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ALTERNATIVE REAGENTS TO CYANIDE IN GOLD LEACHING - A CASE STUDY: APPLICATION OF THE AMMONIACAL- THIOSULPHATE SYSTEM ON CASTROMIL ORES

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GOLD LEACHING - A CASE STUDY:
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THIOSULPHATE SYSTEM ON CASTROMIL
ORES**

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*“The important thing is not to
stop questioning. Curiosity has its
own reason for existing.”*

Albert Einstein

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ABSTRACT

Environmental awareness has made a name for itself in the past few decades. Due the negative impact of the mining industry, a growing need for cleaner and “greener” processing technologies has risen.

Cyanide is commonly used in mineral processing, more precisely, in gold leaching. Since it is a pernicious substance, some alternatives have been studied, sodium thiosulphate being one of them.

Before describing the laboratory procedures, a theoretical approach to this concern is presented, taking into consideration some of the most relevant substances to substitute cyanide.

This work compiles a set of experiments which studied the behavior of the ammoniacal-thiosulphate system applied to two Castromil ore samples: one a sulphide and the other an oxide. Parameters such as the concentration of each substance, temperature, speed rotation and leaching duration time were varied in order to find out what the optimal conditions were.

The conclusions were different for each sample. Regarding the sulphide ore sample, the thiosulphate concentration should be 1 M, the copper 0.01 M and the ammonia 2 M. The speed rotation should be kept at level 0 and the temperature between 40 and 45 °C. On the other hand, the oxidized ore sample should be leached with a solution composed by sodium thiosulphate 0.5 M, copper 0.01 M and ammonia 3 M. Both temperature and speed rotation should be kept the same as the previous sample.

KEYWORDS: gold leaching, Castromil, cyanide, sodium thiosulphate, ammoniacal-thiosulphate system

RESUMO

A consciência ambiental associada ao desenvolvimento sustentável tem sido um dos temas com maior destaque nas últimas décadas.

Devido à conotação negativa à qual a indústria mineira frequentemente se encontra associada, levanta-se uma necessidade crescente de tecnologias de processamento mais “limpas” e amigas do ambiente.

O cianeto é usado num desses procedimentos, mais precisamente, na lixiviação do ouro. Atendendo a que é uma substância perniciosa, algumas alternativas têm sido estudadas, sendo o tiosulfato de sódio uma delas.

Antes da descrição dos procedimentos de laboratório, é apresentada uma abordagem teórica ao tema, onde algumas das substâncias mais relevantes na substituição do cianeto são elucidadas.

Esta obra compila um conjunto de experiências que estudou o comportamento do sistema amónia-tiosulfato aplicado a duas amostras de minério providas de Castromil: um sulfureto e um óxido. Parâmetros tais como a concentração de cada substância, a temperatura, a velocidade de rotação e duração da lixiviação foram variados, a fim de perceber quais eram as condições ideais de lixiviação para cada minério.

As conclusões obtidas foram diferentes para cada amostra. Para o tratamento da amostra de minério sulfuretado, a concentração de tiosulfato deve ser 1 M, a de cobre 0,01 M e a de amónia 2 M. A velocidade de agitação da polpa deve ser mantida no nível 0 e a temperatura entre 40 e 45 ° C. Por outro lado, a amostra de minério oxidado deve ser lixiviada com uma solução composta por tiosulfato de sódio 0,5 M, cobre a 0,01 M e amónia a 3 M. Tanto a temperatura como a velocidade de agitação da polpa devem ser mantidas nos pressupostos anteriores: nível zero e entre 40 e 45 ° C.

PALAVRAS-CHAVE: lixiviação do ouro, Castromil, cianeto, tiosulfato de sódio, sistema amónia-tiosulfato

I declare, under oath, that this work is original and that all non-original contributions were properly referenced with identifying the source.

Joana Alexandra Silva Duarte
Porto, 23 de Julho de 2015

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NOMENCLATURE

VARIABLES

Variable	Definition	Unit
Eh	Redox potential	mV
pH	Ionic Hydrogen Potential	pH scale values

SYMBOLS

Symbol	Definition
#	mesh
Ag	Silver
Al	Aluminum
As	Arsenic
Au	Gold
B	Boron
Ba	Barium
Be	Beryllium
Bi	Bismuth
Ca	Calcium
Cd	Cadmium
Cl	Chlorine
Co	Cobalt
Cr	Chromium
Cu	Copper
d ₈₀	Caliber at which 80% of the material is under
Fe	Iron
Ga	Gallium
Hg	Mercury
K	Potassium
La	Lanthanum
LD ₅₀	Lethal Dose, 50%

Mg	Magnesium
Mn	Manganese
Mo	Molybdenum
Na	Sodium
NaCN	Cyanide hydrogen
NaOH	Sodium hydroxide
Ni	Nickel
P	Phosphorus
Pb	Lead
Rb	Rubidium
S	Sulfur
Sb	Antimony
Sc	Scandium
Se	Selenium
Sn	Tin
Sr	Strontium
Th	Thorium
Ti	Titanium
Tl	Thallium
U	Uranium
V	Vanadium
W	Tungsten
Zn	Zinc
Zr	Zirconium

ABBREVIATIONS

Abbreviations	Definition
ASTM	American Society for Testing and Materials
LNEG	Laboratório Nacional de Energia e Geologia
ORP	Oxidation-Reduction Potential
rpm	Revolution per minute
USA	United States of America
wt	weight

1. INTRODUCTION

Background and motivation

Environmental awareness has taken over every field of every industry and during the 21st century, sustainable development made a name for itself.

According to the United Nations World Commission on Environment and Development in “Our Common Future”, a 1987 report, sustainable development is described as the development that seeks to meet the needs of the present generation without putting into jeopardy the needs of generations to come [1]. It is believed that the three main pillars of sustainable development are environmental, social-political and economic and it is their steadiness which provides the ecological balance planet Earth needs.

The Mining industry is believed to be one of the most environmentally damaging industries since it deals with a huge amount of subsurface material, sometimes distressing the natural balance of the surrounding ecosystems. On top of that, all of the extracted material has to be processed and divided into one of two categories: ore or waste.

Usually the waste goes into the heaps, where some very unstable and toxic compounds begin to occur.

Sometimes it is the methods used to process the ore itself that begins to cause concern. Leaching is one of those methods.

Cyanide leaching has been used for the economic extraction of gold in the past decades despite its perniciousness (mainly toxic). It uses solutions of sodium or potassium cyanide as lixiviants in order to extract the valuable mineral from a solid mass.

Due to the negative impact cyanide has on the environment there is an urgent need to develop alternative and environmentally friendly extraction methods in order to prevent hazardous occurrences.

Objectives of this work and project presentation

This dissertation aimed to contribute to the study of alternative reagents in gold leaching (in opposition to cyanide).

The present work includes the assembly of an experimental setup in order to make various assays using those reagents, studying the impact of the variation of some parameters on the performance of the tests, such as effect of thiosulphate concentration, effect of ammonia, effect of cupric ions, and a few other variables like time of the experiment, agitation and temperature.

This work proposal was based on the studies made in Akashi and Hishikari mines [2], and adapted to the Castromil's samples.

Besides the engineering component, the development of this project enabled a systematic learning about the phenomena involved in the leaching of gold ores.

Main contributions of this work

One of the main contributions of this work was the assembly of experiments, concerning the ammoniacal-thiosulphate system in order to better understand the reagent, and how to prove its yield in gold leaching.

This setup is of great importance for the industrial mining processing field since cyanide is a dangerous substance, despite its greater use, and needs imperative replacement.

Although environmental and safety concerns were the prime reasons to study alternatives to cyanide leaching, there is also great interest in achieving higher recoveries rates of the valuable mineral using different reagents. Only by showing that alternative reagents to leaching can be either as economical or more than cyanide can we expect to create change in the industry.

Outline of this work

Chapter 1 - Introduction

A brief introduction to the topic of sustainable development and the increasing need for sustainable mining in the current society was made.

The present situation of gold leaching, and the repercussions of cyanide use were also put into perspective. The objectives and contributions of this work were presented as well.

Chapter 2 - State of the Art

Important aspects of the general leaching working procedures were presented. An overview concerning cyanidation, the chemical equations and the most important techniques were described and a summary of the most significant alternative reagents were displayed.

Chapter 3 - Castromil Samples

A brief geological and geographical context was presented for the Castromil Mines along with each sample foremost chemical characteristics and gold occurrences expected for them.

Chapter 4 - Technical description and procedures

The gold leaching assembly of the experimental setup used in this project was explained in detail. Procedures for the sample preparation and each assay for the different samples were described in this chapter, as well as their main goals.

Chapter 5 - Results and discussion

The main results obtained in this study were presented and discussed in this chapter, namely which were the optimal conditions for the gold leaching using an ammoniacal-thiosulphate system and the results of the various assays made on the experimental setup components considering pH, Eh and other variables. All chemical analysis performed during the assays and to the system feeds were also discussed.

2. STATE OF THE ART

2.1 Gold – historical insight

Gold has been used for centuries in many societies due to its characteristics; for example its malleability, allowing the production of thin sheets, ensured its decorative applications.

Egypt was one of the first cultures to take advantage of this metal; the country was rich in mineral resources and gold mines were intensively exploited in Nubia [6].

Gold was extremely abundant in the eastern desert since it could be found in a vast region of mountainous rocks between Red Sea and the Nile. Not only did those ancient rocks had deposits of gold but also silver, copper, lead, iron and zinc aggregated.

The occurrence of gold could manifest in one of two ways: alluvium or white quartz veins of the mountainous rocks formerly mentioned.

The gold extracted from alluvium did not need qualified personnel for it was processed simply by washing with water, on a sloping surface. Heavier materials (gold) were left behind in nuggets shape whilst lighter were dragged.

Concerning the quartz veins the rock was heated to become brittle, it was hit with heavy equipment where it fragmented by the hands of skilled hammers and then transported to the outside of the mine, where it was milled cutting it into peas' size. Afterwards the material was crushed into powder and then washed in sloping surfaces in order to separate the metal from it [7].

The final purpose of such work was to manufacture metal tools, jewellery, statues and monuments, figure 2.1.



Figure 2.1 - Tutankhamun's Mask

During the Renaissance, Vannuccio Biringuccio (1480-1539) published “De La Pirotechnia” (figure 2.2), the first printed book of metallurgy, consisting of ten chapters, the first four fully dedicated to minerals, metal and separation of gold and silver.



Figure 2.2 - De La Pirotechnia book cover

By then, the common sense was “(...) this metal is malleable and of a shining colour almost like that shown to us by the sun. It has in it a certain natural and intrinsic attraction which causes men to desire it when they see it.” [8].

Georgius Agricola (1494-1555) wrote “De Re Metallica” in a time when “Any persons hold the opinion that the metal industries are fortuitous and that the occupation is one of sordid toil, and altogether a kind of business requiring not so much skill as labour.” [9]. The book has twelve chapters and six of them are about geology, mining and ore processing.

Lazarus Ercker (1528/30 - 1594) was the last contributor for these insights writing “*Beschreibung allerfürnehmsten mineralischen Ertzt und Bergwercksarten*” in 1574. His book consisted of five chapters: the first four dedicated to minerals, concentrations of metals in ores and refining of metals.

In 1887, John Macarthur and William Forrest patented the cyanide process which was the birth of modern hydrometallurgy [10].

2.2 Main features and occurrences

With a characteristic yellow colour due to the intense absorption of light caused by the transitions of its electrons between its energy bands, gold is the most malleable and ductile metal of them all, being 2-3 on the Mohs scale of mineral hardness (out of 10). The only substance capable of making some corrosion to it is the so-called *aqua regia*: a mixture of both nitric and hydrochloric acids.

Au is the scientific symbol used for gold; its atomic number is 79, and it belongs in the 11th group and 6th period of the Periodic Table of Elements [12].

On opposition to other metals, gold cannot be tarnished or corroded by moisture. It is not oxidized (due to water and oxygen) and ordinary acids do not have influence in it: nitric acid only cleans the ore's surface rather than dissolving it.

Gold's density is 19.3 g/cm³ and it depends both on its crystal structure as well as its atomic mass [13].

The temperature on which pure gold melts is 1064 °C, even though when it is combined with other substances, it has a wider range of temperatures. When transforming from liquid to gaseous state, the temperature is usually around 2860 °C, according to the same source.

The electrical resistivity of gold is 0,022 micro-ohm m (at 20 °C). At the same temperature, its thermal conductivity is 310 W m⁻¹ K⁻¹.

According to the Periodic Table of Elements, gold's atomic radius is 1.79 Å, the covalent radius is about 1.34 Å and the ionic radius is 0.85 Å. Its atomic volume is 10.2 cm³/mol.

Some of its physical properties are:

- Elastic Modulus:
 - Bulk: 171 GPa
 - Rigidity: 26 GPa
 - Youngs: 78.5 GPa
- Enthalpy of atomization: 364 kJ/mole (at 25 °C)
- Enthalpy of fusion: 12.55 kJ/mole
- Enthalpy of vaporization: 324.4 kJ/mole
- Heat of vaporization: 334.4 kJ/mol
- Molar volume: 10.2 cm³/mole
- Physical State (at 20 °C and 1atm): Solid

The only natural occurrence of gold isotope is the ^{197}Au . Every other is listed as radioisotopes, like ^{194}Au , ^{195}Au , ^{196}Au , ^{198}Au and ^{199}Au .

From the above mentioned one of the most important is the radioisotope ^{198}Au which is used for treating cancer and other medical conditions [14].

Due to its advantageous features, gold has a wide application: from jewelry to textile industry. It can be also applied in science fields, such as medicine (dental applications, for instance) and engineering (radiation-control coating for spacecrafts) [11].

Gold is commonly associated with pyrite, quartz and other minerals and it is known that two thirds of the world's supply of gold comes from South Africa. This metal is widely distributed, despite its very low concentration.

Mineral classification, when referring to metals, can be provided by some parameters such as abundance in nature, mineralogical composition or treatment process [15].

According to Mc Quiston and Shoemaker (1975) and Adison (1980), the classification for the gold ores is:

- ✓ Native gold, varying its purity and grain size (ranging from several centimeters to micron sizes). Small sized gold can be found in sedimentary deposits, but more often in magmatic deposits such as epithermal. The most well-known alluviums are in the United States of America, Canada, Alaska, Brazil, Venezuela, and Australia. The usual processing for this metal are gravimetric concentration, amalgamation and flotation. In addition to silver, native gold, may contain small amounts of copper and iron [35].
- ✓ Combined gold, being tellurium (with or without silver) one of the most usual forms of it. It can contain small amounts of other elements such as antimony, lead or mercury. However, deposits having good economic exploitation conditions due only to telluride concentrations are rare [35]. Some of them can be found in Cripple Creek and Jamestown (Colorado, USA), Golden Mile (Kalgoorlie, Australia) and Vatukoula (Fiji islands, South Pacific Ocean). The most common processing for this metal are flotation and cyanide leaching (the residues are processed with sulfur dioxide).
- ✓ Gold in association with other elements such as iron sulphides (pyrite, marcasite, pyrrhotite), arsenic and antimony sulphides (arsenopyrite, estibinite), copper sulphides (mainly chalcopyrite), other sulphides (galena and blende) and sulfosalts, iron oxides, uranium ores, carbonaceous and

graphitic minerals, silicates and carbonates, micaceous minerals and manganous ores. Some of the processing methods are flotation associated with cyanide leaching for the tailings or cyanide leaching associated with flotation of the tailings.

In “Mineria Quimica” there is a table containing the main gold occurrences, which has been adapted to table 1.

