded in granite and, in the Paraíba deposit, they are included in orthogneisses and basic metavolcanics rocks. Both have in common the presence of pyrite, chalcopyrite, tetradimite, chalcocite, magnetite, hematite, as well as quartz, K-feldspar and sericite. The mineralization of the Santa Helena deposit is related to ounces of gold (production to date more reserves; Sillitoe, 2008).

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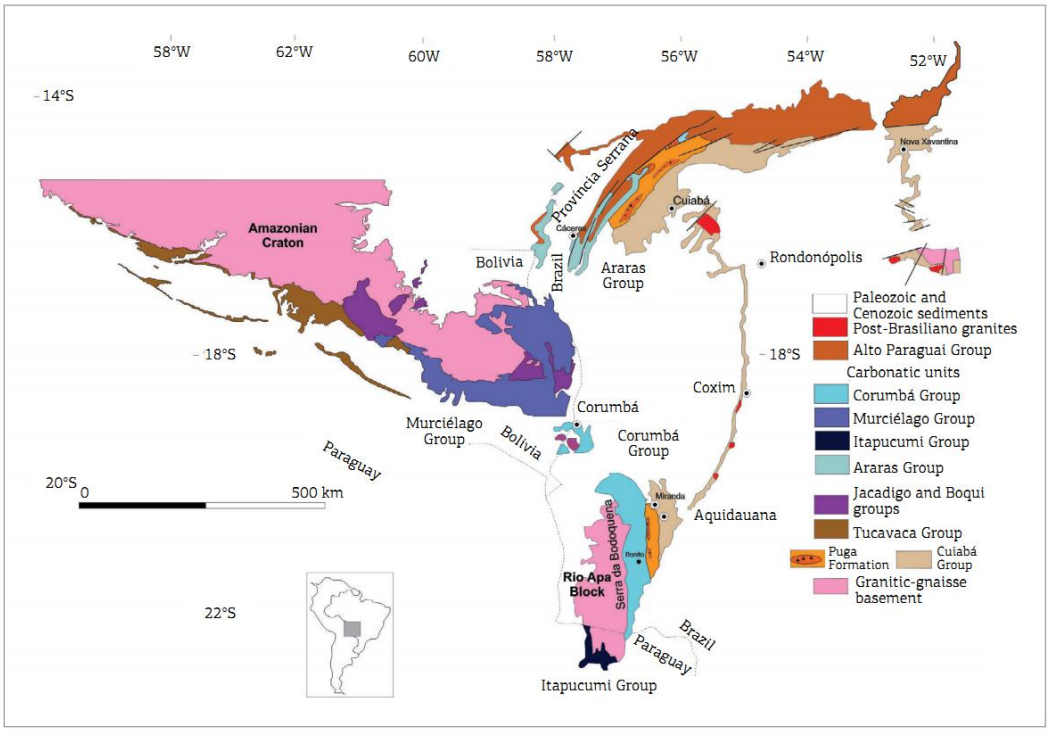

Fig. 1. Simplified geological map of the Paraguay Belt showing the areas of outcrop of the Araras, Cuiabá, Corumbá, Itapucumi and the Murciélago groups (from Sial et al., 2016).

