Revista Brasileira de Geografia Física v.11, n.03 (2018) 934-947.

ISSN:1984-2295

Revista Brasileira de
Geografia Física

Homepage: www.ufpe.br/rbgfe

Preliminary Results in Gold Provenance Characterization using LA ICP MS

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Artigo recebido em 13/11/2017 e aceito em 30/03/2018

ABSTRACT

We present some preliminary results of Pb isotopes characterization of gold samples.The first aim of this work is to apply an archeometry technique, that is, geographical origin characterization of ancient artifacts in order to characterize gold provenance. Using Pb isotopes in gold samples in which that metal is found as a trace is possible using ICP MS techniques. In this work was also used laser ablation to extract material from the sample.Samples have been obtained from three brazilian geographically distinct gold-digging sites. The samples were processed only by fusion into a furnace at 1,200 ºC. Laser ablation and analysis by aa ICP MS was performed. We have measured Pb isotopes at ppb levels and have derived a few relations among them and made some graphics. In those graphics one can see the measured relations are characteristic fingerprints of each single site. Therefore, the pure gold provenance can be quite well characterized using mass spectrometry instrumentation and its techniques. Further work is in progress to determine the behaviour of mixed gold samples from different provenances. Besides that, measurements with different sample preparation will be made, in order to compare results with laser ablation extraction.

Resultados preliminares na caracterização da proveniência de ouro usando LA ICP MS

RESUMO

Neste estudo são apresentados alguns resultados preliminares para caracterização de amostras de ouro pelos isótopos do chumbo presentes na amostra. O primeiro objetivo deste trabalho é aplicar uma técnica de archeometria, isto é, caracterização de origem geográfica de artefatos antigos para caracterizar a proveniência de ouro. O uso de isótopos Pb em amostras de ouro em que esse metal é encontrado como um rastreio é possível usando técnicas de ICP MS. Neste trabalho também foi utilizado ablação a laser para extrair material da amostra. As amostras foram obtidas de três locais de garimpo de ouro geograficamente distintos do Brasil e de um na Colômbia. As amostras foram processadas apenas por fusão em um forno a 1.200 ºC. A extração por ablação a laser e análise por espectrometria de massas usando um ICP MS foi realizada. Foram medidos os isótopos de chumbo em níveis de partes por bilhão (PPB), foram derivadas relações entre eles e apresentadas em gráficos. Nesses gráficos, pode se ver que as relações medidas são muito características ("impressões digitais") de cada sítio. Portanto, a proveniência do ouro puro pode ser bastante bem caracterizada usando a espectrometria de massa e suas técnicas. Outras pesquisas estão em andamento para determinar o comportamento de misturas de amostras de ouro de diferentes procedências. Além disso, serão feitas medições com diferentes métodos de preparação das amostras, para comparar os resultados obtidos com a extração por ablação laser.

Introduction

Using Pb isotopes ratios in ancient artifacts is an overspread technique to study their geographical provenance provenance (Junk and

Pernicka, 2003;Ponting, Evans and Pashley, 2003; Pinarelli, 2004; Bendall 2009; Cattin et al, 2009; Stos-Gale and Gale, 2009; Wilson and Friedman, 2010; Klein et al, 2012; Albarede et al, 2012). This is quite true for Europe and Middle Eastern because there is a large database which provides information on the mines of the Roman empire, principally those producing the main metals used in the production of coinage: gold, silver and copper. Since many silver mines also produced lead from the same ores, many lead mines are listed, and some iron mines have also been included, although the coverage of lead and iron mines cannot yet be regarded as systematic. Furthermore, Pb is a largely spread contaminant of many ancient and new artifacts besides raw materials. Nevertheless, the central question was to know if raw non processed gold would have a detectable amount of Pb in order to evaluate isotopes ratios. The first tests have shown that our equipment and techniques were quite suited to make the intended measurements. Therefore, we have proceeded to analyse three gold samples of well known provenance. Those are from gold diggings at Cuiabá, Lavrinhas and Peixoto de Azevedo.

Materials and methodology

The gold samples were obtained in the places where gold is extracted. We chose to purchase unprocessed gold because in the case of a processed one there is a risk of finding a mixture of gold from quite different sources, increasing the dispersion of the measures and providing erratic results. The samples were acquired in the form of powder or small nuggets. In addition, often gold processed in Brazil contains mercury in small amounts, but these amounts are still sufficient to hide or mask Pb isotope signals preventing their measurement.

Cuiabá

These samples were extracted from the geological place Cuiabá - Poconé Gold Province, which is known from the 18th 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). This period was known as the gold first cycle. 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. In this province are registered more than fifty small deposits and occurrences of gold, 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*Rafae*l (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 CuiabáGroup, internal zone of the Paraguayan Band, in metarenites, filitos, metadiamictitos, marbles and metassiltites. The mining areas are located in the hinge and NW flank areas of the *Bento Gomes* Anticlinal. Between 1982 and 1995, when more effective control of the production of gold by municipality was obtained, it is observed that this province produced around 26 tons of gold (official data). The data also show that the peak in production occurred in the period between 1990 and 1993, reaching 16.3 tonnes of gold. Production since 1999 has remained around 2 ton year, despite the decrease in the number of enterprises in operation.