Table 1 - Gold bearing minerals, according to Gómez - Minería Quimica

Designation	Chemical Formula
(Native) Gold	Au
<i>Electrum</i>	(Au, Ag)
Cuproauride	(Au, Cu)
Porpezite	(Au, Pd)
Rhodite	(Au, Rh)
Iridic gold	(Au, Ir)
Platinum gold	(Au, Pt)
Bismuthian gold	(Au, Bi)
Amalgam	Au ₂ Hg ₃
Maldonite	Au ₂ Bi
Auricupride	AuCu ₃
Rozhkovite	(Cu, Pd) ₃ Au ₂
Calaverite	AuTe ₂
Krenneirte	(Au, Ag) Te ₂
Montbrayite	(Au, Sb) ₂ Te ₃
Petzite	Ag ₃ AuTe ₂
Muthmannite	(Ag, Au)Te
Sylvanite	(Au, Ag)Te ₄
Kostovite	AuCuTe ₄
Nagyagite	Pb ₅ Au(Te, Sb) ₄ S ₅₋₈
Uytenbogaardtite	Ag ₃ AuS ₂
Liujinyite	Ag ₃ AuS
Aurostibite	AuSb ₂
Fischesserite	Ag ₃ AuSe

Native gold is commonly found disseminated so that the identification of mineral species usually associated with it has an important role in the various recovery stages [35]. On the table 2 there is an assembly of the main mineral associations with gold.

Table 2 - Minerals most commonly associated with gold

Name of the mineral
Arsenopyrite
Barite
Biotite
Blende
Carbonates
Chalcopyrite
Feldspars
Galena
Kaolinite
Pyrite
Quartz

Some scientists believe that the gold found on the earth's surface was once deep inside the earth and was transported during volcanic activity. The cooling of the earth's outer crust led to the appearance of many fissures and cracks from which water vapors escaped, filling those spaces with forms of silica, forming quartz veins. The same water vapors carry other elements, namely iron, silver, gold, etc. [11].

This metal is almost never a 100-percent pure, having, most of the times, other components such as: copper, silver and iron - gold containing 10% or more silver is usually called *electrum*.

The reasons why gold is associated to silver and other metals in sulphides deposits are due mainly to: difference of atomic diameter, relationship between the number of electrons transferred and the atomic diameter, crystalline system and the difference of electronegativities.

Gold exploitation is reliant on economic viability, so gold occurrence with low content should not be put aside as improvements on the extraction operating technology may also be enhanced [35].

A curious fact about this metal is that about 13.7 million tons of gold can be found in seawater, considering that the total volumes of the oceans are $1.37 \times 10^9 \text{ km}^3$ and the amount of gold in there is supposed to be 10 ng.l^{-1} . However, nobody has found and effective economic process of extraction yet. The first person who ever tried it was Fritz Haber, after First World War in an attempt of paying Germany's debts. First the gold suffered reduction by sodium polysulfide and then it was removed by sulphur-coated sand filters [16].

The features which influence the most in the choice of the auriferous ores treatment are: the particle size and minerals and tailings nature. Concerning the particle size, the figure 2.3 illustrates the possibilities for the ore treatment, adapted from Almeida (1987).

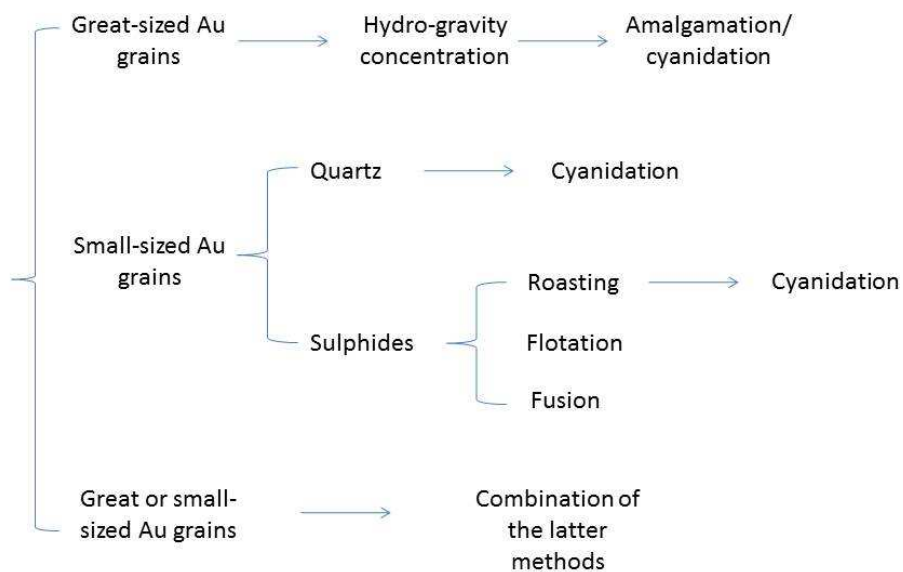


Figure 2.3 - Different procedures for the Au treatment depending on particle size

2.3 The general principles behind leaching

Leaching consists in the selective extraction of an interest component from a solid mass, using for that purpose a solvent; the solid is mixed with a liquid where the minerals dissolve.

Through this process, one of two purposes can be achieved: recover the metal by its dissolution or concentrate even more the interest ore, by leaching impurities [17].

The so called “leaching agents” can be chosen, depending both on the characteristics of the reagent, such as corroding action, selectivity, ability to regenerate and cost, or the material to be leached: physical and chemical character [18].

From the previously mentioned, one of the topics which matters the most is the selectivity of the leaching reagent, which is dependent on three parameters: temperature, contact time and concentration (of the leaching agent). Increasing the temperature sometimes has a contrary result to the expected: the level of impurities increases, but it has little impact on the leaching yield. The expansion of the contact time could also increase the percentage of impurities in solution. Regarding the leaching’s agent concentration, when it increases the dissolution of other minerals may also take place, so it is not advisable in some cases.

The most common leaching agents are water, aqueous salt solutions, acids (being the sulfuric acid one of the most important) and bases (such as sodium hydroxide). They are normally combined with an oxidant or a reductor.

The reaction’s mean velocity can be described by the following equation, knowing that C_1 is the substance concentration on the t_1 instant and C_2 its concentration on the t_2 instant [10].

$$\text{Reaction mean velocity} = \frac{C_2 - C_1}{t_2 - t_1} \quad (1)$$

However, the velocity might act differently from experiment to experiment: it can be constant - in the case of a heterogeneous reaction, where the surface of the solid does not vary -, the speed may increase over time - where the reaction product keeps reacting with the reagent -, and speed decreasing over time, which is the most common situation - the concentration of the reagents decrease.

On a heterogeneous reaction (solid-liquid) the main factors influencing the rate of the leaching process are: particle size, temperature, pulp density, rotation speed, and nature of the products [17].

The rate of leaching increases in an inversely proportional manner to particle size since the smaller the particles are, the larger is the surface area per unit weight, until full interaction is reached.

Increasing the temperature increases the leaching rate. However, the more the temperature rises, the less the process is controlled by diffusion, being chemically-controlled.

When the pulp is not very dense, the rate of leaching increases. This situation takes place when a great amount of leaching agent is added to a poor volume of solids.

The rotation speed may influence the process rate if it is diffusion-controlled. Nevertheless, the main reason for keeping the system in rotation is to prevent the solids from settling.

If an insoluble product is formed during the leaching reaction, then the rate depends on its nature: if it forms a porous layer, it will not affect the rate. However, if the solid product forms a nonporous layer the rate will decrease.

2.4 Gold leaching

The most suitable method for the processing of a gold ore is determined by many factors, such as the gold-bearing mineralogy, mineralogy of the ore, size of the speck of gold and release pattern of the gold-bearing phase [17].

Cyanide

Cyanide extraction was first used around 1887, when the MacArthur-Forrest Process was developed in Glasgow by John Stewart MacArthur.

This substance has a chemical ion that contains one carbon atom (C) and one nitrogen atom (N), represented by the chemical formula CN^- . Moreover, cyanide is a triple-bonded molecule with a negative 1 charge, where the carbon is in the +2 oxidation state and the nitrogen in the -3 oxidation state.

This technique is basically a procedure of electrochemical corrosion with Au complexation, whose speed is controlled by the diffusion of the cyanide ion (CN⁻) and oxygen (O₂).

CN⁻ is a great leaching agent, regardless of the metal. Although there are some options on the market such as sodium, potassium and calcium, sodium cyanide is preferred due to its high purity (almost 98%), among other characteristics.

Some of the characteristics to take into account in relation to this substance are present on table 3.

Table 3 - Main features of the sodium cyanide, Minería Química

Chemical formula	NaCN
Aspect	White crystals
Specific gravity	1.6
Fusion point	563.7 °C
Boiling point	1496 °C
Specific heat	0.33 kcal/kg °C
Solubility (at 15 °C)	34.2 g/100g of solution

In industrial plants, the usual feed is 15-25% solution (on weight), with about 2% of sodium hydroxide (or other base) as a pH stabilizer. The hazard temperatures are over 40 °C (release of toxic-gases) and under 4 °C (crystals began to appear) [15].

Before scrutinizing the leaching process, it is important to analyze the gold stability in aqueous solutions using Pourbaix diagrams.

Metallic gold includes the entire water stable domain and the Au⁺ ion does not appear in the following diagram (figure 2.4) once it turns spontaneously into Au (III) and metallic gold, according to the equation [10]:



Au³⁺ and other oxidized gold forms only occur to potentials above the higher threshold of water stability (represented by the dotted-lines). However, in this diagram area, water is oxidized to oxygen and the oxidized gold species are reduced to metallic state.

Gold cannot be oxidized in strong bases or acids in the absence of complexing ligands.

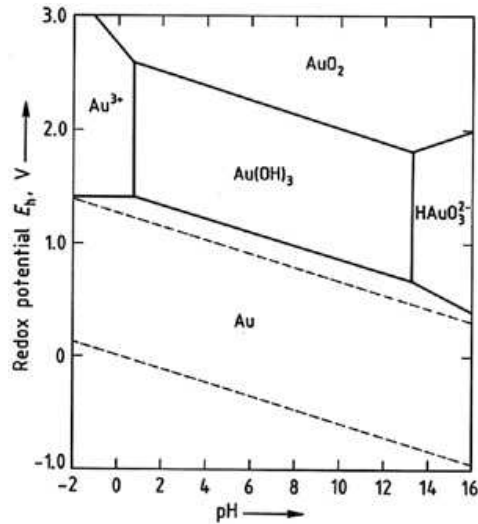


Figure 2.4 - Eh-pH diagram for the system Au-O₂-H₂O

Despite being a very stable metal, the presence of cyanide as complexing agent originates the large stability area for the $Au(CN)_2^-$ (figure 2.5) as long as there is enough potential for oxidation, leading to the effectiveness of the cyanidation process.

A main characteristic for the gold system is that the stability region of the auro-cyanide complex, $Au(CN)_2^-$ depends on the cyanide concentration: it grows with the growth in cyanide concentration and declines with the increase in the dissolved metal concentration [37].

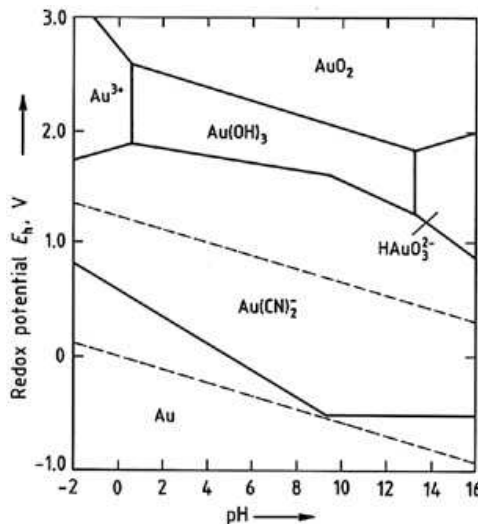


Figure 2.5 - Eh-pH diagram for the system Au-CN-H₂O

The kinetics of heterogeneous reaction aims to identify the variables and steps which affect the reaction's speed and quantify the effects of those variables. Despite being thermodynamically very favored, the gold dissolution using cyanide is really slow in terms of reaction speed. On top of that, the rate of this process cannot be increased significantly, because gold is passivated by cyanide.

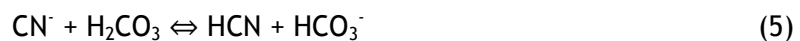
According to Souza (2002), one of the variables which influence that rate is the cyanide concentration: the reaction speed increases with the increasing of cyanide concentration. At a low cyanide concentration, the dissolution rate depends only on the cyanide concentration. On the other hand, having high cyanide concentrations, the rate rests only on the oxygen pressure [18]. However, the optimal concentration depends on the ore mineralogy and is determined case by case.

With relation to oxygen, some of the factors which exert a decisive role on the reaction speed are: the oxygen concentration in solution (depending, mostly, on the partial pressure and temperature), the transfer of oxygen in the reaction interface (conditioned by the pulp and the rheology of the dispersion) and, finally, the existence of parallel reactions, reagent consumers, etc. The oxygen solubility increases with the increase of the gas pressure and decreases with the temperature increase.

It is crucial that the cyanide solution be kept alkaline (studies show that the pH range should be from 11 to 12) during the gold leaching for the reason that it prevents the hydrolysis of the cyanide ion [18].



And also prevents the cyanide decomposition by atmospheric carbon dioxide:



In both cases HCN, hydrogen cyanide, a deadly gas, is released.

An efficient agitation also favors the diffusion of the cyanide reagents and oxygen to the interface reaction because it decreases the thickness of the boundary layer.

The particle size of the ore influences directly its recovery, because the contact with the leaching solution is vital for the implementation of the process.

On the other hand, the comminution can expose mineral substances detrimental to cyanidation, increasing the reagents consumption [33].

The pulp dilution is also important because the more diluted it is, better the contact between the gold and the leaching agent (cyanide) is; the only inconvenience being the need of bigger agitation tanks and other equipment.

As previously mentioned, increasing the temperature, increases the rate of dissolution, but the solubility of oxygen in the solution decreases. Therefore, there is an optimal temperature for which the rate hits its maximum. On the figure 2.6 there is a practical example of this theory, however, using potassium cyanide (KCN) [18].

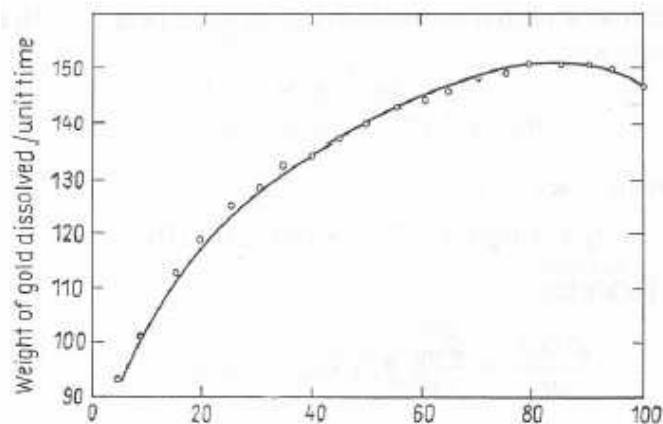


Figure 2.6 - Effect of temperature on the rate of gold dissolution in a 0.25% KCN solution

According to the previous image, the optimal temperature is approximately 85 °C. From that temperature on, the weight of gold dissolved per unit of time starts decreasing.

Likewise, Julian and Smart as well found out in their studies that 85 °C could be the optimal temperature, but there are some records indicating that 80 °C could also be a fair temperature. A fact which should be mentioned is that above 110 °C cyanide decomposition becomes a relevant factor [33].

