



## Arsenic determination in complex mining residues by ICP OES after ultrasonic extraction

Jéssica F.R. Paula <sup>a</sup>, Roberta E.S. Froes-Silva <sup>b,c,\*</sup>, Virgínia S.T. Ciminelli <sup>a,b</sup>

<sup>a</sup> Universidade Federal de Minas Gerais, Escola de Engenharia, Departamento de Engenharia Metalúrgica e de materiais, Brazil

<sup>b</sup> Instituto Nacional de Ciência e Tecnologia, Recursos minerais, Água e Biodiversidade, Brazil

<sup>c</sup> Universidade Federal de Ouro Preto, Instituto de Ciências Exatas e Biológicas, Departamento de Química, Morro do Cruzeiro, Campus Universitário, Ouro Preto/MG, 35.400-000, Brazil

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### ABSTRACT

The impact caused by mining residue deposits with high amounts of arsenic is a potential environmental problem and motive for investigations. Arsenic is always associated with gold ores and is present in mining areas, probably due to sulfide oxidation and the high pH range where arsenic is soluble. It has been shown that some samples of residues coming from the studied deposits presented arsenic concentrations above 2500 mg kg<sup>-1</sup>. The aim of this work is to develop methods for arsenic extraction in mining residues employing ultrasound assistance extraction and analyte determination by hydride generation ICP OES. The analytical method provided analysis precision and a better evaluation of environmental impact of mining residue deposits. Optimal conditions for ultrasound assisted extraction were obtained by evaluation of parameters such as the position of the sample flask inside the ultrasound bath and temperature as a function of the immersion time. These tests identified the ideal position of the sample flask and indicated that the maximum time that the sample could be analyzed without arsenic lost due to increase of the temperature was 180 minutes. With these conditions, an experimental design was developed to obtain the optimal conditions of arsenic extraction, evaluating the type of extraction solution and the sonication time. A 2<sup>2</sup> Central Composite Design (CCD) was employed to evaluate the acid concentration (HCl, HNO<sub>3</sub> and H<sub>3</sub>PO<sub>4</sub>) and the sonication time (maximum 180 minutes). The quadratic equation of response surface generated by CCD, employing HNO<sub>3</sub>, indicated that the best condition for arsenic extraction is using an acid concentration of 2.7 mol L<sup>-1</sup> and sonication for 136 minutes to give an average concentration of 1847 ± 17 mg kg<sup>-1</sup>. Recovery studies were carried out and the obtained values were close to 100%. Optimal conditions of arsenic extraction were obtained by evaluating the operational parameters of ultrasound bath and the analysis conditions with the experimental design. The method developed contributes significantly to a more precise evaluation of the real environmental impact caused by the mining residues deposit.

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### 1. Introduction

Arsenic in mining areas has created an environmental problem. The presence of arsenic in certain mining areas has been highlighted in the scientific literature by various research groups [1–5]. Arsenic is always associated with gold ores and is present in mining areas, probably due to sulfide oxidation and the high pH range where arsenic is soluble [6]. It has been shown that some samples of residues coming from a deposit of mining waste presented arsenic concentrations above 2500 mg kg<sup>-1</sup> [7].

Some mining wastes are difficult to solubilize and new so extraction procedures are needed to assess the free arsenic. Alternative studies evaluating the potential arsenic extraction are being realized to understand the arsenic readiness in the residue and soils. Sequential chemical

extraction procedure was used to evaluate the arsenic concentration in mining waste and soils by [8]. Ruiz-Cancho et al. [9] used a block digester and a microwave digestion to obtain the extract to evaluate the arsenic species present by LC-HG-AFS. The application of ultrasound is very promising in hydrometallurgy, including accelerating the processes of extraction and leaching. The ultrasound procedure can make available a chemical species present in a solid matrix for the extraction or for the dissolution of the species of interest. The energy required for extraction of metals is provided by the field of cavitation, which causes an increase in local temperature in the order of hundreds of degrees and a pressure increase of several hundred atmospheres. In addition, micro turbulent pulsations in thin layers of liquid in contact with the solid particles [10]. This technique promotes the larger interaction between the liquid phase and the solid surface by reduction of particles size and acceleration of chemical dissolution and thus increasing the concentration of inorganic species in the liquid phase [11,12].

A multivariate optimization can be applied in the development of new methodologies for arsenic extraction. This technique can promote

\* Corresponding author. Tel.: +55 31 3559-1233; fax: +55 31 3559 1228.

E-mail address: [robertafroes@iceb.ufop.br](mailto:robertafroes@iceb.ufop.br) (R.E.S. Froes-Silva).

a fast and precise design evaluating multi parameters at the same time [13–15]. The surface response generated by this design can indicate the optimal condition for analysis. The application of multivariate optimization in the arsenic extraction could be an alternative for analysis of the complex mining waste, which can be useful for the evaluation of real impact caused by the complex mining waste deposit.

The aim of this work is the development of a new and alternative methodology for arsenic extraction from complex mining waste with difficult decomposition using ultrasound extraction.

## 2. Experimental

### 2.1. Instrumentation

An analytical balance Kern 410 model was used to measure the mass of the samples. An ultrasonic cleaning bath GA1000/TA1800 (Thornton; São Paulo, Brazil) with a frequency of 25 Hz was used for the extraction of arsenic from mining tailings samples.

A closed vessel microwave oven (Ethos 1, Milestone; Sorisole, Italy) was used to decompose the sample in order to compare the efficiency between the open ultrasonic bath using optimized conditions and the experiment performed under microwave conditions. The extracts were analyzed in an inductively coupled plasma optical emission spectrometer (ICP OES) model: DV 7200 (Perkin Elmer instruments, Shelton, USA) with hydride generation. The operational conditions are presented in Table 1.