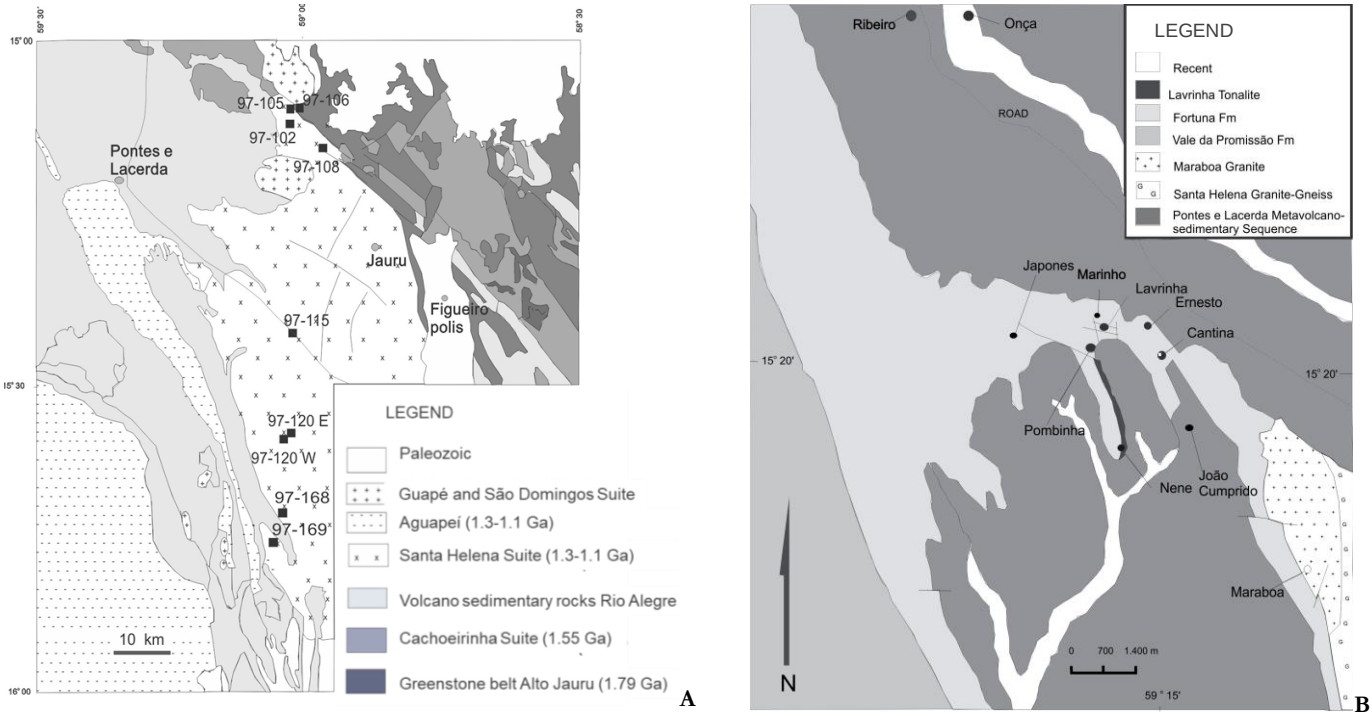

Fig. 2. A. Regional map with the most important stratigraphic units of SW of Amazonian Craton in Mato Grosso State. B. Detail of Fig. 2A showing Lavrinha and other deposits. Adapted from Geraldes et al. (1997).

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The "Ancient Granite" is intruded by the "Young Granite" of this region. Auriferous quartz veins occur sub-parallel to a shear zone. In the "Ancient Granite", the veins are deformed and occur along the shear zone. The host pyrite of gold is stretched in the direction of shear. The veins and venules of the new granite show less deformation and do not follow any preferential orientation.

The isotopic Pb data of the pyrites show that the upper crust was the main geodynamic environment for the deposit mineralization formation. Pyrite of the deposit has an age of about 1,930 Myr and the granite an age of ≈1,967 Myr and displays the temporal and genetic relationship of the mineralization with the Santa Helena "Young Granite". The association Au-As-Pb-Bi-Cu allows to infer that it is a granite of a magmatic arc, based on orogenic deposits related to intrusions.

In the Paraíba deposit the auriferous quartz veins are embedded in granitoid milonites or near their contact with less deformed tonalites and amphibolites. The sulfides in the mineralized zone occur in the hypidiomorphic and idiomorphic forms, with shadow of pressure, which denotes a pre- or early-shear mineral growth. The isotopic data of Pb in the pyramids of the Paraíba reservoir suggest that the origin of the hydrothermal fluids is associated to a mixture between mantelic and crustal sources.

The pyrite age (1,809±40 Myr) of the Paraíba deposit is temporally related to the host rocks of the mineralized shaft and show that the deposit is late in relation to evolution of the Cuiú-Cuiú magmatic arc. The Paraíba deposit resembles the Orogenic gold deposits model and has characteristics of the lode-gold type. Both studied deposits show great similarity in mineralogy, hydrothermal alteration and the occurrence of gold within the mineralized zone. The different geological types observed in both deposits demonstrate that the occurrence of gold in the Alta Floresta Aurífera Province is related to orogenic processes.