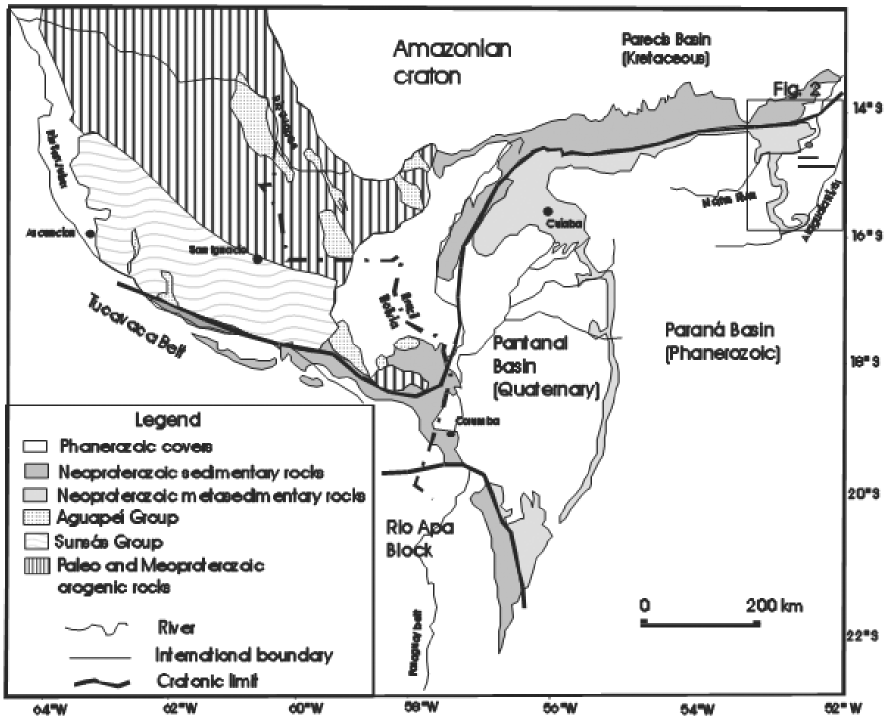

Figure 1: General overview of the Paraguay belt. The rocks of the Paraguay belt may be divided in undeformed sedimentary rocks (cratonic cover) and deformed sedimentary rocks, corresponding to the orogenic external and internal zones, respectivelly.

Lavrinhas

Lavrinha Gold Deposits 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. 1), where outcop gneiss and granulites of the basement, granites of Rondoniano mobile belt and rocks deformed during Sunsas-Aguapei mobile belt, the last acrecionary event to the Craton. They are distributed along a NW striking, 40 km wide and more than 200 km long belt of deformation actived during the Middle Proterozoic Aguapé-Sunsas tectonic event. Cassiterite deposits in the north of this area are another important resource in the region (Geraldes et al, 1997; Fernandes et al, 2003;Geraldes, Teixeira and Heilbron, 2004).

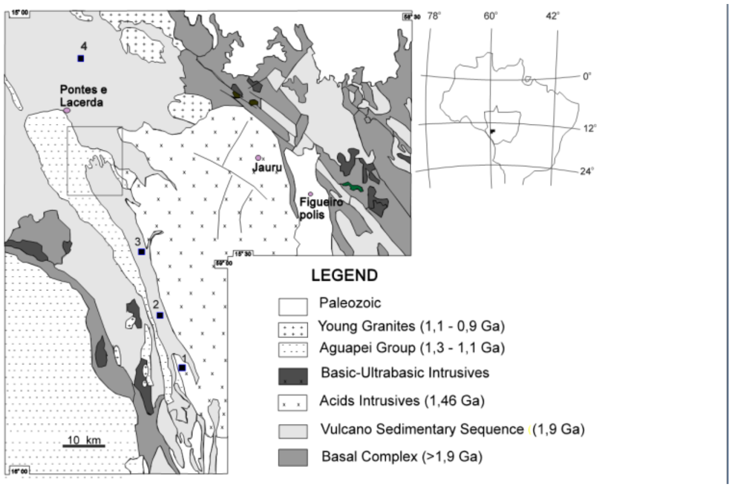

Figure 2 - Regional map with most important stratigraphic units of SW of Amazonian Craton in Mato Grosso State. Black squares: 1- Ellus deposit; 2- Mineiros deposit; 3- Pau-a-Pique deposit; and 3- Incra deposit. Details of rectangle area are shown in Fig. 3.