### 2.2. Reagent, solution and sample

A standard  $1000 \text{ mg L}^{-1}$  arsenic solution (ICP Standard, Merck Certipur®, Darmstadt, Germany) was used to prepare the calibration curve and reference solutions. All the solutions were prepared with ultrapure water. For hydride-generating were used hydrochloric acid solution ( $2 \text{ mol L}^{-1}$ ) (37% HCl, Merck), sodium borohydride ( $\text{NaBH}_4$  tablets, Fluka, purum, purity >97%) at 1.5% in 0.2% NaOH (Merck). The  $\text{HNO}_3$ , HCl and  $\text{H}_3\text{PO}_4$  (Merck, analytical grade) concentrated acids used were diluted with ultrapure water to prepare the solutions for analysis. The reference material Buffalo River Sediment (National Institute of Standards and Technology, NIST RM 8704), were used as reference for some tests. Real sample of complex mining waste, collected in a deposit of mining, was used in the multivariate optimization and the recovery studies.

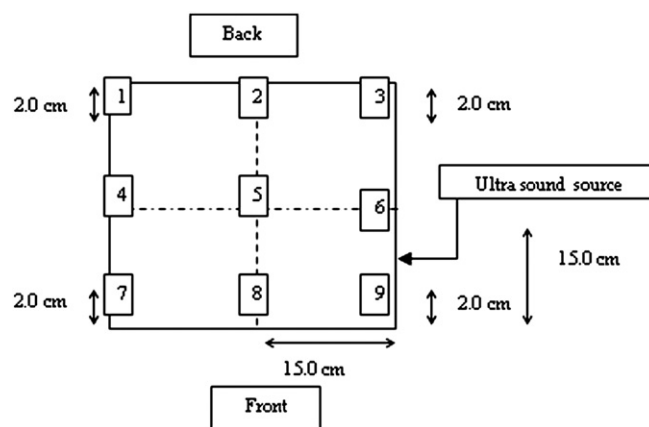
### 2.3. Evaluation of axial position in the flask

In order to assess the influence of the position of the sample inside the ultrasonic bath, the tub was divided into four quadrants, 15 cm by 15 cm. Next, nine locations in the tub were selected, as indicated in the layout of Fig. 1.

The samples are prepared in duplicate using 0.1 g of a reference material Buffalo River Sediment 8704 and mixed in a flask containing 10.0 mL of  $\text{HNO}_3$   $0.5 \text{ mol L}^{-1}$  solution. The flasks were sonicated for 1 hour. Subsequently, the samples were centrifuged and the supernatant was transferred to 15 mL polyethylene tubes for analysis of the arsenic concentration via ICP-OES.

**Table 1**  
Operational conditions of ICP OES.

Parameter	
Auxiliary gas flow rate ( $\text{L min}^{-1}$ )	0.2
Plasma gas flow rate ( $\text{L min}^{-1}$ )	15
Injector tube diameter (mm)	2.0
View	Axial
Interface	Shear gas
Applied power (kW)	1.3
Nebulization gas flow rate ( $\text{L min}^{-1}$ )	0.6



**Fig. 1.** Layout of selected positions in the ultrasound bath.

### 2.4. Evaluation of the temperature as a function of time in the ultrasound bath

The effect of sonication in the sample system cannot be considered only as a mechanical wave effect. The temperature increasing in the bath due the energy liberated [16] should also be considered. The monitoring of the temperature was made in a range of 180 minutes, by measuring the temperature at intervals of 25 minutes.

To evaluate the influence of temperature on the extraction of arsenic, the reference material "Buffalo River Sediment 8704" was weighed in duplicate samples with a nominal mass of 0.2 g and then placed in 50 mL polyethylene tubes. To the tubes containing the samples 20.0 mL of  $\text{HNO}_3$   $2.0 \text{ mol L}^{-1}$  was added before washing them in the ultrasonic bath. One sample was placed in the bath at room temperature,  $25^\circ\text{C}$ , and the other to the initial temperature of  $40^\circ\text{C}$ . Both were sonicated for 1 hour. Subsequently the samples were centrifuged for 10 minutes and the supernatant was stored in 50 mL polypropylene tubes until analysis via ICP-OES.

### 2.5. Multivariate optimization

For the extraction of arsenic by ultrasound sonication it was used a mass ratio of solid / solution volume of extractant  $0.01 \text{ g mL}^{-1}$ . This mass ratio is based in the work where the same ratio was used to evaluate arsenic extraction to speciation in soil using a probe ultrasonic [17].

Ten samples were weighed in duplicate, 0.1 g of solid waste (with arsenic concentration estimated in  $2000 \text{ mg kg}^{-1}$ ) in polyethylene bottles (15 mL). To each bottle 10.00 mL of extractant solution was added, minutes before the samples were subjected to ultrasonic bath. The flasks were shaken manually to allow contact between the entire solid with the acid solution.

A Central Composite Design  $2^2$  (CCD) was used in order to assess the concentration of acid (HCl,  $\text{HNO}_3$  and  $\text{H}_3\text{PO}_4$ ) and sonication time (maximum 180 minutes). To avoid the mixing of the acids, the same study design was employed individually for HCl,  $\text{HNO}_3$  and  $\text{H}_3\text{PO}_4$ . The implementation of the design was done in Statistica ® 7.0 software. The design data are shown in Table 2.

The bottles were placed in the ultrasound bath tub in the region determined to have higher-intensity ultrasonic waves (see layout) at room temperature initially, and subjected to ultrasonic energy at a frequency of 25 kHz.

After soaking in a bath of ultrasound during the specific time for each sample, the resulting mixture was filtered and the supernatant was stored in polyethylene bottle and cooled to  $5^\circ\text{C}$  and subsequently analyzed by ICP-OES.

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