2.4 Colombia

The Buriticá gold deposit is located on the eastern flank of the Western Cordillera of Colombia (Fig. 3). The Andean orogeny is described as a typical Cordilleran type system, formed by the subduction of oceanic lithosphere below the continental margin (Irving, 1975). Colombia's gold deposits represent a large fraction of the known gold mineralization in the Andes, with over 50 million ounces of gold (production to date plus reserves and measured and indicated resources; Sillitoe, 2008).

Neogene deposits are located in the Middle Cauca Belt close to the Cauca-Romeral fault system, and are related to subduction magmatism associated with approach of the Baudó terrane during the Miocene. The Middle Cauca Belt contains epithermal as well as porphyry Au deposits, and includes the Buriticá gold deposit, located toward its northern end. Several structural features have been identified in the Buriticá area, some of which controlled gold mineralization. An early folding episode has deformed the rocks of the Barroso Formation, and by asymmetric parasitic folds observed in mudstone units.

The Buriticá deposit is currently being mined on a small scale at the Yaragua mine, which exploits high-grade gold-bearing quartz-carbonate veins (the Murcielagos, San Antonio, and Centena veins) hosted by the Buriticá andesite, and produced about 331.5 kg of gold from 2001 to 2007 (Lesage, 2011; Guillaume et al., 2013).

Gold mineralization is hosted by two different sets of veins with slightly different orientations. The largest and most strongly mineralized vein set includes the San Antonio and Murcielagos veins. The second vein set includes the Centena vein. A total of 14 individual veins have been identified in the Yaragua area, of which the two bigger and better defined are the Murcielagos and San Antonio veins. Vein mineralogy is characterized by quartz and calcite, with variable amounts of chalcopyrite, galena, pyrite, sphalerite, tetrahedrite/tennantite, stibnite, and minor native gold or electrum. Geochronological $^{40}Ar/^{39}Ar$ analyses using step-heating methods in hornblende yielded a plateau age of 7.59±0.16 Myr. Two samples of muscovite from the alteration halo around the mineralized zones yielded similar ages, with well-defined plateaus at 7.73±0.12 Myr and 7.74±0.10 Myr.

2.5 Additional information about the areas of the samples origin in Brazil

The Cuiaba gold deposits are characterized by a strong correlation between deformation of country rocks and the ore minerals. With the available data, it is not possible to define if the metal concentration in Cuiabá deposit was related to the Neoproterozoic (≈540 Myr) evolution of the Paraguai mobile belt and the magmatic or linked to shearing and fluid remobilization processes during the orogen evolution, recorded at about 543-520 Myr 40Ar/39Ar cooling ages in biotite.

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The Pb isotope age model of the Peixoto de Azevedo deposit are roughly coeval to the 1,762-1,755 Myr U-Pb LA-ICP-MS ages of the volcanic-plutonic rocks of the Ata Floresta Gold Province cratonic. The granitic intrusions with ≈1.80 Ga accompanied by hydrothermal solutions deposition suggests an orogenetic origin for Peixoto de Azevedo gold deposit.

In Lavrinha gold deposits, the ore formation is correlated with the movement of the hydrothermal fluids during the Aguapei tectonic event. The mineralization with ≈ 920 Myr is coeval with the deformation and the hydrothermal solutions percolating through older granitic (1.45-1.42 Gyr Santa Helena) and basic (1.51-1.49 Gyr Rio Alegre) and sedimentary rocks (Aguapei Group).