Regarding the process itself, when a solid gets immersed in a liquid solution, its contact surface forms a 0.03 mm thick, stagnant layer (Nernst Boundary Layer) through which the reagents have to diffuse before they get to the interface in which they will react. Summarizing the procedure, the reagents are brought to the Nernst boundary layer, where their molecules diffuse. The solid adsorbs those reagents, reacting with them. A desorption occurs in the solid's surface and the reaction products are transported to the surface's exterior once again through the Nernst boundary layer [10].

Following the classical model of diffusion for fluid phase, Habashi (1970) proposes an equation that explains that the cyanide ion and the oxygen diffuse into the gold surface, where they are absorbed, reacting with the metal and creating the peroxide ion, as well as the cyanide complex. Afterwards they leave the reaction interface, spreading into the solution.

Although this theory explains the oxygen and cyanide diffusion control, its practical application hampered the need of estimating the surface area of the gold particles in ore, the Nernst boundary layer thickness and the diffusion coefficients [19].

Brittain (1975) suggests that at the same time the cyanidation process decreases the gold concentration on the solid, the resistance of the metal dissolution increases. For that matter, the equation explaining the process is:

$$E = E_0 (1 - a[Au]) \quad (6)$$

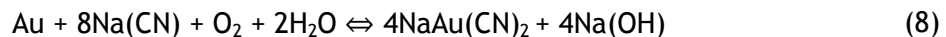
Where E is the dissolution resistance, E_0 is the same resistance when [Au] is approximately zero, a is a constant and Au is the gold concentration of the ore.

This “resistance” is related to the activation energy and it can be replaced into the Arrhenius equation (which is a formula for the temperature dependence of reaction rates) [18].

$$k = k_0 e^{-E/RT} = k_0 e^{-E_0 (1 - a[Au])/RT} \quad (7)$$

Where k is the velocity constant, R is the gas constant (8.314 J/mol.K); k_0 is the Arrhenius constant and T is the temperature in Kelvin. However, using this equation is not easy because of the difficulty of quantifying the “a” constant.

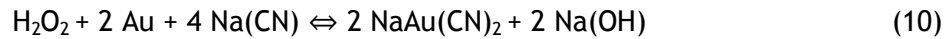
In the understanding of the process of cyanidation, the greater contributions are from Elsner (1846), who noticed that the gold dissolution in cyanide solutions requires the presence of oxygen [18].



And Bodlaender (1896) who suggested that the process of dissolution would take place in two stages [33]:



The hydrogen peroxide (H_2O_2) reacts again resulting in



Nonetheless in 1934, through the calculation of the free formation energies (known as Gibbs) of the complexes, by Barsky, Swainson and Hedley, it was possible to conclude that the equation which fitted best was the Bodlaender's [33].

In the case of gold ores containing organic matter (refractory to cyanidation) they need the preference of a stronger oxidant processing, otherwise the carbon reabsorbs the gold. Nonetheless, the prior destruction of that organic matter by oxidation, chlorine or sodium hypochlorite allows the usual procedures in cyanidation [10].

Along the practice of cyanidation several interferences were detected, due the presence of some elements, namely [35].

- Silver - high cyanide concentrations can lead to the formation of $\text{Ag}(\text{CN})_3^{2-}$ and even $\text{Ag}(\text{CN})_4^{3-}$. Despite having similar behavior during cyanidation, they dissolve at different velocities. It is usual to find some Au-Ag compounds having 15-35% of silver within the cyanidation tailings.
- Copper - almost every copper minerals dissolve easily with cyanide. $\text{Cu}(\text{CN})_3^{2-}$ is one of the most likely compounds to form.

Regarding the leaching process, the first stage is crushing the ore into a powder so that the small gold particles become exposed, and then water is added. This mixture is reacted with cyanide in the presence of oxygen.

Gold leaching may need two stages of oxidation: one of them is pre-aeration (which includes the agitation of the pulp in an alkaline environment), overcoming the chemical need of oxygen by minerals such as pyrrhotite or other sulphides and ventilation to provide oxygen for the leaching reactions [18].

The availability of oxygen in the solution reduces the time needed for the leaching process, increases recovery and reduces cyanide consumption.

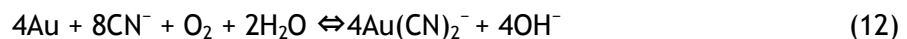
From an economic point of view, the introduction of air is less onerous than oxygen, but taking into account the previous factors, capital and operational costs of the compressors surpass the use of oxygen generators on site and high-yield reactor.

The gold dissolution in cyanide includes two reactions: an anodic (gold oxidation) and a cathodic (oxygen reduction). The first one occurs in alkaline solutions where gold is oxidized and dissolved forming the Au(I) cyanide complex. This reaction can be affected positively or negatively by the presence of impurities in the leaching solution: for instance, heavy metals such as mercury and lead can have a positive effect, by reducing passivation of the gold surface, while the presence of sulfur forms creates a passive layer on the gold surface, reducing its oxidation [24]. In the cathodic dissolution reaction in cyanide solutions, hydrogen peroxide forms as a strong oxidizing agent, taking part in further oxidation reactions [18].

The oxygen may be reduced directly to hydroxide ions, as the following equation suggests [24].



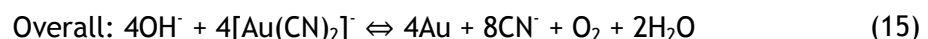
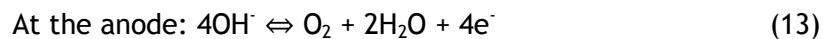
The overall dissolution reaction can then be described as [24].



In subsequent hydrometallurgical unit operations for gold processing, it is necessary to concentrate the metal, so that the gold in solution can be converted back to solid gold. One of the most efficient ways of doing such is using activated carbon, since most of impurities remain in solution.

The final procedure is the recovery and refining, which occurs at 110 °C mixing the carbon with sodium hydroxide (NaOH) and sodium cyanide (NaCN), creating a new solution of NaAu(CN)₂ - elution.

The gold is converted to its elemental form by a reaction know as electrowinning and this process can be divided into the following stages [18].



The gold then precipitates as a fine black mud, which is smelted and poured into moulds in order to form bars.

It is known that sulphides are a common constituent of gold ores and its decomposition during the leaching process using cyanide creates two main sources of contaminants: the metallic cations and sulfur compounds.

Another problem is that species such as Cu, Fe, Ni, Co, Zn, Cd, As and Sb tend to form ionic complexes with cyanide, reducing the availability of cyanide for gold dissolution [26].

The technical difficulties associated with gold leaching, more precisely when leaching pyrite and arsenopyrite are determined mainly by the small size of the mineral gold host and not by the interference of arsenic and sulphur compounds as expected.

The techniques for leaching gold ores can be divided basically into two groups [33]:

- Percolating leaching or heap leaching - this technique is used in small gold deposits, low content ores. Still, it requires that the ore has a specific processing. The recoveries are not as high as the ones obtained through dynamic leaching.
- Dynamic (agitation) leaching - this type of leaching is the most common. Despite its higher investment and operation costs, it allows up very high recoveries.

Toxicity of Cyanide Compounds

Cyanide is poisonous with its toxicity depending on the type of cyanide complexes that are present.

There is a lot of uncertainty related with cyanide compounds noxiousness due to the limitations of routine analytical techniques for measuring cyanide as well as the presence of breakdown cyanide forms in mining waste waters [20].

Mining-related waters usually contain mixtures of potentially toxic metals along with the cyanide (and its related compounds) so the exact determination of which are harmful is difficult.

According to the European Economic Community Council Directive of 15th July, 1980, relating to the quality of water intended for human consumption, the maximum admissible cyanide concentration (in water) is 50 µg/l.

“Acute toxicity” is defined by the cyanide concentrations which lead to the death of more than 50% of the test population, within 96 hours. For instance: for fish these concentrations are lower than they are for birds (micrograms per liter against milligram per liter). Not only this kind of exposure is deadly but also the chronic exposure may affect reproduction, physiology and activity levels.

The liquid or gaseous hydrogen cyanide as well as cyanide salts can penetrate the body through inhalation, ingestion or absorption (eyes or skin contact) and it is readily distributed throughout the body via blood. The initial symptoms of cyanide poisoning may include headache, dizziness, redness of the face, nausea and vomiting. The late symptoms are slow and irregular heartbeat, the body temperature drops and the lips, face and the extremities of the body become blue. In case of no immediate assistance, the person can go into a coma [21].

In conclusion, the greatest fears about cyanide are the health and environmental problems associated with it, including the formation of deadly hydrogen cyanide gas, the ingestion or adsorption through skin of cyanide salts and the formation of free forms of cyanide in effluent water [26]. These are the main reasons that lead to a growing need to find an alternative reagent to cyanide.

Alternative substances to cyanide

The uppermost challenge in producing a suitable substitute for cyanide in gold processing is the development of an equally effective and degradable leaching reagent, which has less environmental concerns.

Some of the most appealing substances to do so are considered next.

Chloride

The use of chlorine (Cl) happened when the ores had weak response to gravity concentration and amalgamation. It was first used by Plattner in 1851.

This method is based on the fact that, in the presence of moisture, it complexes gold to AuCl_3 (soluble in water).

Chloride can be precipitated from solutions by sulfur dioxide (SO_2), hydrogen sulfide (H_2S) or adsorbed on coal; this last one requiring high loadings (greater than 50 kg/tonne) [23].

Until the appearance of cyanide, it existed several industrial processes using chlorine: process Plattner and the process Deetken, among others.

Gold is leached by chlorine at low pH and high temperatures, and those are the optimal conditions.

This process consists of applying chlorine gas into moistened minerals, more precisely into the amalgamation waste and the roasting products of pyrites, transforming it into a soluble solution in water [35].

The reaction occurs in two stages: on the first Au(I) is formed on the gold surface. The second stage is referent to the AuCl_2^- formation.

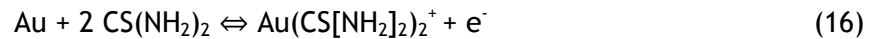
Although this dissolution occurs faster than the one using cyanide, low concentrations of sulfides can increase the reagent consumption and the incomplete dissolution of sulfides will result in the reduction of the soluble gold [22].

This type of processing requires very acidic conditions, the reason why this method is not commercially applied.

Thiourea

Thiourea $\text{SC}(\text{NH}_2)_2$ is perhaps the reagent with the greatest potential to replace cyanide: it poses less of a threat given its toxicity (the lethal dose is 10 g/kg for humans) and makes the reaction occur faster than cyanide, when referring to gold dissolution (up to 12 times faster). It is usually supplied in a powder form and its fusion point is between 180-182 °C [25].

Under acidic conditions (pH between 1 and 3), thiourea dissolves gold, forming a cationic complex. The anodic reaction follows the equation [28].



In order to adjust pH it is recommended the use of sulphuric, hydrochloric or nitric acid, therefore the corrosion of the equipment occurs sooner.

This substance has a high rate of degradation by oxidation (its consumption is twice that of cyanide, in the best of circumstances) and it costs at least four times as much [27].

On top of that, this process is not sufficiently studied, regarding the recovery of metal in solution.

Despite its low toxicity, thiourea is not a safe reagent: it is a thyroid poison (for humans) and recognized as potentially carcinogenic [22].

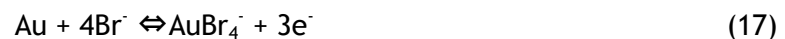
Another disadvantageous feature is its low selectivity for gold over gangue minerals, when in comparison to cyanide as well as the complexity of the regeneration and purifying procedures. It is also associated with high detoxification costs, has a very limited recyclability, and features process parameters that are difficult to control [28].

Bromine

Bromine (Br) was first introduced as a solvent for gold in 1846.

Elemental bromine is perilous and difficult to transport and store, so that sodium bromide is the substance used in the leaching process [25]. It can be added to the leaching solution, along with hypochlorite (as oxidant) and chlorine, converting bromide to bromine [22].

The gold dissolution occurs according to the following equation:



Some of the aspects which affect the gold dissolution are the concentrations of gold and bromine itself, pH and the electrochemical potential of the reaction.

The main advantages of this substance are its fast extraction, its action occurs in a wide range of pH and it is not toxic. On the other hand, the disadvantages are its high consumption and great cost.

Iodine

Iodine (I) leaching had been used to recover gold in the late 1800s and early 1900s.

This substance leaches gold in a wide pH range and can be applied in very low concentrations. It has a large capacity of penetration into ore, with low uptake when referring to gangue particles and it is non-toxic.

During the dissolution process insoluble gold iodine may form, and hinder further reaction between gold itself and the iodine solution.

The gold dissolution rate depends upon the iodine concentration and the ionic strength [23].

There are three methods: Prichard method, Harrison method and in-situ leaching. The first one consists of using an excess of iodine dissolved in potassium iodide in an aqueous solution. The Harrison method uses an aqueous solution of iodine and potassium iodide, plus nitric acid (to prevent the formation of insoluble gold salts). In situ leaching is safer as the gold is recovered by activated charcoal [25].

Thiosulphate

Thiosulphate ($S_2O_3^{2-}$) has been considered a potential substitute for cyanide in recent years since it causes fewer environmental impacts, therefore fewer pollution concerns [28].

Moreover, thiosulphate has a low toxicity with a LD_{50} (needed dose to kill half of a population) of 7.5 g/kg for mice.

The main chemical components derived from this leaching process are ammonium thiosulphate and ammonium sulphate, which are common non-harmful fertilizers.

The leaching process using this substance is enhanced by the presence of copper ions as they increase the thiosulphate oxidation rate [22]. The copper speeds up the leaching reaction 18 to 20 times [34].

The addition of ammonia to thiosulphate solution has improved gold dissolution and reduced its consumption, by creating soluble ammine complexes. On top of that, the ammonia stabilizes the copper (II) in solution, according to the equation [24].



Nevertheless, it should be noted that ammonia is a volatile and noxious substance, which can escape easily from vessels.

The chemistry of the ammonia-thiosulphate (figure 2.7) is relatively complex due to side reactions which may occur. For instance: reduction of copper (II) to copper (I) and the decomposition of thiosulphate into different species of polythionates - from a pH range of 8.5 to 9, tetrathionate and pentathionate are the main reaction products and at 10.4 trithionate is generated [24].

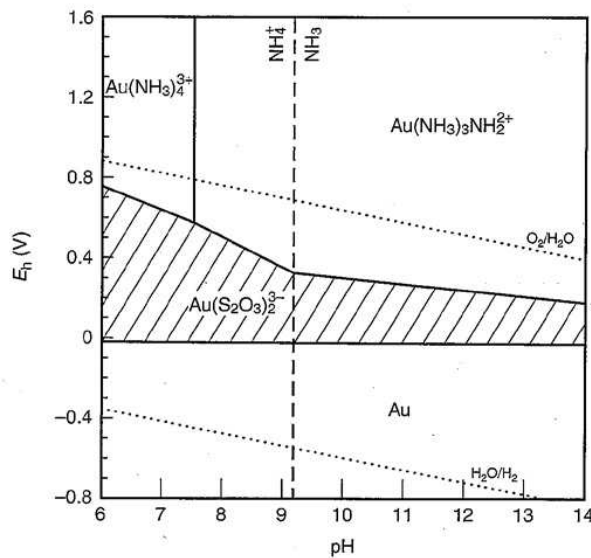
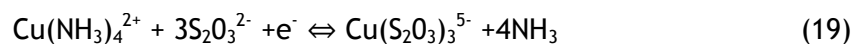


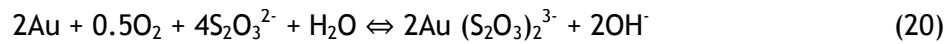
Figure 2.7 - Eh-pH diagram for the Au-NH₃-S₂O₃²⁻-H₂O

The relation between cupric and cuprous species in the ammoniacal solution is shown in the following equation [30].