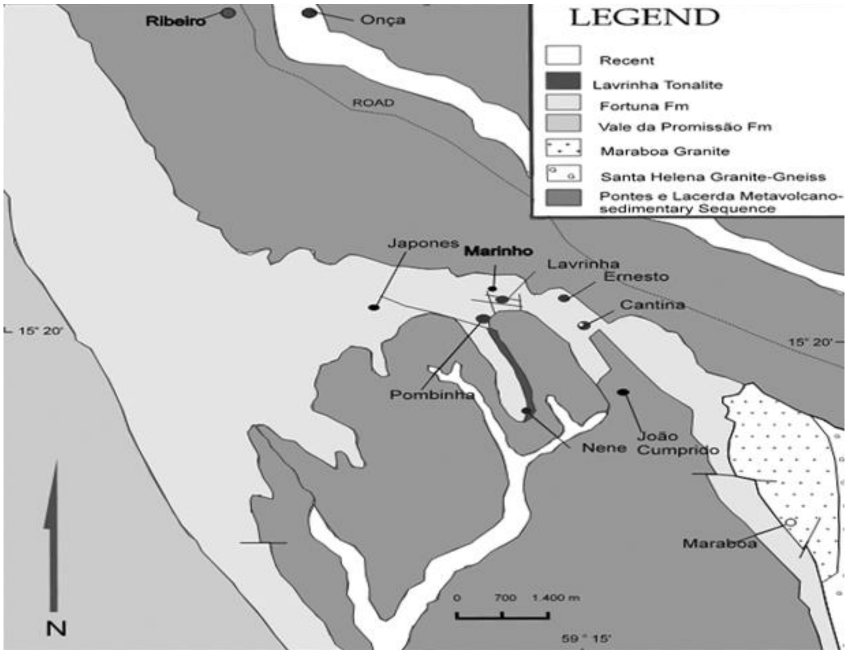

Figure 3 - Detail of Fig. 2 showing Lavrinha and other deposits

Peixoto de Azevedo

The Alta Floresta *Aurífera* Province is located in the north of the State of Mato Grosso and includes the Paraíba and Santa Helena deposits, located near the municipalities of Peixoto de Azevedo and Nova Santa Helena respectively. The area studied is related to the development of successive magmatic arches during the Paleopreterozoic, called Cuiú-Cuiú (2.10 to 1.85 Gy) and Juruena (1.85to 1.75 Gy) (Tassinari et al, 2000; Geraldes et al, 2001). The objective of the refered articles was the petrographic description and isotopic studies of lead of mineral areas, with the aim of characterizing the typology and the genesis of deposits. In the Santa Helena and Paraíba deposits the gold occurs in quartz veins. In St. Helena the veins are embedded in granite, in the Paraíba deposit in ortognaisses, and basic metavulcanics. 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 St. Helena deposit is related to the granite St. Helena Ancient that is intruded by the St. Helena Young. Auriferous quartz veins occur sub-alleles to a shear zone. In the old granite the veins are deformed and occur along the shear zone. The host pyrite of gold is stretched in the direction of shear. No granite New veins and venules 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 formation of deposit mineralization. The pyrite of the deposit has an age of about 1,930 My and the granite age around 1,967 My shows the temporal and genetic relationship of the mineralization with the Santa Helena Jovem Granite. The association Au-As-Pb-Bi- Cu allows to infer a granite of 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 form the idiomorphic, 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 mantleic and crustal sources; With main part of the upper crust. The pyrite age (1,809±40 My) of the Paraíba deposit are 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 characteristics of the lode-gold type. The two deposits studied show great similarity in mineralogy, hydrothermal alteration and the occurrence of gold within the mineralized zone. The different geological types observed in the two deposits demonstrate that the occurrence of gold in the *Alta Floresta Aurífera* Province is related to orogenic processes.

Figure 4: Location map of the main municipalities and highways of the state of Mato Grosso, highlighting the municipalities of Santa Helena and Peixoto de Azevedo where the gold deposits are located.