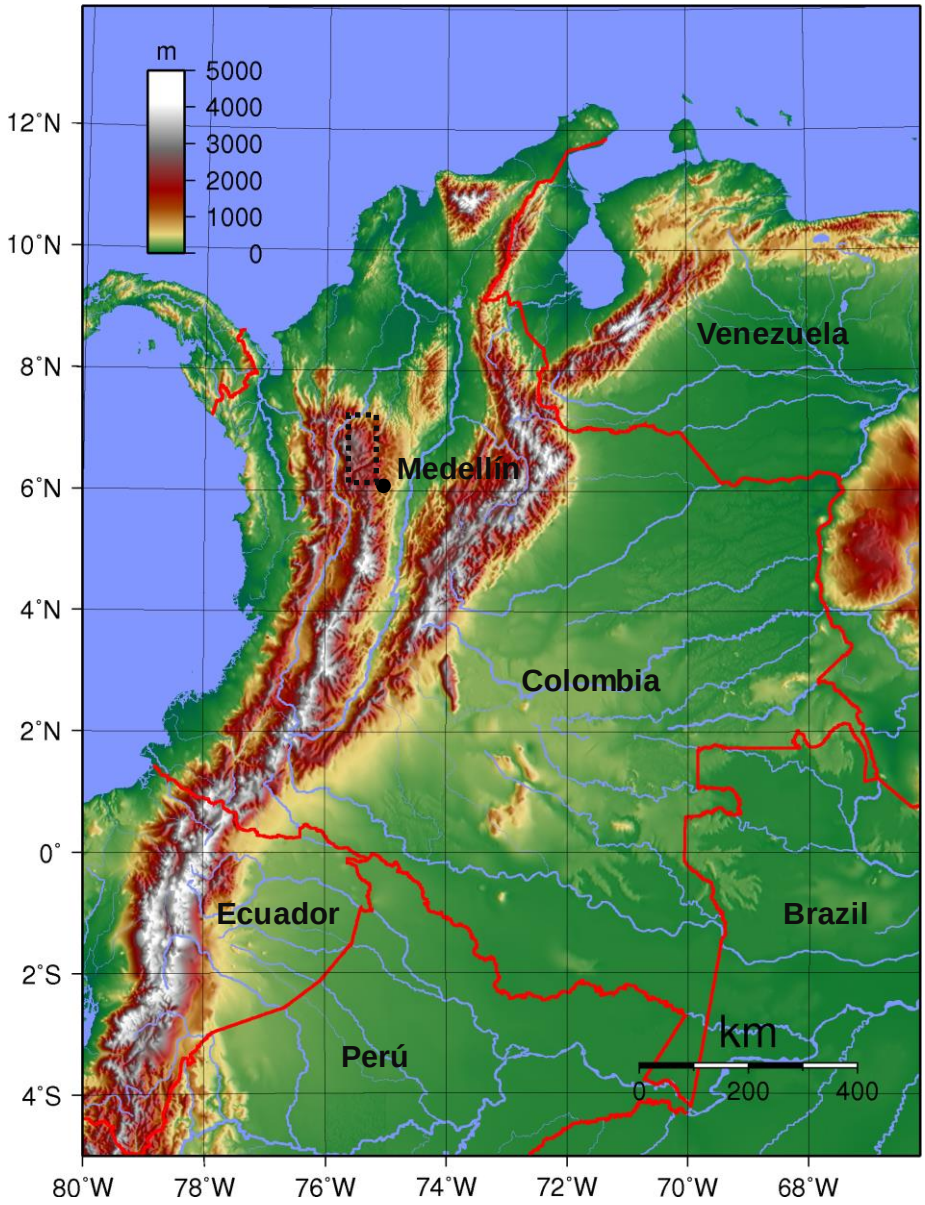

Fig. 3. Location of Colombia in South America, with Medellin in the Andes Mountains; the rectangle surrounds the region of interest, at northwest of Medellin (adapted from Wikimedia Foundation, Inc.).

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3. Material and methods

Five samples were obtained in places where gold is being extracted, at Cuiabá, Lavrinha, Peixoto de Azevedo (Brazil) and Buriticá (Colombia). Unprocessed gold was bought in the form of powder or small nuggets. This option was taken because in processed gold there is a risk of finding a mixture of gold from quite different sources, increasing the dispersion of the measures and getting erratic values. In addition, very often the gold processed in Brazil contains small amounts of mercury, but these amounts are still enough to interfere with the measurements. The gold samples were placed in an alumina crucible and melted in an oven at 1200ºC for one hour. After cooling, the samples were cleaned with concentrated nitric acid PA and washed with deionized water. Subsequently, the samples were cut into smaller pieces and mounted in epoxi resin for sanding and polishing. This procedure allows to examine samples with an optical or electronic microscope.