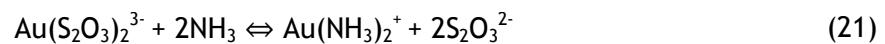
The major factors affecting the rate of dissolution are the temperature of the process and the concentrations of thiosulphate as well as the dissolved oxygen. A study from 2002 (carried out by Molleman and Dreisinger) determined that using air or oxygen in a thiosulfate solution has a positive effect on gold extraction but it decreases thiosulfate stability.

Gold forms a stable anionic complex with thiosulphate, according to the subsequent reaction [28].



This reaction must occur in alkaline conditions to prevent thiosulphate decomposition by acid action.

In an ammoniacal thiosulphate solution, both thiosulphate and ammonia have the aptitude to form complexes with gold. The following equation shows this ability [24].



Some of the disadvantages of using this substance are its high consumption, due to several reactions such as oxidation, and the lack of a suitable gold recovery method [29].

Thiosulphate is the only reagent being consumed during the leaching process in a 40-50% rate [30].

3. CASTROMIL SAMPLES

3.1. About Castromil Mines

Castromil mines are located in the north of Portugal, in Oporto's district, more precisely in Paredes.

The municipality of Paredes is about 157 km² long and has a population of 85 428 inhabitants. It is limited by Paços de Ferreira on the north side, Lousada and Penafiel on the east, Gondomar on the southwest and Valongo by the west, figure 3.1.

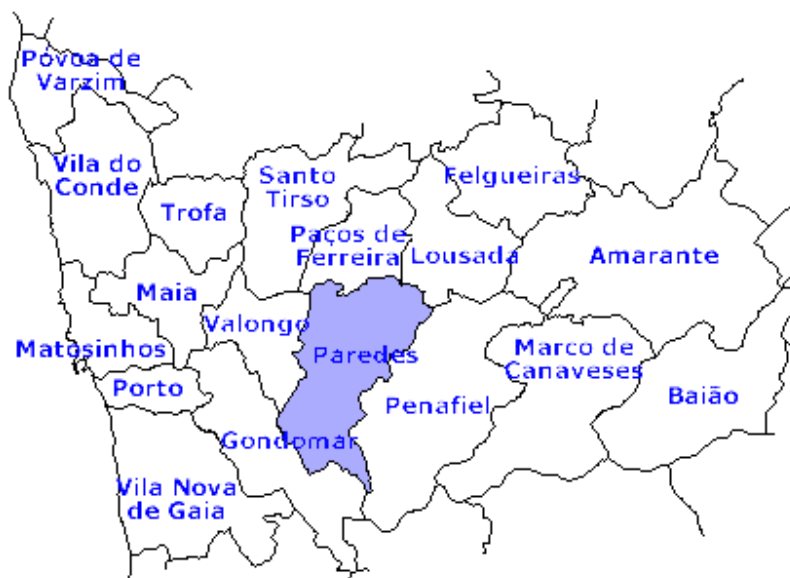


Figure 3.1 - Paredes location on the Oporto's district

The complete profile of Paredes is the combination of two geologic maps: 9-D (Penafiel) and 13-B (Castelo de Paiva).

Castromil mines refers to auriferous deposits consisting of two different mass minerals separated by Sousa River: Covas de Castromil, located at northwest of the river and Serra da Quinta, situated on southwest. Covas de Castromil can be seen on the figure 3.2.

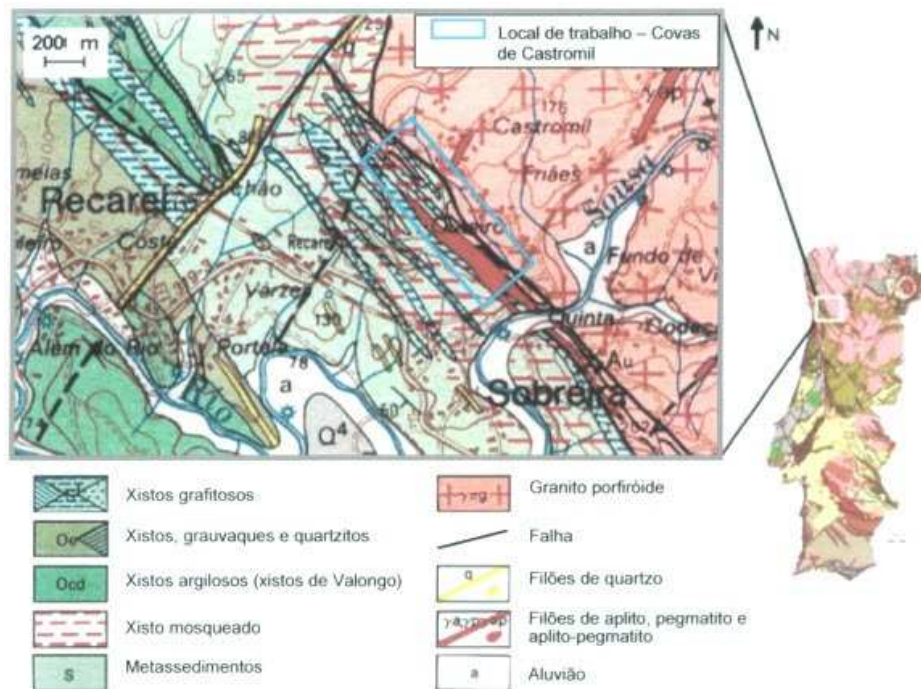


Figure 3.2 - Covas de Castromil on the geologic map

This region does not exceed the 670 meters above sea level, with the Valongo hills, in the southwest area, reaching 300-400 meters. These hills are composed by quartzite and greywacke, above a shale bed with NW-SE direction.

In the southeast there are granitic rocks, crossed by the Tâmega River (in the NE-SW direction) [38].

The gold in Portuguese deposits mainly occurs in association with sulphides, the most important being arsenopyrite. Apart from Castromil, other portuguese mines are Jales, Penedono, Latadas and Serra da Lousã.

During the Variscan orogeny, most of the total area was covered by the appearance of granitic rocks, which exhibited fractures with two dominant directions: NW-SE (coincident with the long axis of the granite massifs) and NE-SW, where masses of aplite, pegmatite, apelite-pegmatite and quartz as well as dykes embedded themselves [36].

Castromil gold deposit is located in one of those apelite masses, and its mineralization occurs in the granite, taking advantage of the existing faults for the sulphide deposition. This process appears to have occurred in two distinct phases: in the first one, pyrite and arsenopyrite seem to have deposited in association with carbonic fluids (resulting from the interaction between water and metasediments with organic matter). Later, in a second phase, gold and bismuth precipitated in the fractures.

In the Castromil area the contact between Silurian sediments and granitic rocks coincides with a northwest trending shear zone (against which the granite has been thrust over the metasediments), which had the purpose of draining the mineralized fluids movement.

The gold occurs in the form of microscopic grains on the surface, inside of microfractures of the pyrite or included in secondary oxides and it is disseminated along veins in the silicified granite, commonly associated with sulphides. It is thought that this mineralization was induced by a series of fractures and faults.

There are two types of ore, considering this location: the first one consists of sulphides abundant in this region, previously mentioned and slightly oxidised (arsenopyrite and pyrite) and the second type shows the same sulphides disseminated in the quartz.

Portugal was one of the prime locations for mining held by the Romans, who exploited preferably gold, iron and copper. They mastered some techniques such as underground mining and even opencast holdings.

Since that time and until 1941, Castromil stood abandoned, and no activity was performed there. During five years, “Minas do Ouro do Douro” explored the region, but in 1946 they stopped, due to lack of funds.

Eighteen years later, a Canadian company, “Noranda of Canada”, tried its luck, which little success because of 1966 drop in the price of gold.

Some economic feasibility studies were made during 1973, by “Anglo American”, but they concluded that the small size of the deposit, added to the market conditions of the time, did not enable the exploitation of the mines on a profitable way.

From 1988 to 1992, “Minas do Douro” conducted the exploration of that place and in 1994, “Connary Minerals” concluded, through various studies, that the exploitation of Castromil was feasible and they made a proposal which was denied by the Portuguese government during 2000 due to environmental issues (Silva and Félix, 2009).

In January 2014, Medgold acquired the licence for the mine exploitation.

3.2. Castromil samples

In this research, two different materials were considered: the first, a less oxidized sample designated CSQ10 (a “sulphide”, figure 3.3) and the second, a more oxidized labeled CSQ30 (an “oxide”, figure 3.4). Both were taken from the mining gallery number 2 (figure 3.5).



Figure 3.3 - CSQ10 sample



Figure 3.4 - CSQ30 sample



Figure 3.5 - Gallery number 2 from where the samples were collected

According to Anamet's (an entity providing consultation and metallographic preparation) report from 1994, both samples are similar when referring to mineralogical composition and appearance. However, the degree of mineralization is variable, dominated by rich phases of iron, with greater amounts of primary sulphide minerals on the CSQ10 than on the CSQ30 sample.

Regarding the primary ore minerals, the weathering made sulphide, sulpharsenide and arsenide minerals become oxidized and decomposed, with their ions being removed to groundwater or re-precipitated.

ALS Laboratory Group, S.L on February of 2015 sent a report on those samples, showing its chemical compositions: tables 4 and 5.

Table 4 - Chemical composition of the sample CSQ10 performed by ALS Laboratory

CSQ10	
Ag (ppm)	52.8
Al (%)	0.32
B (ppm)	<10
Ba (ppm)	70
Be (ppm)	<0.5
Bi (ppm)	94
Ca (%)	0.01
Cd (ppm)	<0.5
Co (ppm)	<1
Cr (ppm)	6
Cu (ppm)	94
Fe (%)	9.47
Ga (ppm)	<10
Hg (ppm)	<1
K (%)	0.12
La (ppm)	10
Mg (%)	<0.01
Mn (ppm)	38
Mo (ppm)	<1
Na (%)	0.01
Ni (ppm)	1
P (ppm)	370
Pb (ppm)	3490
S (%)	4.51
Sb (ppm)	48
Sc (ppm)	1
Sr (ppm)	5
Th (ppm)	<20
Ti (%)	<0.01
Tl (ppm)	<10
U (ppm)	10
V (ppm)	41
W (ppm)	<10
Zn (ppm)	4
Au (ppm)	9.23
As (%)	1.91

Table 5 - Chemical composition of the sample CSQ30 performed by ALS Laboratory

CSQ30	
Ag (ppm)	38.1
Al (%)	0.86
B (ppm)	<10
Ba (ppm)	220
Be (ppm)	4.4
Bi (ppm)	175
Ca (%)	0.01
Cd (ppm)	3.2
Co (ppm)	3
Cr (ppm)	18
Cu (ppm)	379
Fe (%)	20.4
Ga (ppm)	<10
Hg (ppm)	2
K (%)	0.15
La (ppm)	10
Mg (%)	0.01
Mn (ppm)	32
Mo (ppm)	28
Na (%)	0.01
Ni (ppm)	10
P (ppm)	2950
Pb (ppm)	5620
S (%)	0.41
Sb (ppm)	112
Sc (ppm)	1
Sr (ppm)	19
Th (ppm)	<20
Ti (%)	<0.01
Tl (ppm)	<10
U (ppm)	40
V (ppm)	97
W (ppm)	<10
Zn (ppm)	90
Au (ppm)	15.75
As (%)	3.4

From the analysis of the table 5 the oxide sample (CSQ30) consists of materials derived from the surface weathering, resulting in low sulphur content (0.41%), which on the sulphide sample is much greater (4.51%). It is also observable that the oxide has much more gold than the sulphide (15.75 against 9.23 ppm).

The high arsenic (As) content indicates that As-bearing phases such as arsenopyrite are present in both samples. This arsenopyrite has almost unaltered discrete, well-formed, rhomb-shaped crystals. Scorodite can also be a weathering product of arsenopyrite, which, because of its porosity, may enclosure small particles of gold.

A significant degree of ferruginisation can be explained by former Fe-rich phases such as pyrite. Its presence is in the form of discrete, euhedral crystals and it can exhibit replacement by jarosite and goethite because of the extensive weathering suffered by both ore samples. Jarosite is an alteration product of pyrite, consisting of iron (Fe), with some amounts of sulphur (S) and minor potassium (K) [3].

Goethite aggregates commonly display a moderate degree of porosity, being very permeable, a detail with enormous importance considering leaching tests - these spaces can enclose small gold particles. It is considered to be the dominant phase in the heavy mineral concentrates, intergrowing with quartz and muscovite [4]. The figure 3.6 was taken from the Anamet Services' report and shows the gold-goethite relationship on a computer enhanced electron image of the nature and appearance of a highly porous grain of gold (yellow) intergrown with a goethite aggregate (pinkish red).

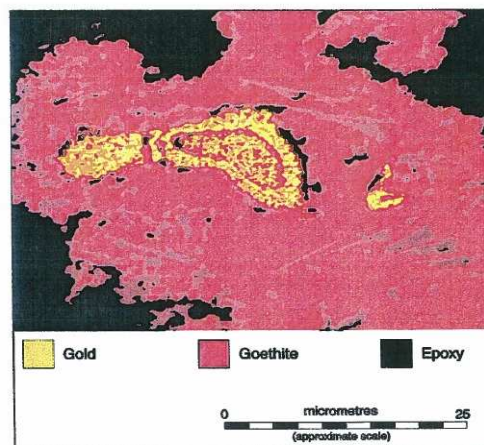


Figure 3.6 - Gold-goethite relationship

Both arsenopyrite and pyrite are important since they represent a common host phase for gold or even silver.

Small inclusions of loellingite and galena (usually within the pyrite grains) can also be found within both ore samples.

On the CSQ10 ore there are some traces of bismuthinite and metallic bismuth (portrayed as inclusions within the arsenopyrite).

Concerning the gangue minerals, the host material is dominated by quartz both on the form of mineral grains and fragments of veins and slightly oxidized fine to medium grained muscovite.

What's more, it concluded that CSQ10 was a sample of massive sulphides while CSQ30 was a mixture for both sulphides and oxides.

3.3. Gold: nature and occurrence mode

Within these ore samples, the gold can occur in three ways: primary metallic gold, supergene gold and refractory gold.

According to Anamet's there are individual particles of gold, with size under 50µm (fine grained) and tend to occur in clusters of larger particles, one of them being goethite aggregates. It can be also present in discrete stringers or extended bodies, sometimes reflecting prior fractures. The presence of a high proportion of this fine type of gold indicates that it is appropriated to leaching tests.

Small particles of *electrum* (a mixture of gold and silver) can be found growing among scorodite and it is believed that they grow the same way, with the *electrum* becoming enclosed within arsenopyrite. Figure 3.7 shows this relationship on a computer enhanced electron image as well as the appearance of a cluster of small *electrum* (yellow) within the scorodite (greenish blue).