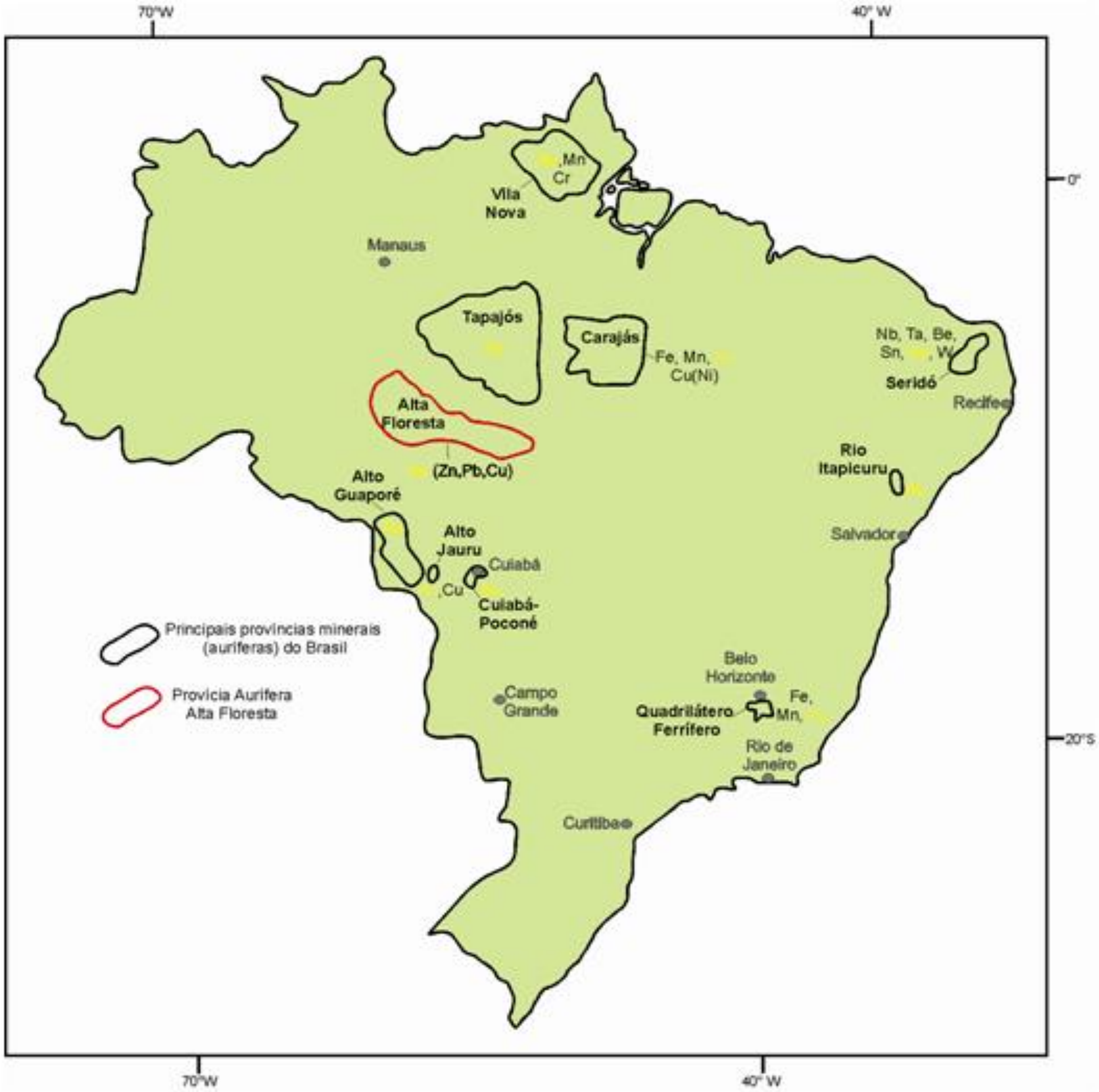

Figure 5: Main gold provinces of Brazil, modified from Dardenne Amp; Schobbenhaus (2000). Highlighted is the province of Alta Floresta, where the Paraíba and Santa Helena deposits are inserted.

Colombia

The Buriticá• gold deposit is located on the eastern flank of the Western Cordillera of Colombia. The Andean orogeny is described as a typical Cordilleran type system, formed by the subduction of oceanic lithosphere below a 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 Baudó³ 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 11,694 ounces 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 to date 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 hornblend yielded a plateau age of 7.59±0.16 My. Two samples of muscovite from the alteration halo around the mineralized zones yielded similar ages, with well-defined plateaus at 7.73±0.12 My and 7.74±0.10 My.

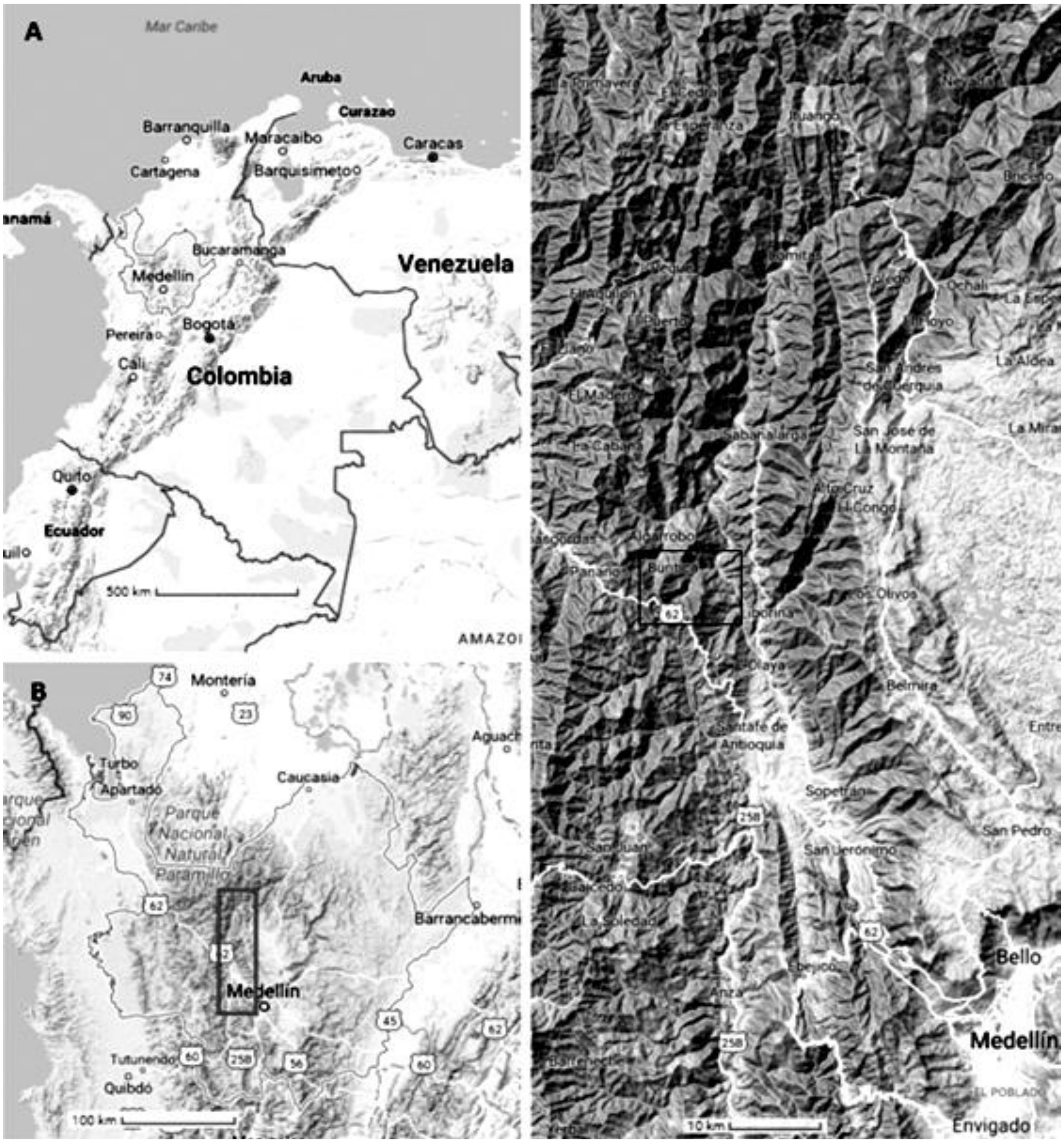

Figure 6 - Left side: A (top) - Location of Colombia in South America, with Medellin in the Andes Mountains; B (bottom) - Rectangle surrounding the region of interest, northwest of Medellin; Right side: Magnification of the rectangle shown in B, topographic map of the mining region.