To determine the concentrations of gold, silver and copper in the samples, the XRF technique was used. It should be remembered that any methodology will be valuable only if it is possible to differentiate geographically distinct samples easily. So, the purpose of our procedures was to improve the measurements and interpretation of data in such a way that our main goal was reached.

X-ray fluorescence spectrometry (XRF) is a well-established non-destructive analytical technique for qualitative and quantitative element analysis of a wide variety of samples. XRF is largely used to obtain qualitative and quantitative information on the elemental composition of several types of samples. Among its many features, it allows to analyze multi-element concentrations with acceptable rapidity and economy, ease of automation and can analyze solid samples.

When an element of a sample is excited with an X-ray beam, it tends to eject electrons from its innermost orbitals. Thus, electrons from the outer levels perform a quantum transition to fill the vacancy left by the released electron. Each electron transition constitutes a loss of energy for the electron. This energy, which is well defined, is emitted in the form of characteristic X-rays and represents the signature of the chemical element in the studied sample.

Since the 1960's, the XRF has been used for non-destructive analysis of geological and archaeological materials. Although recently XRF applications have been obscured by other instrumentation such as LA-ICP-MS, XRF remains one of the cornerstones of non-destructive chemical analysis in both geology and archeology, namely for volcanic rocks and more particularly for obsidian.

During the last decade, the X-ray fluorescence technique has evolved from the dependence on laboratory units to the field use of portable and light devices. These portable instruments have given to researchers in conservation of art and archeology the opportunity to study a wide range of materials with greater accessibility and flexibility than ever before. The recent development and commercialization of benchtop and portable instrumentation, offering extreme simplicity of operation in a low-cost design coupled with high measurement throughput, have extended XRF applications to many other analytical problems.

In addition, the low relative cost of handheld XRF has led many museums, academic institutions, and cultural centers to invest in the devices for routine materials analysis purposes. Although these instruments often greatly simplify data collection, proper selection of analysis conditions and data interpretation still require an understanding of the principles of x-ray spectroscopy. These instruments are often marketed and used as "point-and-shoot" solutions; however, their inexpert use can easily generate deceptive or erroneous results.

The tube excites an element of a sample with an X-ray beam, which tends to eject electrons from its innermost orbitals. Thus, electrons from the most distant levels perform a quantum transition to fill the vacancy left by the released electron. Each electron transition constitutes a loss of energy for the electron. This energy, which is well defined for each element, is emitted in the form of a characteristic X-ray and represents the signature of the chemical element in the studied sample that is captured by the detector.

The calibration of the equipment was done using 24, 18, 16, 14, 12 and 10 karat certified gold standards from the Polytechnic Institute of the Universidade do Rio de Janeiro, UERJ. The standards were prepared with 1.0 cm^2 area, ≈1.0 mm thick and with Au-Cu and Au-Ag metal alloys.

For more information about this methodology see, for example, Verma (2007), Margui and van Grieken (2013), Shackley (2012) and Shugar (2014). According to this methodology, the resulting data are normalized and presented in percentage after being calibrated with the data obtained by the standards. After this step, they were exported to Microsoft Excel™ for statistical treatment and to preform convenient charts.

In order to proceed the characterization of copper, gold and silver in each sample, the facilities of the Electronic Instrumentation and Analytical Techniques Laboratory were used (LIETA - Laboratório de Instrumentação Eletrônica e Técnicas Analíticas). The samples previously mounted in epoxi resin were cleaned with ultra-pure Mili-Q water and placed in a BRUKER portable commercial equipment to obtain the XRF data.

A typical example of results is like that shown in Fig. 4, which presents a graph with a number of the detector counts in one hundred seconds for each characteristic X Ray line.

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This equipment (model ARTAX 200) features Mo anode tube, low power and XFlash SDD detector with advanced technology for accurate fast data acquisition and has an integrated camera and a laser in order to positioning the sample.