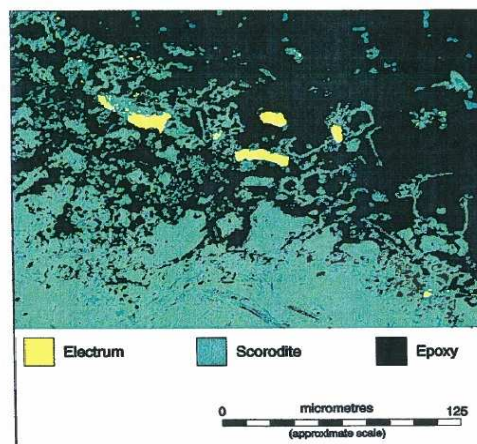


Figure 3.7 - Electrum-scorodite relationship

Supergene gold is also found growing within the goethite and developed within the oxidation zone.

The remaining refractory gold is abridged within the arsenopyrite and pyrite and it is less than 5% of the total and it occurs in the form of small gold and *electrum* particles but it also can be attached to quartz.

In the sulphide sample, the complex nature of the Au-bearing particles may be the result of the decomposition of electrum: it decomposes in oxide environments to form metallic gold, silver halide minerals and silver bearing sulphides. The metal gold might nucleate, forming very irregular and porous aggregates.

4. TECHNICAL DESCRIPTION AND PROCEDURES

SAMPLE PREPARATION

To perform the leaching experiments two different ore samples were used: CSQ10 and CSQ30 both described in the previous chapter.

Each lot of material was separated on 5 sub-samples.

Sub-sample 1

The first step was to crush the material in the crushing rolls (figure 4.1) and a particle size analysis (figure 4.2) was carried out using the ASTM (American Society for Testing and Materials) sieve series: 4# (4.76 mm) 5# (3.36 mm), 8# (2.36 mm), 12# (1.68 mm), 16# (1.19 mm), 20# (0.841 mm), 30# (0.59 mm), 40# (0.42 mm), 50# (0.297 mm), 70# (0.21 mm), 100# (0.149 mm), 140# (0.105 mm) and 200# (0.074 mm), weighting the retained material in each as well as the *passant* on the 200#.



Figure 4.1 - Crushing rolls: lateral view (left) and front view (right)



Figure 4.2 - Sieve series used in the particle size analysis

For each ore sample, the results appear in tables 6 and 7.

Table 6 - Particle size analysis for CSQ10 sub-sample 1 using the crushing rolls

Size (mm)	Sieve fractions		Cumulative (%)
	wt (g)	wt (%)	
	0.00	0.00	1.00
4.760	50.42	0.02	0.97
3.360	138.10	0.07	0.90
2.360	237.77	0.13	0.78
1.680	274.20	0.15	0.63
1.190	216.91	0.12	0.52
0.841	169.77	0.09	0.43
0.590	133.71	0.07	0.36
0.420	130.05	0.07	0.29
0.297	115.46	0.06	0.23
0.210	95.15	0.05	0.18
0.149	71.08	0.04	0.14
0.105	66.94	0.04	0.10
0.074	45.14	0.02	0.08
<0.074	150.50	0.08	0.00
Sum	1895.2	1.00	---

Table 7 - Particle size analysis for CSQ30 sub-sample 1 using the crushing rolls

Size (mm)	Sieve fractions		Cumulative (%)
	wt (g)	wt (%)	
	0.00	0.00	1.00
4.760	28.32	0.03	0.97
3.360	70.85	0.07	0.91
2.360	139.56	0.13	0.77
1.680	170.01	0.16	0.61
1.190	132.86	0.13	0.48
0.841	97.25	0.09	0.39
0.590	71.71	0.07	0.32
0.420	64.05	0.06	0.26
0.297	53.76	0.05	0.210
0.210	46.30	0.04	0.17
0.149	34.57	0.03	0.13
0.105	31.43	0.03	0.10
0.074	21.78	0.02	0.08
<0.074	86.84	0.08	0.00
Sum	1049.29	1.00	---

Analyzing the previous tables, for both ore sample the d_{80} was 2.36 mm, which means that 80% of the feed material was under 2.36 mm.

To carry out these experiments, according to previous works (Akashi and Hishikari mines [2]), the particle size should be considerable smaller (under $74\mu\text{m}$). The material above 0.074mm was then put into the cutting mill (figure 4.3) to continue the particle size reduction process.

A second particle size analysis took place using the sieve series: 12# (1.68 mm), 16# (1.19 mm), 20# (0.841 mm), 30# (0.59 mm), 40# (0.42 mm), 50# (0.297 mm), 70# (0.21 mm), 100# (0.149 mm), 140# (0.105 mm) and 200# (0.074 mm) and the same methodology was applied.



Figure 4.3 - Cutting mill used in the particle size reduction

For each ore sample, the results appear in tables 8 and 9.

Table 8 - Particle size analysis for CSQ10 sub-sample 1 using the cutting mill

Size (mm)	Sieve fractions		Cumulative (%)
	wt (g)	wt (%)	
	0.00	0.00	1.00
1.680	2.98	0.003	0.99
1.190	1.58	0.002	0.99
0.841	1.70	0.002	0.99
0.590	2.69	0.003	0.99
0.420	6.34	0.006	0.99
0.297	9.28	0.01	0.98
0.210	13.05	0.01	0.96
0.149	129.89	0.13	0.84
0.105	33.44	0.03	0.80
0.074	15352	0.15	0.65
<0.074	672.52	0.65	0.00
Sum	1026.99	1.00	---

Table 9 - Particle size analysis for CSQ30 sub-sample 1 using the cutting mill

Size (mm)	Sieve fractions		Cumulative (%)
	wt (g)	wt (%)	
	0.00	0.00	1.00
1.680	0.10	0.00	0.99
1.190	0.25	0.00	0.99
0.841	0.61	0.00	0.99
0.590	2.28	0.00	0.99
0.420	9.94	0.01	0.98
0.297	15.76	0.02	0.97
0.210	55.99	0.06	0.90
0.149	88.90	0.10	0.80
0.105	73.55	0.08	0.72
0.074	88.55	0.10	0.62
<0.074	551.04	0.62	0.00
Sum	886.97	1.00	---

Analyzing the former tables, for the CSQ10 ore sample the d_{80} was 0.105 mm and for the CSQ30 ore sample was 0.149 mm, which leads to the conclusion that the feed material needed another particle size reduction.

On the figure 4.4 all of the cumulatives for the multiple processes are represented, showing the particle size evolution obtained from the equipment used.

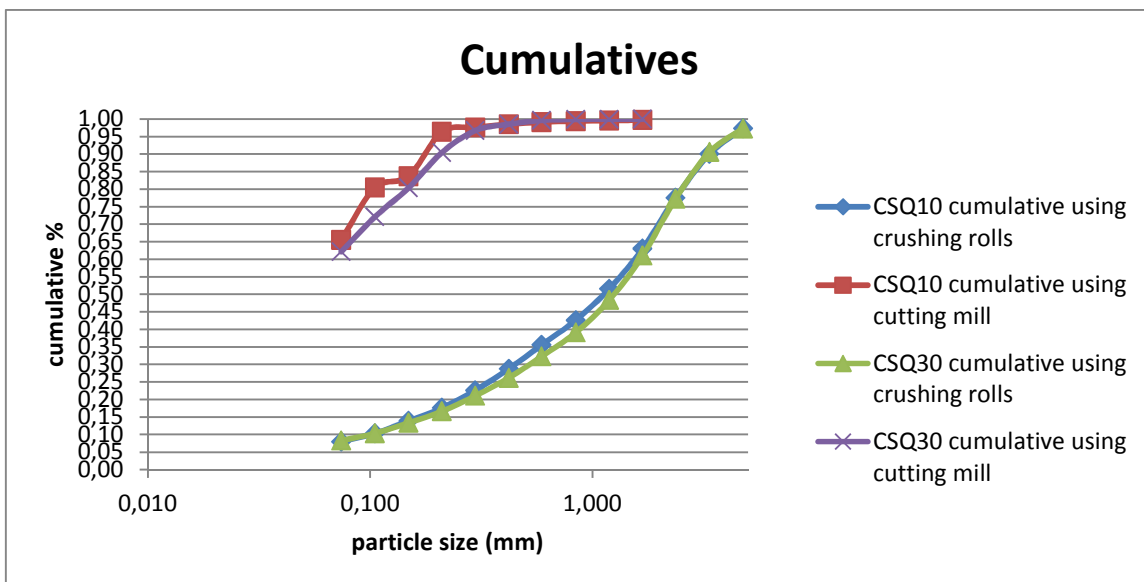


Figure 4.4 - Different cumulatives for the particle size reduction process, regarding both ore samples

For both ore samples, there are considerable changes in the geometric form of the cumulative, showing the variation of the particle size. CSQ10 and CSQ30 have similar cumulatives when comparing the same equipment and vary almost in the same way when considering the other. The reduction of the particle size is noteworthy when comparing the crushing rolls and the cutting mill for both ore samples.

As previously mentioned, another particle size reduction had to be performed. Not knowing the gold distribution within the different fractions of the material, all of it was put in the vibratory disc mill (figure 4.5) for a final size reduction.



Figure 4.5 - Disc mill used in the particle size reduction

The Mastersizer 2000 (figure 4.6) (laser diffraction) was used for the determination of the particle size for the final lots, since it is the appropriated equipment to determine small calibers up to $0.24 \mu\text{m}$.

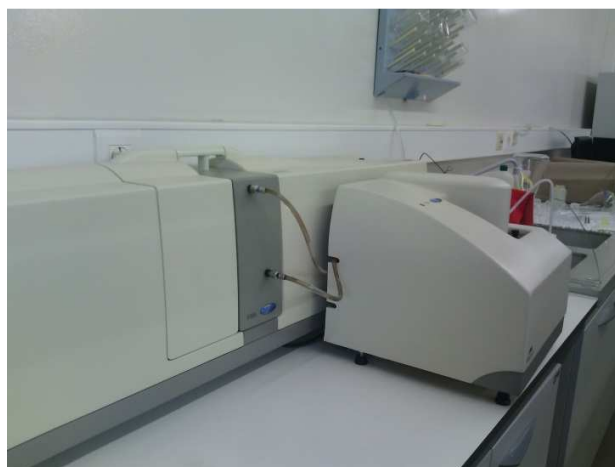


Figure 4.6 - Mastersizer 2000

The results obtained by using this equipment are shown in table 10 and figure 4.7 for the CSQ10 sub-sample and in table 11 and figure 4.8 for the CSQ30 sub-sample.

Table 10 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ10 sub-sample 1

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	8.78	11.482	53.86
0.011	0.00	0.120	0.00	1.259	9.93	13.183	58.48
0.013	0.00	0.138	0.00	1.445	11.11	15.136	63.13
0.015	0.00	0.158	0.00	1.660	12.38	17.378	67.78
0.017	0.00	0.182	0.00	1.905	13.79	19.953	72.36
0.020	0.00	0.209	0.00	2.188	15.37	22.909	76.85
0.023	0.00	0.240	0.00	2.512	17.14	26.303	81.19
0.026	0.00	0.275	0.01	2.884	19.14	30.200	85.28
0.030	0.00	0.316	0.12	3.311	21.37	34.674	89.06
0.035	0.00	0.363	0.53	3.802	23.88	39.811	92.40
0.040	0.00	0.417	1.16	4.365	26.66	45.709	95.22
0.046	0.00	0.479	2.01	5.012	29.74	52.481	97.42
0.052	0.00	0.550	3.01	5.754	33.12	60.256	98.95
0.060	0.00	0.631	4.12	6.607	36.79	69.183	99.81
0.069	0.00	0.724	5.29	7.586	40.75	79.433	99.98
0.079	0.00	0.832	6.47	8.710	44.94	91.201	100.00
0.091	0.00	0.955	7.64	10.000	49.33	104.713	100.00

From the former table it possible to conclude that:

- 100% of the material is under 91.201 µm
- D_{80} is 26.303 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The figure 4.7 illustrates the particle size distribution for this sample.

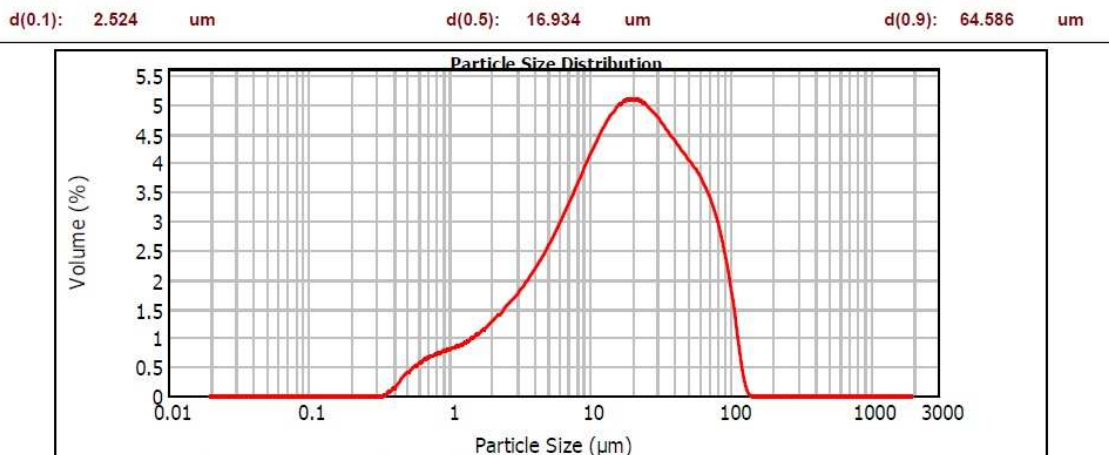


Figure 4.7 - Particle size distribution, using Mastersizer 2000 for the CSQ10 sub-sample 1

Table 11 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ30 sub-sample 1

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	3.85	11.482	37.85	120.226	99.87
0.011	0.00	0.120	0.00	1.259	4.64	13.183	41.99	138.038	100.00
0.013	0.00	0.138	0.00	1.445	5.49	15.136	46.34	158.489	100.00
0.015	0.00	0.158	0.00	1.660	6.43	17.378	50.85	181.970	100.00
0.017	0.00	0.182	0.00	1.905	7.48	19.953	55.44	208.930	100.00
0.020	0.00	0.209	0.00	2.188	8.65	22.909	60.04	239.883	100.00
0.023	0.00	0.240	0.00	2.512	9.95	26.303	64.56	275.423	100.00
0.026	0.00	0.275	0.00	2.884	11.40	30.200	68.96	316.228	100.00
0.030	0.00	0.316	0.00	3.311	13.01	34.674	73.19	363.078	100.00
0.035	0.00	0.363	0.01	3.802	14.79	39.811	77.24	416.869	100.00
0.040	0.00	0.417	0.10	4.365	16.77	45.709	81.11	478.630	100.00
0.046	0.00	0.479	0.35	5.012	18.96	52.481	84.80	549.541	100.00
0.052	0.00	0.550	0.73	5.754	21.40	60.256	88.31	630.957	100.00
0.060	0.00	0.631	1.22	6.607	24.10	69.183	91.60	724.436	100.00
0.069	0.00	0.724	1.80	7.586	27.09	79.433	94.58	831.764	100.00
0.079	0.00	0.832	2.43	8.710	30.38	91.201	97.08	954.993	100.00
0.091	0.00	0.955	3.12	10.000	33.97	104.713	98.94	1096.478	100.00

From the former table it possible to conclude that:

- 100% of the material is under 138.038 µm
- D_{80} is 45.709 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The figure 4.8 illustrates the particle size distribution for this sample.