Methods

The gold samples were placed in an alumina crucible and melted in an oven at $1200^{o}C$ remaining at this temperature 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 resin for sanding and polishing. This permits one to examine samples with a microscope, optical or electronic. The polishing of the samples facilitates also the laser ablation procedures.

Measurements were made using an *ArF* excimer laser, *Photon Machines™* which was coupled to an ICP MS,*Thermo*™, generating 5 ns pulses at 193 nm. The laser chamber was buffered with helium gas and laser spots onto samples were $30\,\mu m$ circles (Fig. 7). Repetition laser rate was maintained at 10 pps (pulses per second) and the laser energy per pulse was 0.8 mJ. Therefore, per pulse laser energy density was about $1.1\times10^{8}J/m^{2}$. In order to obtain good signal/noise rate and low instability from detectors output the ablation in each point was made with laser bursts varying from 600 to 1800 pulses. excimer laser, *Photon Machines™* which was coupled to an ICP MS,*Thermo*™, generating 5 ns pulses at 193 nm. The laser chamber was buffered with helium gas and laser spots onto samples were $30\,\mu m$ circles (Fig. 7). Repetition laser rate was maintained at 10 pps (pulses per second) and the laser energy per pulse was 0.8 mJ. Therefore, per pulse laser energy density was about $1.1\times10^{8}J/m^{2}$. In order to obtain good signal/noise rate and low instability from detectors output the ablation in each point was made with laser bursts varying from 600 to 1800 pulses.

Mass detectors were set up to *Pb* isotopic masses: ^{208}Pb, ^{207}Pb, ^{206}Pb and ^{204}Pb. An additional detector, an ion counter, was tuned for the^{202}Hg mass in order to evaluate *Hg* presence in the process and subtract the contribution of the ^{204}Hg from that of ^{204}Pb.

The measurement procedure includes a first blank evaluation, then that of a standard (NIST 612), followed by the first sample measurement. Afterwards, there is another measurement on the standard and a last one of the background. For each sample, this procedure is repeated twenty times, that is twenty laser shots in different regions of the sample in order to obtain one data point. In general, this procedure is repeated at least thrice, generating three data points. Nevertheless, in some special cases, due to inhomogeneities in the sample more points must be generated to improve the statistic.

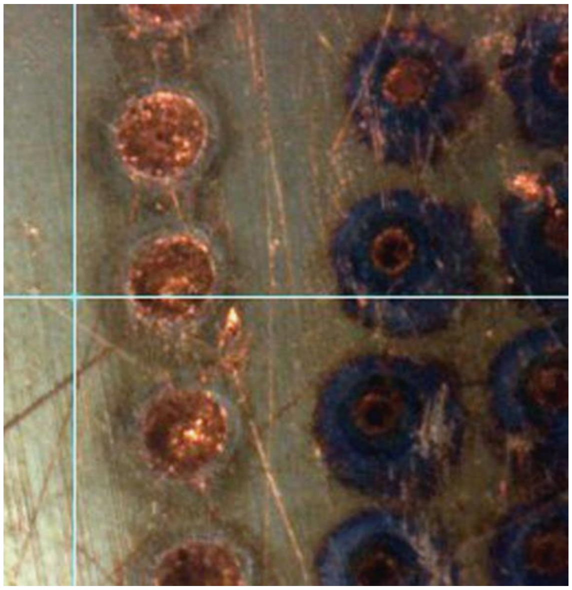

Figure 7 – Laser spots in gold sample. The row of spots in the center are showing good laser ablation while the other two at right not.

The resulting data are registered by the equipment acquisition program and then are transferred to $Microsoft\,Excel^{TM}$ in order to make blank and mass fractionations corrections to calculate isotopes ratios and construct convenient graphics.

Results

The results are synthetically presented in Table 1 and graphically displayed in the figures from 8 to 10.