The following configurations were used as a standard for the measurements: Excitation voltage 40 kV; Beam current 250 µA; Acquisition time 300 s; Al filter thickness 315 µm. Five measurements of each sample were obtained. The spectra of each µXRF analysis were acquired (Fig.4) and evaluated by the SPECTRA software, provided by the equipment manufacturer, where the fluorescence peaks of each element in sample are evidenced. The concentrations of Au, Cu and Ag were then measured in the investigated samples.

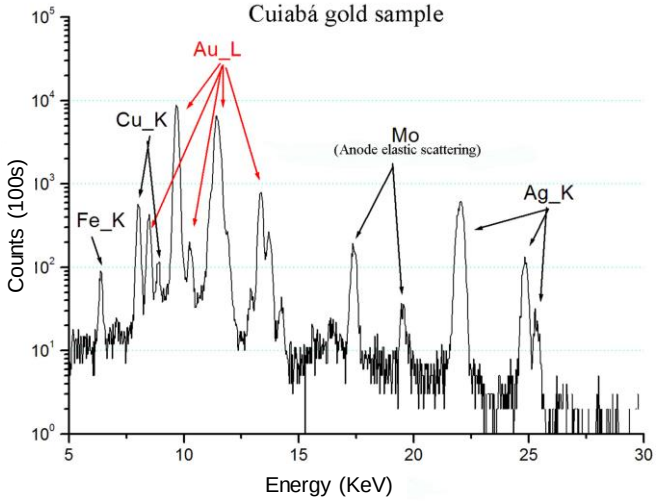

Fig. 4. Typical results from X-ray fluorescence showing X-ray L lines for gold (in red).

Each sample was subjected to five measurements at different points along the surface of the sample. The fluorescence values for Au, Ag and Cu obtained at each point were recorded for each sample. These raw data were treated in order to be compared with those from different samples.

4. Results

Concentrations of Au, Ag and Cu for each point are synthetically presented in Table 1 and graphically displayed in Fig. 5A-C. The mean concentrations of these elements were in: Peixoto for Au 50.3±0.2 %, Cu 1.56±0.02 % and Ag 48.2±0.2 %; Lavrinha for Au 44.6±0.2 %, Cu 10.6±0.07 % and Ag 44.7±0.2 %; Cuiabá for Au 53.7±0.5 %, Cu 1.8±0.08 % and Ag 44.5±0.5 % and; Buriticá (Colombia) for Au 60.5±0.1 %, Cu 0.023±0.09 % and Ag 39.5±0.1 %.

These values and the plots of Au, Ag and Cu concentrations for each local (Fig. 5A-C) show that: the Brazilian samples have higher proportion of Cu and Ag than the Colombian one which in turn have the highest proportions of Au. The results presented in these plots evidence values scattering depending of the gold samples provenance.

The Au/Cu values were plotted against Ag/Cu, for each sample of the studied areas (Fig. 5D). The Au/Cu and Ag/Cu ratio values (Table 1) show clear differences in the composition of gold in each studied region. These differences are also evident in the graphs of Fig. 5A-C, which show clear differences among gold, silver and copper percentages of the samples with different provenance.

The plot of Au/Cu against Ag/Cu values shows that the samples of Peixoto, Lavrinha and Cuiabá form three groups, separated from each other. This graph analysis also allows distinguishing the different gold sources. The best precisions are for those from Lavrinha and Peixoto samples, therefore these are the better characterized ones followed by those from Cuiabá. The Colombia samples show a large dispersion among values. These samples have from hundred to thousand times lower concentrations of Cu than those found in Brazilian samples (see Table 1).

The data dispersion in Colombia samples should be influenced by the low Cu contents. Due to the low concentrations of Cu, the XRF begins to present increasing errors, which rises the fluctuations in the Au/Cu and Ag/Cu values. Another source of this dispersion is related to the inhomogeneous characteristics of each sample of this region.

Nevertheless, samples of each region are easily distinguished by their composition and present a characteristic composition, which prevents any misleading about their provenance.