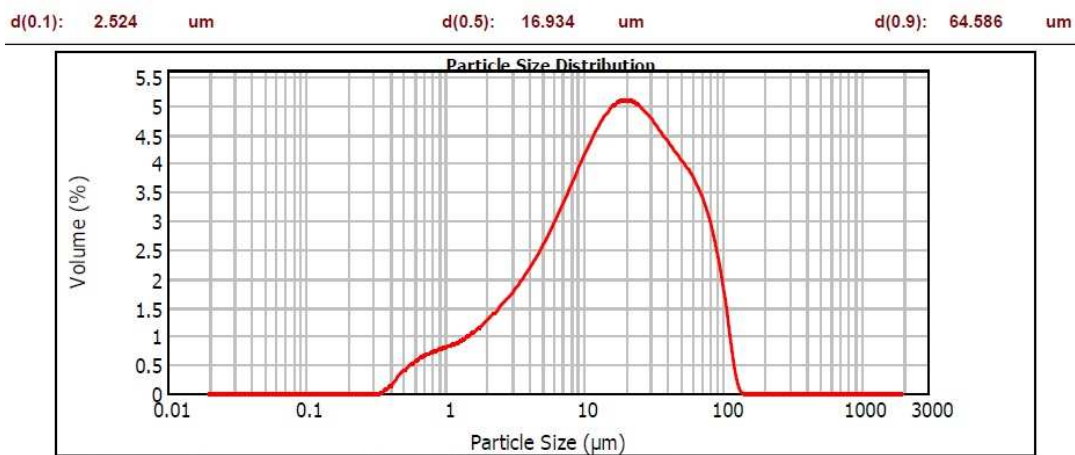


Figure 4.8 - Particle size distribution, using Mastersizer 2000 for the CSQ30 sub-sample 1

The final weight of CSQ10 sub-sample 1 was 1026.99 g and CSQ30 sub-sample 1 was 886.97 g.

Before using the material for the proposed assays, the chemical composition was determined by a portable X-ray fluorescence analyzer, Innov-X (figure 4.9).

The purpose of using such equipment was to control the chemical elements, in order to find out if the leaching process occurs. However, this machine is not capable of measuring the gold concentrations. That analysis was performed later through the flame atomic absorption spectrometer carried out in a specialized laboratory, LNEG (Laboratório Nacional de Energia e Geologia).



Figure 4.9 - Portable X-Ray Fluorescence Analyzer used to determine chemical composition

The results obtained by using this equipment are shown in tables 12 and 13 for CSQ10 sub-sample and CSQ30, respectively.

Table 12 - Chemical composition for the CSQ10 sub-sample

CSQ10	ppm	+/-
S	98041	11519
Ti	3323	386
Fe	>10%	1%
Co	708	210
Cu	265	20
As	22256	331
Pb	3563	65
Rb	156	5
Zr	66	4
Ag	242	19
I	<854	
Ca	<1169	
K	<8903	
Cl	<10957	
Pb	<113223	
Ba	<481	
Cr	<249	
Mn	<165	
Ni	<109	
Zn	<33	
Hg	<62	
Se	<22	
Sr	<8	
Mo	<13	
Cd	<64	
Sn	<109	
Sb	<115	

Table 13 - Chemical composition for the CSQ30 sub-sample

CSQ30	ppm	+/-
Ti	8037	1871
Fe	>10%	12%
Cu	718	66
Zn	129	35
As	37735	1303
Pb	6037	230
Rb	136	11
Zr	68	10
Ag	378	45
I	<3614	
Ca	<5150	
K	<30061	
Cl	<60681	
S	<114861	
Pb	<497010	
Ba	<2086	
Cr	<1007	
Mn	<712	
Co	<2439	
Ni	<338	
Hg	<159	
Se	<52	
Sr	<19	
Mo	<31	
Cd	<145	
Sn	<236	
Sb	<249	

Sub-sample 2

The material was put into the cutting mill and then in the vibratory disc mill.

A particle size analysis was carried out using Mastersizer 2000 and a chemical analysis was performed using the portable X-ray fluorescence.

The final weight of CSQ10 sub-sample 2 was 644.10 g and for the CSQ30 sub-sample 2 was 884.98 g.

The results for this sub-sample, when compared to the previous are not that different, so, from this sample on, all of them will be presented in the Appendix A.

Sub-sample 3

The methodology for this lot of ore was the same carried out for the previous one.

The weight of CSQ10 sub-sample 3 was 839.08 g and for the CSQ30 sub-sample 3 was 859.82 g.

All of the results are in the same appendix (Appendix A).

Sub-sample 4

The methodology for this lot was the same carried out for the previous one.

The weight of CSQ10 sub-sample 4 was 860.81 g and for the CSQ30 sub-sample 4 was 1008.23 g.

Sub-sample 5

Until this stage, the size reduction was taking plenty of time because the ore would not enter in the mill's chamber properly. For that reason, both lots were put directly into the disc mill.

A particle size analysis was carried out using Mastersizer 2000 and a chemical analysis was performed using the portable X-ray fluorescence.

The weight of CSQ10 sub-sample 5 was 839.08 g and CSQ30 sub-sample 5 was 1084.6 g.

APPLICATION OF THE AMMONIACAL-THIOSULPHATE SYSTEM TO CASTROMIL ORES

Some experiments were carried out with the intent of studying various parameters on gold leaching using a thiosulphate-ammoniacal solution.

The usage of double reagents for the preparation of the leaching solution is not by chance. According to Aylmore and Muir (2000), the use of ammonia prevents the formation of sulfur coatings (result of the thiosulphate's decomposition). In the absence of ammonia gold passivation occurs. However, its main purpose is to stabilize the copper, working as a catalyzer for the process, speeding it up to 18-20 times.

The conditions stipulated for the control experiment were, based on previous works:

- Solid percentage: 40%
- Thiosulphate concentration: 0.5 M
- Ammonia concentration: 1 M
- Copper concentration: 0.001 M
- Medium rotation speed (usually corresponds to 400 rpm) - on the agitator this "medium" was regulated by level 1 of agitation.
- Room temperature
- Residence time: 5 hours

The used equipment did not mention any rotation speed; it only indicated that it varied between 250 and 2200 rpm. Having 11 velocity positions, by interpolation it possible to consider that:

- ✓ 0 - 220 rpm
- ✓ 1 - **445 rpm**
- ✓ 2 - 640 rpm
- ✓ 3 - 835 rpm
- ✓ 4 - 1030 rpm
- ✓ 5 - 1225 rpm
- ✓ 6 - 1420 rpm
- ✓ 7 - 1615 rpm
- ✓ 8 - 1810 rpm
- ✓ 9 - 2005 rpm
- ✓ 10 - 2200 rpm

The control experiment provides results to compare with the following assays.

During the tests, pH and oxidation-reduction potential (ORP) was measured, maintaining the level of the first on 11 by adding sodium hydroxide 20 M. Alkaline solutions must be used to prevent the decomposition of thiosulphate (leading to a poor leaching process).

ORP is a potentiometric measurement very similar to Eh, as the two of them quantify the potential of the medium to transfer electrons. However, ORP is a much less specific designation and its measurement can be made relatively to any reference electrode. In this case, it was used a KCl (potassium chloride) 3 mol/L solution, so the final values for redox potential had to be converted, depending on the solution temperature. The reference table for these values is from the operating manual of the electrode, table 14.

Table 14 - ORP electrode operating manual, converter table for the Eh parameter

T (°C)	U _{Ref} [mV]		T (°C)	U _{Ref} [mV]	
	SenTix® ORP SenTix® Au	SenTix® PtR		SenTix® ORP SenTix® Au	SenTix® PtR
0	+224	+221	35	+200	+187
5	+221	+216	40	+196	+181
10	+217	+212	45	+192	+176
15	+214	+207	50	+188	+171
20	+211	+202	55	+184	+165
25	+207	+197	60	+180	+160
30	+203	+192			

The parameters which were varied from the control assay were:

- Residence time: 8 hours
- Thiosulphate concentration: 0.1 M, 1 M and 2 M
- Copper concentration: 0.0001 M and 0.01 M
- Ammonia concentration: 2 M and 3 M
- Temperature: 40-45 °C and 60-70 °C
- Speed rotation: level zero

A standard test was performed at the beginning using only a thiosulphate solution in order to study the importance of adding ammonia and copper.

As one of the main concerns was the ammonia vapors, every experiment took place in the laboratory's hotte.

Assay 1: Standard test

Every ore samples from sub-sample 1 (250 g) was mixed with a 375 mL solution of thiosulphate 0.5 M, kept at room temperature and level 1 rotation speed during 5 hours in order to determine whether the leaching was effective or not, figure 4.10.



Figure 4.10 - Leaching assays of CSQ10 (on the left) and CSQ30 (on the right)

Samples were collected from time to time: 1 hour later, 3 hours later and then, by the end of the experiment, 5 hours later.

Afterwards each mixture was filtrated (figure 4.11) and then washed using 350mL of distilled water. For each filtration, a vacuum pump was used in order to improve experimentation time and to optimize the filtration itself and a sample was collected.



Figure 4.11 - Filtration of CSQ10 (on the right) and of CSQ30 (on the left)

Only the pH was measured during the tests, since the redox potential electrode was broken at the time. The experiment was performed at room temperature.

After the leaching process, for each ore sample, the solid material was weighted and then it was put into the oven to dry for about 24 hours. Later, it was weighted again and from the difference of water's weight the relative humidity was determined.

Then, an X-ray fluorescence analysis was done to the residue in order to determine if the leaching process took place.

Assay 2: Influence of time on the leaching process

This experiment derives from the control assay: the solution used was composed by thiosulphate 0.5 M, ammonia 1 M and copper 0.001 M.

The volume of 375 mL of solution was mixed with 250 g of sub-sample from the first lot.

The duration of the test was expanded to 8 hours, collecting samples 1 hour later, 3 hours later, 5 and then 8 hours later. During the process the temperature was kept at room's and the speed rotation level was 1.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and a sample was collected.

Both pH and redox potential were measured during the test.

The solid material was weighted for both ore samples and then it was put into the oven, drying for about 24 hours. Later, it was weighted again and an x-ray fluorescence analysis was done.

This assay was repeated for CSQ30 ore sample only since the results were not the expected. The sub-sample used was number 4 and the procedure was the same. Therefore, the results shown are about this repetition and not the original experiment.

Assay 3: Varying thiosulphate concentration to 0.1 M

During this phase, the solution used was composed by thiosulphate 0.1 M, ammonia 1 M and copper 0.001 M.

The volume of 375 mL of solution was mixed with 250 g of sub-sample: the CSQ10 sample was from the sub-sample 2 while the CSQ30 was from the sub-sample 1.

The duration of the assay was 5 hours and only then a sample was collected.

This assay occurred at room temperature and the speed rotation was kept at level 1 on the agitator.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and a sample was collected.

Both pH and redox potential were measured during this experiment.

The solid material was weighted wet and dry for both ore samples and then an X-ray fluorescence analysis was done. For the drying process, an oven was used for 24 hours.

Assay 4: Varying thiosulphate concentration to 1 M

The 375 mL solution mixed with 250 g of each sub-sample from lot 2 was composed by thiosulphate 1 M, ammonia 1 M and copper 0.001 M.

The duration of the assay was 5 hours and the agitation speed was kept at level 1.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and a sample was collected by the end of every procedure.

Both pH and redox potential were measured during this assay, which took place at room temperature.

The solid material for each ore sample was weighted (wet and dry, after being 24 hours in an oven) and an X-ray fluorescence analysis was done.

Assay 5: Varying thiosulphate concentration to 2 M

A 375 mL solution composed by thiosulphate 2 M, ammonia 1 M and copper 0.001 M was mixed with 250 g of each sub-sample from lot 2.

The duration of the assay was 5 hours, room temperature, and level 1 of speed rotation.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and by the end of every steep, a sample was collected.

Both pH and redox potential were measured during this test.

The solid material was weighted wet and dry for both ore samples and then an X-ray fluorescence analysis was done. For the drying process, an oven was used for 24 hours.

Assay 6: Varying copper concentration to 0.0001 M

For this experiment, 250 g of each sub-sample was mixed with a 375 mL solution composed by thiosulphate 0.5 M, ammonia 1 M and copper 0.0001 M. The process occurred at room temperature and both of the sub-samples were from lot 2.

The duration of the assay was 5 hours and a sample was collected by the end of it.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and a sample was collected.

Both pH and redox potential were always measured during the leaching test.

The solid material was weighted, for both ore samples, wet and then dry, after being in the oven for about 24 hours, and then an X-ray fluorescence analysis was done.

Assay 7: Varying copper concentration to 0.01 M

To perform this experiment, 250 g of each sub-sample from lot 3 was mixed with a 375 mL solution composed by thiosulphate 0.5 M, ammonia 1 M and copper 0.01 M.

The leaching assay occurred at room temperature and had the duration of 5 hours, always at level 1 of speed rotation.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and for each step of the process samples were collected.

Both pH and redox potential were measured during this process.

The solid material was weighted wet and dry for both ore samples and then an X-ray fluorescence analysis was done. For the drying process, an oven was used for 24 hours.

Assay 8: Varying ammonia concentration to 2 M

A 375 mL solution composed by thiosulphate 0.5 M, ammonia 2 M and copper 0.01 M was mixed with 250 g of ore sample. The CSQ10 was from lot 3 and the CSQ30 from lot 4.

The leaching experiment occurred at room temperature and had the duration of 5 hours. The speed rotation was always kept at level 1.

Each of the mixtures was filtrated and then the cakes were washed using 350 mL of distilled water. For each filtration, a vacuum pump was used and samples were collected from every step of the experiment.

Only pH was measured since the ORP electrode was broken.

The solid material was weighted, for both ore samples, wet and then dry, after being in the oven for about 24 hours, and then an X-ray fluorescence analysis was done.

Assay 9: Varying ammonia concentration to 3 M

A 375 mL solution composed by thiosulphate 0.5 M, ammonia 3 M and copper 0.01 M was mixed with 250 g of ore sample.

The speed rotation was always kept at level 1 and the experiment occurred at room temperature, having the duration of 5 hours, measuring pH only.

The CSQ10 was from lot 4 and the CSQ30 from lot 3.

Each of the mixtures was filtrated and then the cakes were washed using 350mL of distilled water. For each filtration, a vacuum pump was used and a sample was collected.

The solid material was weighted wet and dry for both ore samples and then an X-ray fluorescence analysis was done. For the drying process, an oven was used for 24 hours.

Assay 10: Varying temperature between 60 and 70 °C

A 375 mL solution composed by thiosulphate 0.5 M, ammonia 1 M and copper 0.001 M was mixed with 250 g of ore sample each one from lot 4.

This experiment occurred during 5 hours and the temperature was kept between 60 and 70 °C, using a heating mantle for that purpose figure 4.12.



Figure 4.12 - Leaching assays of CSQ10 (on the left) and CSQ30 (on the right)

While in level 1 of speed rotation, the system became quite unstable (because of the container's lid). In order to control that instability, it had to be reduced to 0 level (220 rpm).

Both pH and redox potential were measured during the leaching assay as well as the temperature.

Each of the mixtures was filtrated and then the cakes were washed using 350mL of distilled water. For each filtration, a vacuum pump was used and a sample was collected.

The solid material was weighted wet and dry for both ore samples and then an X-ray fluorescence analysis was done. For the drying process, an oven was used for 24 hours.

Assay 11: Varying temperature between 40 and 45 °C

For this experiment, a 375 mL solution composed by thiosulphate 0.5 M, ammonia 1 M and copper 0.001 M was mixed with 250 g of ore sample each one from lot 4.

The temperature was kept between 40 and 45 °C, using a heating mantle.

This test occurred during 5 hours and the speed of rotation was kept at 0 for the previous stated reasons.

Both pH and redox potential were measured during the leaching assay as well as the temperature.