Table 1 - Consolidated results

Gold origin	Isotopes rates					
Cuiabá	^{208}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{206}Pb/^{204}Pb	^{208}Pb/^{206}Pb	^{207}Pb/^{206}Pb	^{206}Pb/^{207}Pb
1	74,14	47,17	47,12	1,57	1,00	1,00
2	73,81	47,40	47,33	1,56	1,00	1,00
3	71,05	45,65	45,61	1,56	1,00	1,00
4	72,38	46,31	46,01	1,57	1,01	0,99
5	75,39	48,12	48,13	1,57	1,00	1,00
6	72,56	45,31	45,86	1,58	0,99	1,01
7	71,73	44,48	45,30	1,58	0,98	1,02
8	69,75	42,90	43,69	1,60	0,98	1,02
9	73,25	45,89	46,22	1,58	0,99	1,01
10	72,82	45,84	46,20	1,58	0,99	1,01
Average	72,69	45,91	46,15	1,58	0,99	1,01
St. Dev.	1,53	1,43	1,16	0,01	0,01	0,01
Lavrinha	^{208}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{206}Pb/^{204}Pb	^{208}Pb/^{206}Pb	^{207}Pb/^{206}Pb	^{206}Pb/^{207}Pb
1	29,06	13,21	15,14	1,92	0,87	1,15
2	60,41	27,65	31,71	1,91	0,87	1,15
3	87,68	36,73	45,87	1,91	0,80	1,25
4	54,95	25,05	28,88	1,90	0,87	1,15
5	47,15	20,60	23,71	1,99	0,87	1,15
6	35,16	20,09	19,33	1,82	1,04	0,96
7	38,06	16,52	19,11	1,99	0,86	1,16
Average	50,35	22,84	26,25	1,92	0,88	1,14
St. Dev.	18,37	7,23	9,64	0,05	0,07	0,08
Peixoto	^{208}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{206}Pb/^{204}Pb	^{208}Pb/^{206}Pb	^{207}Pb/^{206}Pb	^{206}Pb/^{207}Pb
1	92,73	58,03	58,41	1,59	0,99	1,01
2	125,06	84,17	79,77	1,57	1,06	0,95
3	76,44	47,55	48,18	1,59	0,99	1,01
4	99,90	62,03	63,40	1,58	0,98	1,02
5	93,46	56,26	58,18	1,61	0,97	1,03
6	124,51	80,11	79,79	1,56	1,00	1,00
Average	102,02	64,69	64,62	1,58	1,00	1,00
St. Dev.	19,26	14,38	12,74	0,02	0,03	0,03
Colômbia	^{208}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{206}Pb/^{204}Pb	^{208}Pb/^{206}Pb	^{207}Pb/^{206}Pb	^{206}Pb/^{207}Pb
1	31,28	12,97	17,16	1,82	0,76	1,32
2	14,82	5,89	7,74	1,92	0,76	1,31
3	49,70	19,92	25,59	1,94	0,78	1,28
4	27,68	11,30	14,83	1,87	0,76	1,31
Average	30,87	12,52	16,33	1,89	0,76	1,31
St. Dev.	14,40	5,78	7,36	0,05	0,01	0,02

As it can be seen in Table 1 there is a larger stnadard deviation in the isotopes rates which includes the ^{204}Pb, that is, ^{208}Pb/^{204}Pb, ^{207}Pb/^{204}Pb and ^{206}Pb/^{204}Pb. Fig. 8 is a plot of ^{208}Pb/^{204}Pb versus

^{207}Pb/^{204}Pb rates and there it can be see that there is an evident dispersion of the results for each provenance in quite different areas in the graphic. The best precisions are those from Cuiabáand Colombia samples but nevertheless tendency lines were used in order to better characterize each sample. We attribute this dispersion to the^{204}Pb measurement. The signal of ^{204}Pb collected in the mass spectrometer fluctuates in a quite randomic way due the ^{204}Hg present in the process. So, the measurement protocol includes measuring ^{202}Hg presence in the buffer gas flow, and afterwards to estimate from this measurement the contribution of the isotope^{204}Hg to the total of 204 mass. Then, with a little calculation one can obtain the isotopic mass of the ^{204}Pb. There is no doubt that this is a problem, which concerns much more to the sampling using laser ablation, but it can not be neglected still in the measuring from samples in solutions but in this case errors can be easily minimized.