A certain dispersion of the measurements can be observed for the samples of each local in every graph. The main hypothesis to explain this fact is the inhomogeneity of the samples. These samples are basically nontreated or little treated, at most by fusion. By analyzing samples with metallographic procedures, we expect to demonstrate the occurrence of a segregation of silver nodules surrounded by gold or by a matrix of gold and silver alloy. In order to start a geological interpretation about the formation of these gold/silver nuggets, it is necessary to obtain more samples with compatible dimensions for a metallographic investigation. This is the next step of this research. However, the graph of Au/Cu against Ag/Cu (Fig. 5D) shows that the samples from Peixoto, Lavrinhas and Cuiabá have much more similar composition and are quite well separated by their provenances.

There is still much work to do in order to validate this procedure of characterization of the gold provenance. The improvement of this methodology requires the analysis of a larger number of samples and study of a larger number of mining camps and their geographical settlement

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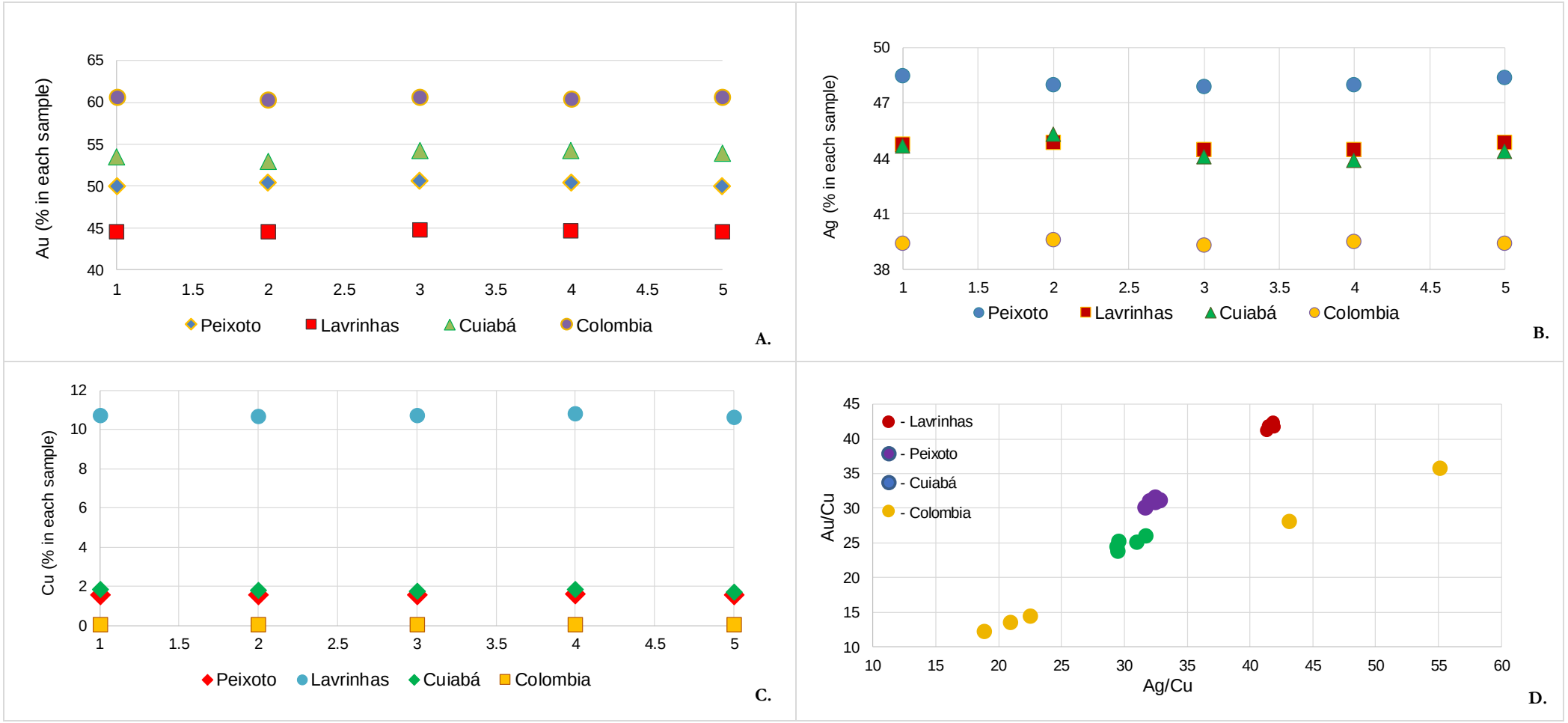

Fig. 5. Geochemical results of the analyzed samples of Peixoto, Lavinhas, Cuiabá (Brazil) and Colombia: A. Gold percentage. B. Silver percentage. C. Copper percentage. D. – Au/Cu versus Ag/Cu plot (in Lavinhas the real values are x10 and in Colombia the real values are x10^2; see Table 1).