The solid material was weighted wet and dry for both ore samples and then an X-ray fluorescence analysis was done. For the drying process, an oven was used for 24 hours.

Assay 12: Varying rotation speed

In order to study the agitation, which had to vary for the reasons stated above, a final assay was carried varying only this parameter which was on its lowest. All the other parameters were equal to the control experiment.

A 375 mL solution composed by thiosulphate 0.5 M, ammonia 1 M and copper 0.001 M was mixed with 250 g of ore sample each one from lot 5.

This experiment occurred during 5 hours, at room temperature.

Both pH and redox potential were measured during the leaching test as well as the temperature.

The solid material was weighted, for both ore samples, wet and then dry, after being in the oven for about 24 hours, and then an X-ray fluorescence analysis was done.

The table that follows summarizes de set of experiments.

Table 15 - Assembly of experimentation

Concentrations (M)			Temperature (°C)	Speed rotation (level)
thiosulphate	copper	ammonia		
0.5	0	0	room temperature	1
0.5	0.001	1	room temperature	1
0.1	0.001	1	room temperature	1
1	0.001	1	room temperature	1
2	0.001	1	room temperature	1
0.5	0.0001	1	room temperature	1
0.5	0.01	1	room temperature	1
0.5	0.001	2	room temperature	1
0.5	0.001	3	room temperature	1
0.5	0.001	1	60-70	0
0.5	0.001	1	40-45	0
0.5	0.001	1	room temperature	0

5. RESULTS AND DISCUSSION

APPLICATION OF THE AMMONIACAL-THIOSULPHATE SYSTEM TO CASTROMIL ORES

For each assay, pH and ORP (Oxidation-Reduction Potential) were measured and corrected whenever possible.

As previously mentioned, pH was tried to be kept at 11 by adding sodium hydroxide because it prevents the decomposition of thiosulphate (leading to a poor leaching process).

Nevertheless, the main purpose of this series of experiments was to determine the yield of leaching and then compare it to the ones using cyanide (in a future work).

There are two ways of calculating such results: through the liquid concentration (main objective of the sample collection) or through the concentration of the residues for each experiment.

Through liquid examination, the expression that shows the yield of the process is:

$$\begin{aligned} \text{yield} &= \frac{\text{Au mass in solution}}{\text{Au mass in the feed material}} \\ &= \frac{\text{concentration of Au} \times \text{volume of solution}}{\text{Au mass in the feed material}} \quad (22) \end{aligned}$$

On the other side, the expression for the yield through the residue is given by:

$$\text{yield} = \frac{\text{Au content in feed} - \text{Au content in the residue}}{\text{Au content in feed}} \quad (23)$$

Having calculated both, the yield through liquid showed small agreement of results, maybe because the gold captured precipitated again onto the residue or the analytical method was not well fitted. Consequently, only the results given by the residue based calculation are going to be displayed on this chapter. However, the methods for determining these concentrations imply a 25-30% error so some results had to be also ignored, as they implied some impossible conclusions.

Despite this, the liquid base calculations are going to be presented on the Appendix C.

5.1 Standard test (assay 1)

The purpose of this experiment was to study the need of adding copper and ammonia to the thiosulphate solution.

During this test, the Eh electrode was broken so that the only readings are from pH.

The readings are on tables 16 and 17.

Table 16 - Standard test, measures for the CSQ10 sample

CSQ10	
Initial pH:	6.32
NaOH added:	70 drops (10M)
Final pH:	10.70

pH 1h later:	9.80
NaOH added:	5 drops (20M)
Final pH:	10.90

pH 2h later:	10.90
NaOH added:	---
Final pH:	---

pH 3h later:	10.40
NaOH added:	3 drops (20M)
Final pH:	10.90

pH 4h later:	10.40
NaOH added:	3 drops (20M)
Final pH:	11.10

pH 5h later:	10.50
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Table 17 - Standard test measures, for the CSQ30 sample

CSQ30	
Initial pH:	6.09
NaOH added:	60 drops (10M)
Final pH:	10.50

pH 1h later:	9.90
NaOH added:	5 drops (20M)
Final pH:	10.90

pH 2h later:	10.40
NaOH added:	5 drops (20M)
Final pH:	11

pH 3h later:	10.40
NaOH added:	5 drops (20M)
Final pH:	10.90

pH 4h later:	10.60
NaOH added:	5 drops (20M)
Final pH:	10.90

pH 5h later:	10.60
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For the oxidized sample (CSQ30), the pH varied more considerably. In some cases both pH are the same, but according to the table 17 it took superior volumes of sodium hydroxide to keep it more a less constant.

To continue the study of the leaching process, it was necessary to compare the results from the chemical analysis before and after the leaching, since the main objective is to have a selective leaching (gold only).

The results are presented on tables 18 and 19. For the following tests, the analysis are portrayed on the Appendix B, since it is easier to read the graphics than the tables.

Table 18 - Chemical analysis for the CSQ10 sample after leaching, standard test

CSQ10	ppm	+/-
S	>10%	3%
Ti	4393	845
Fe	>10%	2%
Cu	140	28
As	9846	264
Pb	1927	67
Rb	65	6
Zr	34	6
Ag	103	32
I	<1827	
Ca	<2977	
K	<19806	
Cl	<39401	
P	<284266	
Ba	<953	
Cr	<521	
Mn	<364	
Co	<743	
Ni	<131	
Zn	<43	
Hg	<70	
Se	<25	
Sr	<11	
Mo	<22	
Cd	<108	
Sn	<178	
Sb	<190	

Table 19 - Chemical analysis for the CSQ30 sample after leaching, standard test

CSQ30	ppm	+/-
S	24561	7975
Ti	3057	373
Fe	>10%	1%
Co	1569	363
Cu	422	27
Zn	53	14
As	21657	362
Pb	4696	91
Rb	73	4
Zr	35	4
Ag	179	21
I	<813	
Ca	<951	
K	<5638	
Cl	<9246	
P	<101985	
Ba	<480	
Cr	<218	
Mn	<164	
Ni	<159	
Hg	<72	
Se	<24	
Sr	<9	
Mo	<15	
Cd	<71	
Sn	<118	
Sb	<126	

Having all the values, the next step was to put them into graphical form. To do so, only the elements associated with exact values were used (for example: Ti, Cu, As, ...).

The portable analyzer can only read the chemical composition up to 10%, so, when a given element is above that percentage, the composition is not known, therefore, cannot be compared to anything specific. The elements with the minor symbol also cannot be used because there are not exact.

It was possible to build the histograms from the figure 5.1 and figure 5.2 with these elements.

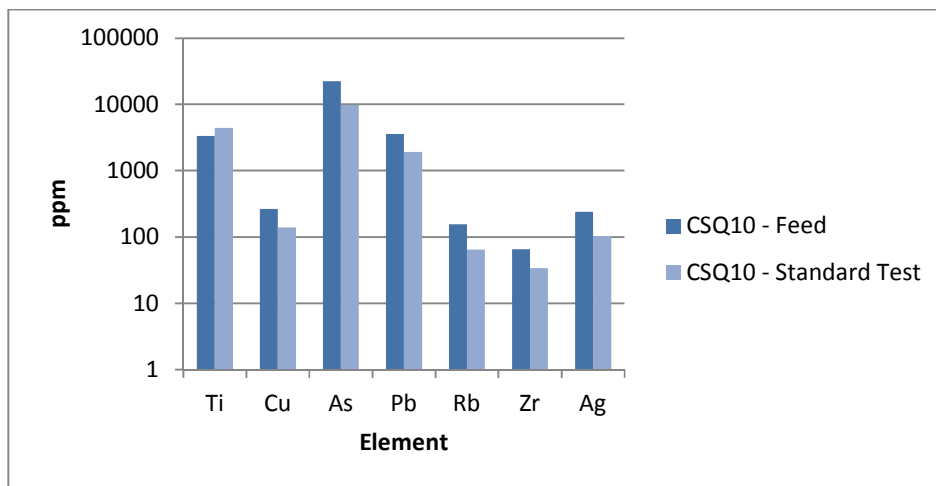


Figure 5.1 - Comparison of the chemical analysis for the CSQ10 sample before and after the standard leaching test

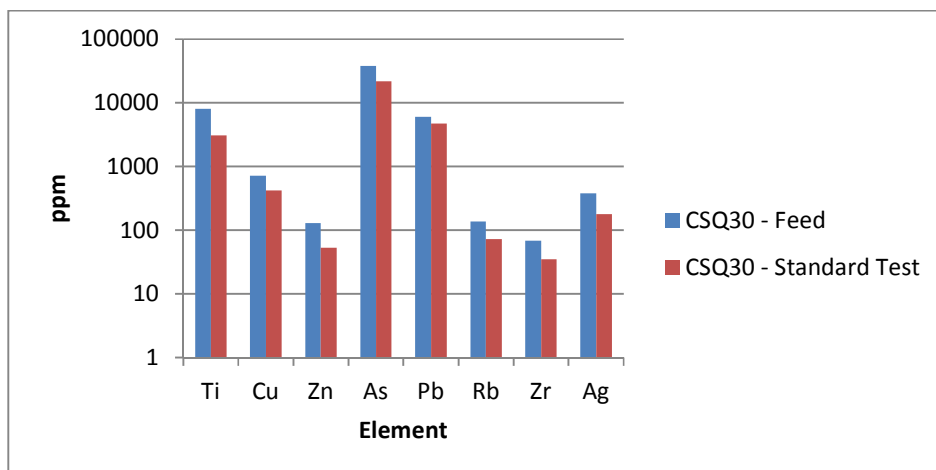


Figure 5.2 - Comparison of the chemical analysis for the CSQ30 sample before and after the standard leaching test

From the previous figures, it is possible to say that leaching occurred for the elements analyzed. However, in the CSQ10 sample, titanium did not behave as expected, having its concentration rise.

This can be due to the uncertainty of the measure, which is about 845ppm for this case.

The conclusion is that the leaching was not selective (for gold) as other elements were also leached.

To determine the gold content of the used ore in the different assays (CSQ10 and CSQ30), the ore was homogenized and then 5 samples for analysis were collected. These values are shown in table 20.

Table 20 - Concentration feeds from sub-sample 1 to sub-sample 5, for both CSQ10 and CSQ30 ore samples

	Sub-sample 1	Sub-sample 2	Sub-sample 3	Sub-sample 4	Sub-sample 5	Mean
CSQ10 (mg/kg Au)	8.2	4.3	2.7	3.3	9.0	5.5
CSQ30 (mg/kg Au)	6.1	12	9.4	9.4	8.0	8.98

From the previous table it is observable that the concentrations obtained were very different, which may suggest that some problems on the sampling or sample homogenization occurred.

However it has to be considered that gold analysis (in solid samples) is difficult due to the “nugget effect”: while a portion of the material may have a high gold concentration, the other may not have as much. Consequently, despite the very different results, it was considered that the gold content on the initial ore sample was the average of all the values obtained for the 5 taken samples.

As stated on Chapter 3, the content of gold (Au) is higher for the CSQ30 sample than for the CSQ10, which can be proven reading the table 20.

The results obtained at the laboratory (residues) revealed that:

- Gold concentration for the CSQ10 sample: 4.6mg/kg
- Gold concentration for the CSQ30 sample: 5.5mg/kg

Applying the expression for the yield (23):

$$yield = \frac{Au \text{ content in feeding} - Au \text{ content in the residue}}{Au \text{ content in feeding}}$$

CSQ10 ore sample:

$$\text{yield} = \frac{5.5 - 4.6}{5.5} \times 100 = 16.36\%$$

CSQ30 ore sample

$$\text{yield} = \frac{8.98 - 5.5}{8.98} \times 100 = 38.75\%$$

The leaching yield was higher for the second sample than it was for the first one, confirming that the sulphide is harder to leach than the oxide.

Furthermore using a thiosulphate only solution, the results are far from what is expected, as supported through theory.

5.2 Influence of time on the leaching process (assay 2)

The aim of this experiment was to study the influence of time on the leaching experiment. What's more, this test serves the purpose of being the control assay. The solution used was composed by sodium thiosulphate (0.5 M), ammonia (1 M) and copper (0.001 M).

Regarding this assay, both pH and ORP parameters were measured and then ORP was converted to Eh according to table 14.

Table 21 - Influence of time, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.8
NaOH added:	15 drops (20M)
Final pH:	11.02
ORP (mv):	-216.90
Eh potential (mV):	-5.90

pH 1h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.01
ORP (mv):	-75.00
Eh potential (mV):	136.00

pH 2h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.2
ORP (mv):	-63.50
Eh potential (mV):	147.50

pH 3h later:	10.50
NaOH added:	10 drops (20M)
Final pH:	11.20
ORP (mv):	-74.80
Eh potential (mV):	136.20

pH 4h later:	10.60
NaOH added:	5 drops (20M)
Final pH:	11.10

Table 22 - Influence of time, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.50
NaOH added:	30 drops (20M)
Final pH:	10.90
ORP (mV):	-88.50
Eh potential (mV):	122.50

pH 1h later:	10.70
NaOH added:	25 drops (20M)
Final pH:	10.95
ORP (mV):	-5.70
Eh potential (mV):	205.30

pH 2h later:	10.40
NaOH added:	25 drops (20M)
Final pH:	11.01
ORP (mV):	-40.90
Eh potential (mV):	170.10

pH 3h later:	10.46
NaOH added:	25 drops (20M)
Final pH:	11.01
ORP (mV):	-61.10
Eh potential (mV):	149.90

pH 4h later:	10.60
NaOH added:	8 drops (20M)
Final pH:	10.98

ORP (mv):	-78.00
Eh potential (mV):	133.00

ORP (mV):	---
Eh potential (mV):	---

pH 5h later:	10.60
NaOH added:	5 drops (20M)
Final pH:	11.08
ORP (mv):	-76.40
Eh potential (mV):	134.60

pH 5h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.05
ORP (mV):	---
Eh potential (mV):	---

pH 6h later:	10.60
NaOH added:	3 drops (20M)
Final pH:	11.10
ORP (mv):	-74.80
Eh potential (mV):	136.20

pH 6h later:	10.70
NaOH added:	3 drops (20M)
Final pH:	10.98
ORP (mV):	---
Eh potential (mV):	---

pH 7h later:	10.80
NaOH added:	2 drops (20M)
Final pH:	11.07
ORP (mv):	-76.50
Eh potential (mV):	134.50

pH 7h later:	10.70
NaOH added:	5 drops (20M)
Final pH:	11.03
ORP (mV):	---
Eh potential (mV):	---

pH 8h later:	10.81
ORP (mv):	-77.20
Eh potential (mV):	133.80

pH 8h later:	10.70
ORP (mV):	---
Eh potential (mV):	---

As previously mentioned, for the CSQ30 sample this assay had to be repeated so that the results appearing on the table are from the repetition and not from the original assay.

ORP parameter was also measured for both samples. However, during the repetition of the time influence for the CSQ30 sample, the electrode broke not allowing keeping measuring this parameter.

Once again, for the oxidized sample, the pH varied more considerably, taking anew superior volumes of sodium hydroxide to keep it more a less constant.

The analysis of the chemical elements of the leaching processed is presented in the graphics that follow, figure 5.3 for the CSQ10 sample and figure 5.4 for the CSQ30. As already assumed, the corresponding tables can be found in the Appendix B.