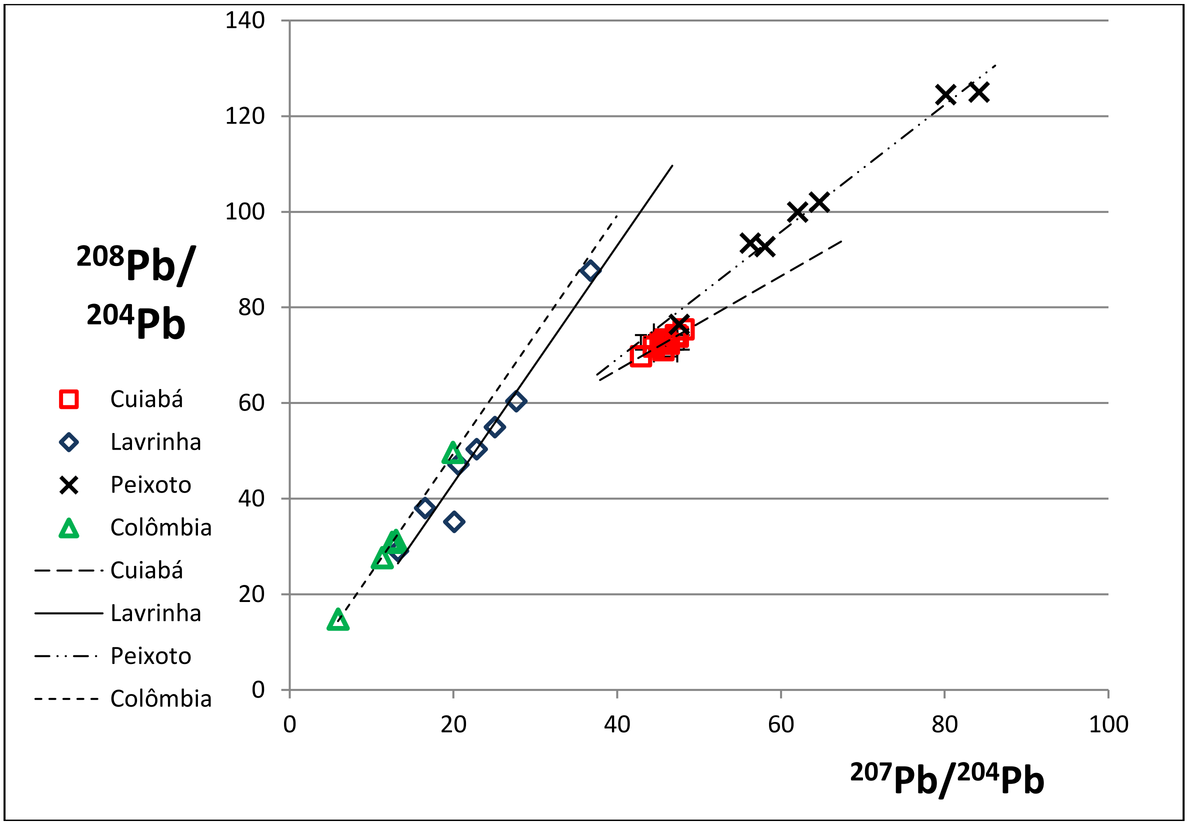

Figure 8: ^{208}Pb/^{204}Pb versus ^{207}Pb/^{204}Pb rates plot showing tendency lines

The second and third plots, Figs. 9 and 10respectively, follow the isotopic rates and their graphs routinely used in Archeometry research [9]. The second one is a plot of^{208}Pb/^{206} versus^{207}Pb/^{206} rates. In Fig. 9 it can be seen a little more discrepancy among different data points for the same provenance chiefly for Peixoto de Azevedo and Colombia but the Cuiabá sample shows a quite good precision disagreeing from those of Peixoto de Azevedo and Colombia.

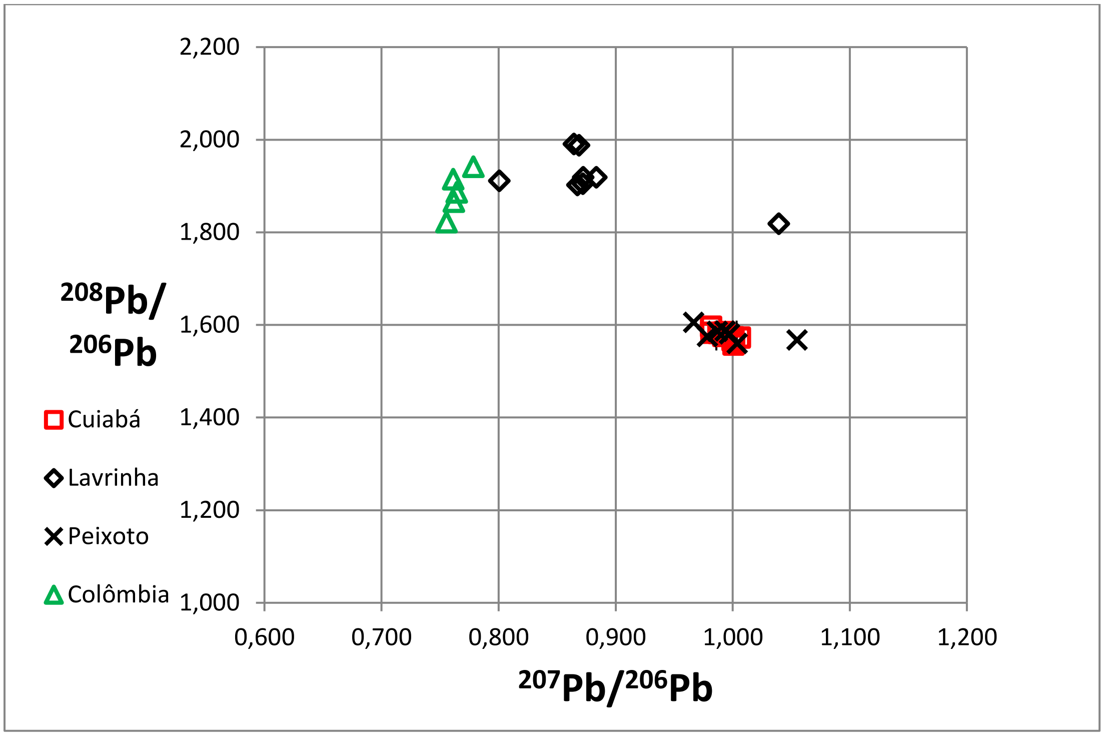

Figure 9 - $^{208}Pb/^{206}Pb$ versus $^{207}Pb/^{206}Pb$ rates plot

Fig. 10 illustrates the results of $^{206}Pb/^{204}Pb$ versus $^{207}Pb/^{206}Pb$ and there it can be seen a larger dispersion in the data of almost all samples except in that of Cuiabá. We can attribute this fluctuations in the results to the measurements of ^{204}Pb, which is strongly influenced by the ^{204}Hgnatural presence in the discharge gas and in the buffer gas, argon and helium, respectively.