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Tab. 1. Obtained results in the analyzed samples of Peixoto, Lavinhas, Cuiabá (Brazil) and Colombia.

PEIXOTO	Au (%)	Cu (%)	Ag (%)	Au/Cu Ratio	Ag/Cu Ratio
1	50.000	1.540	48.500	32.468	31.494
2	50.400	1.550	48.000	32.516	30.968
3	50.600	1.540	47.900	32.857	31.104
4	50.400	1.590	48.000	31.698	30.189
5	50.000	1.560	48.400	32.051	31.026
Average	**50.300**	**1.560**	**48.200**	**32.244**	**30.897**
Standard Deviation	**0.200**	**0.020**	**0.200**		
Coefficient of variation	**0.005**	**0.010**	**0.005**		

LAVRINHAS	Au (%)	Cu (%)	Ag (%)	Au/Cu Ratio	Ag/Cu Ratio
1	44.500	10.710	44.800	4.155	4.183
2	44.500	10.630	44.900	4.186	4.224
3	44.800	10.670	44.500	4.199	4.171
4	44.700	10.800	44.500	4.139	4.120
5	44.500	10.620	44.900	4.190	4.228
Average	**44.600**	**10.690**	**44.700**	**4.172**	**4.181**
Standard Deviation	**0.100**	**0.070**	**0.200**		
Coefficient of variation	**0.003**	**0.007**	**0.004**		

CUIABÁ	Au (%)	Cu (%)	Ag (%)	Au/Cu Ratio	Ag/Cu Ratio
1	53.500	1.820	44.700	29.396	24.560
2	52.900	1.790	45.300	29.553	25.307
3	54.200	1.750	44.100	30.971	25.200
4	54.200	1.840	43.900	29.457	23.859
5	53.900	1.700	44.400	31.706	26.118
Average	**53.700**	**1.780**	**44.500**	**30.169**	**25.000**
Standard Deviation	**0.500**	**0.060**	**0.500**		
Coefficient of variation	**0.010**	**0.030**	**0.010**		

COLOMBIA	Au (%)	Cu (%)	Ag (%)	Au/Cu Ratio	Ag/Cu Ratio
1	60.600	0.029	39.400	2089.655	1358.621
2	60.300	0.032	39.600	1884.375	1237.500
3	60.700	0.027	39.300	2248.148	1455.556
4	60.400	0.014	39.500	4314.286	2821.429
5	60.600	0.011	39.400	5509.091	3581.818
Average	**60.500**	**0.023**	**39.500**	**2630.435**	**1717.391**
Standard Deviation	**0.100**	**0.009**	**0.100**		
Coefficient of variation	**0.002**	**0.400**	**0.003**		

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6. Conclusion

This work presents some preliminary results aiming to characterize pure gold, only fusion processed. The Au, Ag and Cu signatures determined by XRF allowed to distinguish different geologic sources for metallic deposits. The results presented in this work define important parameters for regional metallic exploration in SW Amazonian craton and in other places like it was made to a sample of Colombia. A subsequent investigation should be made analyzing samples from more provenances and increasing the number of samples extracted from each area.

Interestingly, the measurements showed a dispersion between the relative concentrations of Au and Ag for the same sample. This seems to point to an inhomogeneous distribution of Ag and Au in those samples, even though they have been fused. Apparently, the resulting alloys do not appear to have been perfectly homogeneous as would be expected from an Au/Ag alloy. A more detailed study is being done to better understand what is happening. This is particularly important when samples are not processed, that is, they are raw material from mining camps, like small nuggets. This aspect requires a more detailed investigation using metallographic techniques and/or an electron micro probe analyzer (EMPA).

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