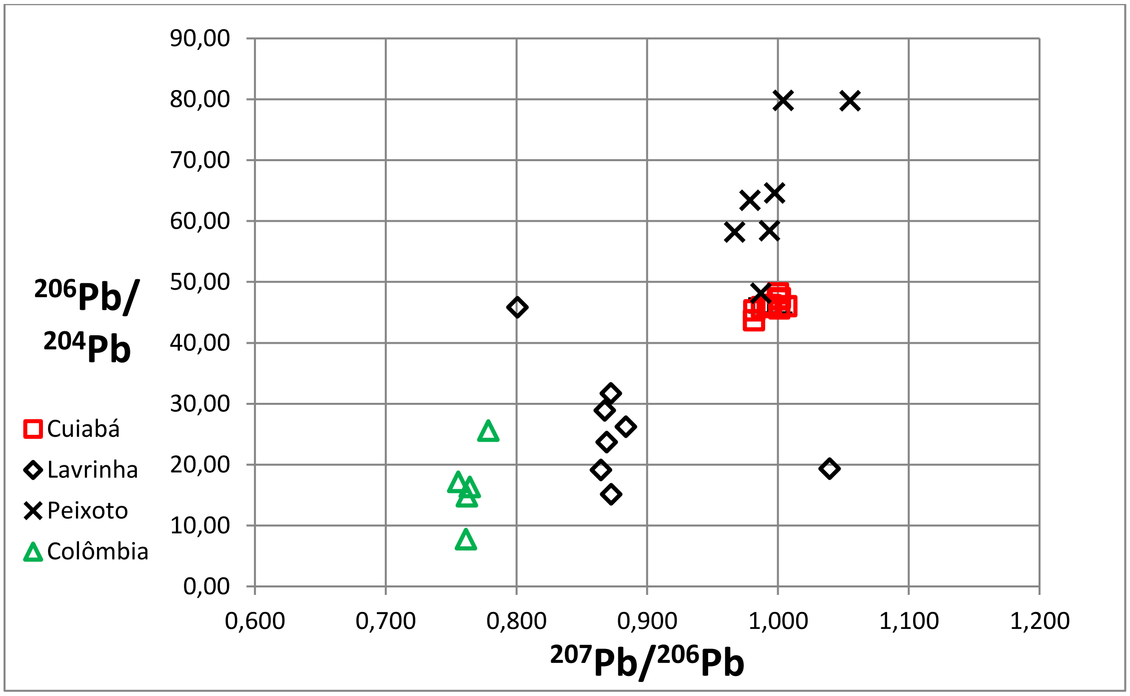

Figure 10 - $^{206}Pb/^{204}Pb$ versus$^{207}Pb/^{206}Pb$ rates plot

Conclusions

This paper presents some preliminary results in characterizing pure gold, only fusion processed, using ICP-MS with laser ablation. In the beginning there were some doubts about the quantity of Pb isotopes in the samples and equipment ability to detect and process them in order to obtain reliable results. However, first tests showed gold samples with a well measurable Pb presence, comparable to that usually found in zircon crystals, much used in Geology for datation by U-Pb method. In despite of these results being preliminary it can be seen that is possible to well characterize gold samples by their Pb isotopes rates connecting the results to the geographical regions of gold mining. In the present case we are reproducing and creating the protocols in order to make a data basis for different gold provinces in Brazil and Latin America. This data base will serve also to retrace provenance of old jewelry, which is an branch of archeometric research. As long we are measuring Pb traces in a metal, these procedures can be used to identify provenances in artifacts of other metals like silver, brass, bronze, tin, copper, iron and others. In metallurgy of antique civilizations lead is an omnipresent contaminant, and not only in those but it still can be found in pottery and glass artifacts. We must remind that our detection levels are in the range

from a few to a few tens of parts per billion, that is, a few tens of milligrams by ton.

The Pb isotopes signatures allowed to distinguish different geologic sources for metallic deposits. The results here presented define important parameters for regional metallic exploration in SW Amazonian craton.

1. The Cuiaba gold deposits are characterized by a strong correlation between deformation of country rocks and the ore minerals. With the available data is not possible to define if the metal concentration in Cuiabá Deposit was related neoproterozoic (ca 540 My) evolution of the Paraguai mobile belt when magmatic processes or linked to shearing and fluid remobilization during the orogen evolution, as recorded by 543-520 My 40Ar/39Ar cooling ages in biotite.
2. The Pb isotope model ages of the Peixoto de Azevedo deposit are roughly coeval to the 1762-1755 Ma U-Pb LAICPMS ages of the volcanic-plutonic rocks of the Ata floresta Gold Province cratonic. The ca.1.80 Ga granitic intrusions accompanied by hydrothermal solutions deposition suggest a orogenetic origin for Peixoto de Azevedo gold deposit.
3. In the case of Lavrinha gold deposits, the ore formation is correlated with the movement of the hydrothermal fluids during the Aguapei tectonic event. The ca. 920 My mineralization is coeval with the deformation and the hydrothermal solutions percolating through older granitic (1.45-1.42 Gy Santa Helena) and basic (1.51-1.49 Gy Rio Alegre) and sedimentary rocks (Aguapei Group).
4. In the case of Lavrinha gold deposits, the ore formation is correlated with the movement of the hydrothermal fluids during the Aguapei tectonic event. The ca. 920 My mineralization is coeval with the deformation and the hydrothermal solutions percolating through older granitic (1.45-1.42 Gy Santa Helena) and basic (1.51-1.49 Gy Rio Alegre) and sedimentary rocks (Aguapei Group).

There is still large room to other research in this area. It should be advisable compare mesures made by laser ablation with those using solutions in order to compare errors in both cases. The method application to archaeometric pieces is being considered because laser ablation is very little invasive, preventing damages in ancient artifacts. Therefore, the study of different materials artifacts like glass, ceramic and still wood is being considered.

Acknowledgements

We thank very much Finep, FAPERJ and CNPq for the financial support necessary to mount the infrastructure used for this research.

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