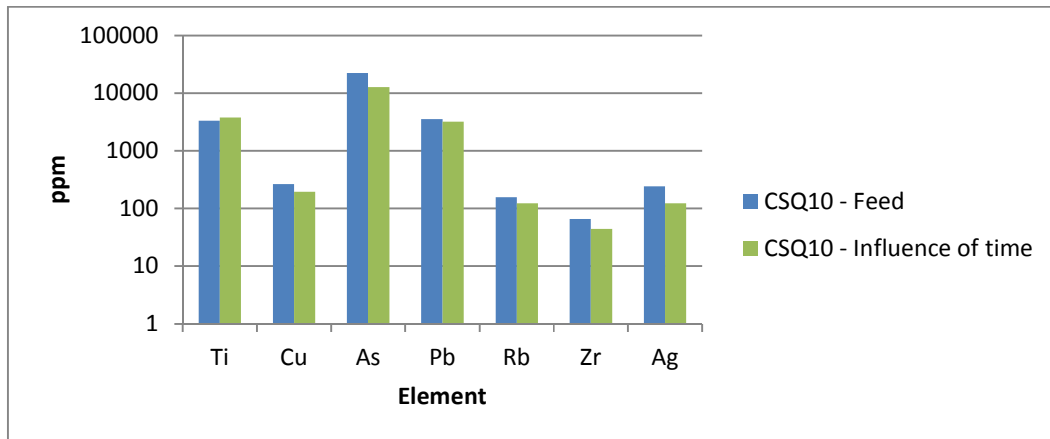


Figure 5.3 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the influence of time

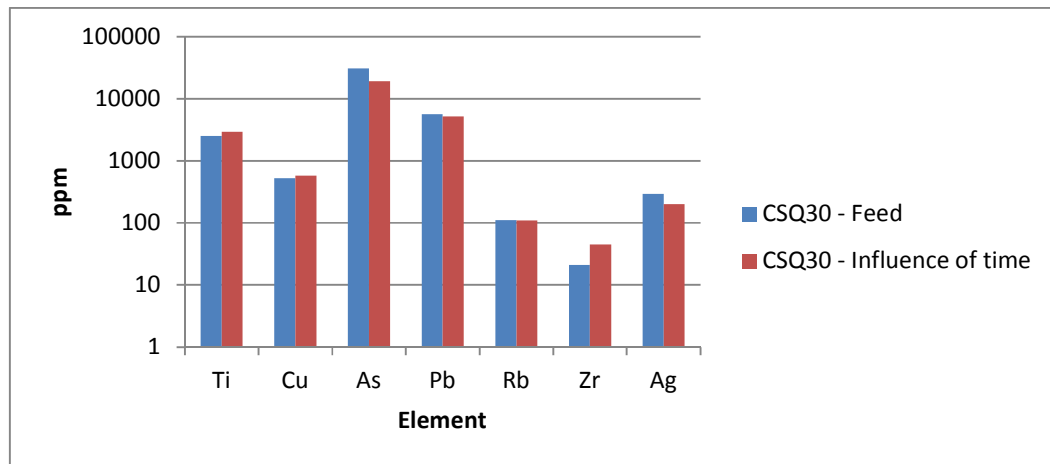


Figure 5.4 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the influence of time

For both cases it is observable that some leaching occurs, given the fact that the concentration of the elements have lowered: this leaching was not selective for gold for this reason. Despite this fact, both titanium and copper concentrations apparently rose for previous stated motives (within the error of measurement).

Having in mind that the feed was 5.5mg/kg of Au for the first sample and 8.98mg/kg of Au for the second, and having the concentration residue results from the laboratory, it is possible to apply the same equation (2) for the yield. All these values are summarized on the tables 23 and 24 for both ore samples.

Table 23 - Leaching yield for the CSQ10 sample taking into consideration the influence of time

Influence of time - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	4.6
Yield (%)	16.36

Table 24 - Leaching yield for the CSQ30 sample taking into consideration the influence of time

Influence of time (repetition) - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	4.9
Yield (%)	45.43

The yield was superior for the oxidized sample, once again supporting the theory.

As previously mentioned, the purpose of this specific procedure was to evaluate if time had influence on the yield of the leaching process. However, that study could only take place through the analysis of the liquid samples. The tables 25 and 26 and figure 5.5 shows the gold concentrations during time (1h, 3h, 5h and 8h) and how they varied. These concentrations are merely figurative once the method applied by LNEG is not well suited for the aim of this work.

Table 25 - Gold concentration in solution for the CSQ10 ore sample among time

CSQ10	
After 1 hour (mg/L Au)	0.66
After 3 hours (mg/L Au)	0.41
After 5 hours (mg/L Au)	1.6
After 8 hours (mg/L Au)	0.22

Table 26 - Gold concentration in solution for the CSQ30 ore sample among time

CSQ30	
After 1 hour (mg/L Au)	2.3
After 3 hours (mg/L Au)	1.9
After 5 hours (mg/L Au)	1.8
After 8 hours (mg/L Au)	0.87

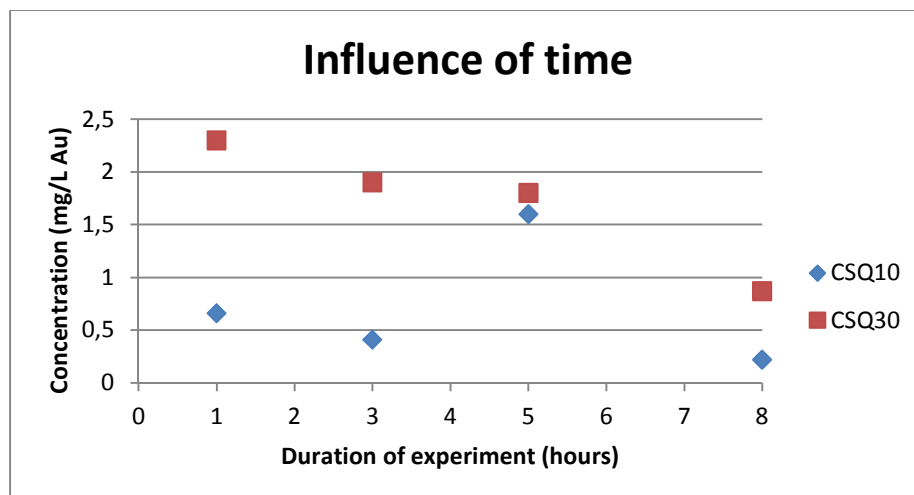


Figure 5.5 - Influence of time on the gold concentration variation

For the CSQ10 sample, the optimal assay duration is 5h, as it was the time when the gold concentration was higher.

However, for the CSQ30, the sample behavior was different: the gold concentration in solution decreased with time and the optimal leaching duration is 1h.

5.3 Varying thiosulphate concentration

The purpose of this set of experiments was to study the influence of the thiosulphate concentration on the leaching process. With this objective, three experiments were carried out: the thiosulphate concentration was varied to 0.1 M, 1 M and 2 M, keeping the ammonia and copper concentrations the same as in the control assay. The results considered for the 0.5 M were taken from the control assay.

- **Thiosulphate concentration: 0.1 M (assay 3)**

Again, both pH and ORP were measured (table 27 and 28). The ORP parameter was then corrected to Eh.

Table 27 - Varying thiosulphate concentration to 0.1 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.62
NaOH added:	20 drops (20M)
Final pH:	11.01
ORP (mV):	-151.10
Eh potential (mV):	59.90

pH 1h later:	10.40
NaOH added:	10 drops (20M)
Final pH:	11.10
ORP (mV):	-50.50
Eh potential (mV):	160.50

pH 2h later:	10.60
NaOH added:	6 drops (20M)
Final pH:	11.00
ORP (mV):	-46.20
Eh potential (mV):	164.80

pH 3h later:	10.60
NaOH added:	6 drops (20M)
Final pH:	11.01
ORP (mV):	-45.10
Eh potential (mV):	165.90

Table 28- Varying thiosulphate concentration to 0.1 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.30
NaOH added:	30 drops (20M)
Final pH:	11.10
ORP (mV):	-112.30
Eh potential (mV):	98.70

pH 1h later:	10.30
NaOH added:	15 drops (20M)
Final pH:	11.00
ORP (mV):	-41.00
Eh potential (mV):	170.00

pH 2h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.00
ORP (mV):	-49.30
Eh potential (mV):	161.70

pH 3h later:	10.60
NaOH added:	8 drops (20M)
Final pH:	11.01
ORP (mV):	-51.90
Eh potential (mV):	159.10

pH 4h later:	10.60
NaOH added:	6 drops (20M)
Final pH:	11.00
ORP (mV):	-46.40
Eh potential (mV):	164.60

pH 4h later:	10.70
NaOH added:	7 drops (20M)
Final pH:	11.00
ORP (mV):	-53.40
Eh potential (mV):	157.60

pH 5h later:	10.60
ORP (mV):	-39.90
Eh potential (mV):	171.10

pH 5h later:	10.70
ORP (mV):	-46.40
Eh potential (mV):	164.60

For these tests, the pH did not vary much for the CSQ10 sample, but it also needed to be fixed every hour by a sodium hydroxide solution.

Contrarily to what happened until this assay, the CSQ10 Eh was not lower than for the CSQ30 from 2 hours on, but both remained more a less constant during the experiment.

Regarding the chemical concentrations the behaviors are shown in figures 5.6 (CSQ10) and 5.7 (CSQ30).

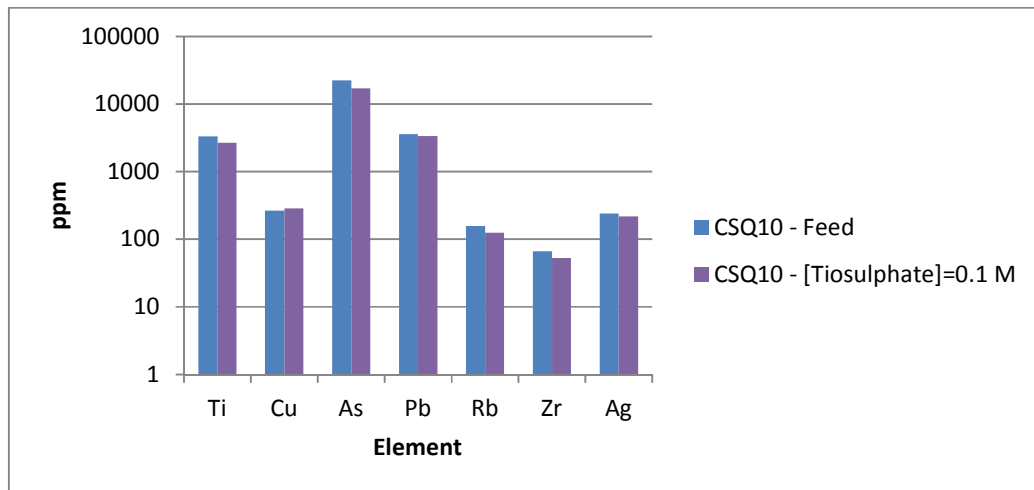


Figure 5.6 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the thiosulphate concentration to 0.1 M

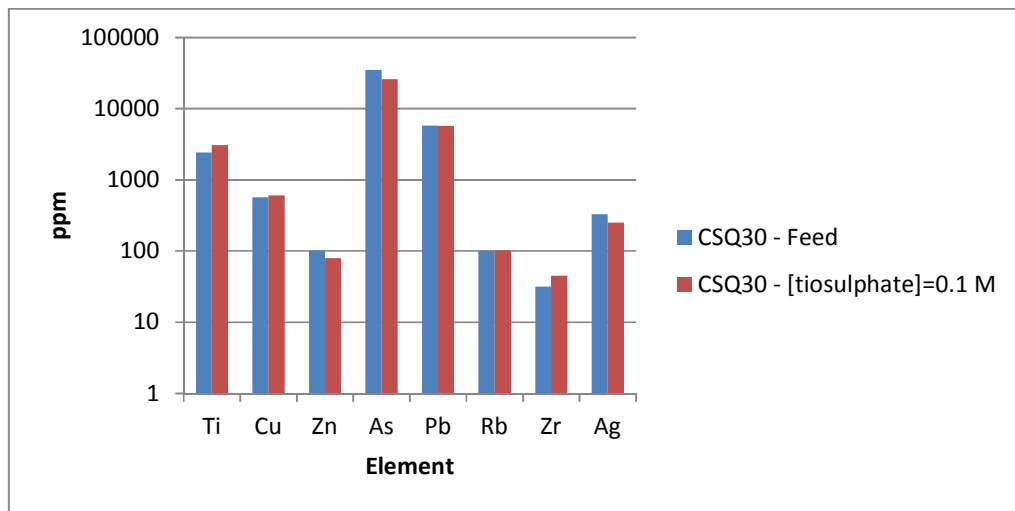


Figure 5.7 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the thiosulphate concentration to 0.1 M

Again, the variation of the element's concentration indicates that the leaching process took place and it was not selective for gold. Titanium concentration increased a little for the CSQ30 sample for previously stated reasons.

Concerning the yield of the process, tables 29 and 30 summarizes them, regarding the solids.

Table 29 - Leaching yield for the CSQ10 sample taking into consideration the influence of thiosulphate concentration (0.1 M)

[Thiosulphate] = 0.1 M - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	4.2
Yield (%)	23.64

Table 30 - Leaching yield for the CSQ30 sample taking into consideration the influence of thiosulphate concentration (0.1 M)

[Thiosulphate] = 0.1 M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	6.00
Yield (%)	33.18

Once again, the yield for the CSQ30 sample was greater than for the CSQ10.

• **Thiosulphate concentration: 1 M (assay 4)**

Both pH and ORP were measured and then the last parameter was corrected to Eh. The results are displayed on tables 31 and 32.

Table 31 - Varying thiosulphate concentration to 1 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.60
NaOH added:	20 drops (20M)
Final pH:	11.00
ORP (mV):	-215.30
Eh potential (mV):	-4.30

pH 1h later:	10.40
NaOH added:	20 drops (20M)
Final pH:	11.10
ORP (mV):	-88.50
Eh potential (mV):	122.50

pH 2h later:	10.40
NaOH added:	10 drops (20M)
Final pH:	11.00
ORP (mV):	-89.30
Eh potential (mV):	121.70

pH 3h later:	10.50
NaOH added:	7 drops (20M)
Final pH:	11.00
ORP (mV):	-94.40
Eh potential (mV):	116.60

pH 4h later:	10.60
NaOH added:	8 drops (20M)
Final pH:	11.10
ORP (mV):	-88.70
Eh potential (mV):	122.30

pH 5h later:	10.70
ORP (mV):	-89.50
Eh potential (mV):	121.50

Table 32 - Varying thiosulphate concentration to 1 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.40
NaOH added:	40 drops (20M)
Final pH:	10.98
ORP (mV):	-119.40
Eh potential (mV):	91.60

pH 1h later:	10.30
NaOH added:	25 drops (20M)
Final pH:	11.00
ORP (mV):	-51.50
Eh potential (mV):	159.50

pH 2h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.00
ORP (mV):	-64.40
Eh potential (mV):	146.60

pH 3h later:	10.30
NaOH added:	15 drops (20M)
Final pH:	11.00
ORP (mV):	-86.00
Eh potential (mV):	125.00

pH 4h later:	10.50
NaOH added:	10 drops (20M)
Final pH:	11.00
ORP (mV):	-80.60
Eh potential (mV):	130.40

pH 5h later:	10.60
ORP (mV):	-78.20
Eh potential (mV):	132.80

The oxidized ore sample took higher volumes of sodium hydroxide in order for its pH to remain more a less constant than the sulphide one, but both pH varied little for this assay.

The redox potential was higher for the CSQ30 sample than it was for the CSQ10; despite this last one did no vary as much.

Regarding chemical composition, figures 5.8 and 5.9 display the results for the CSQ10 and CSQ30 ore samples, respectively.

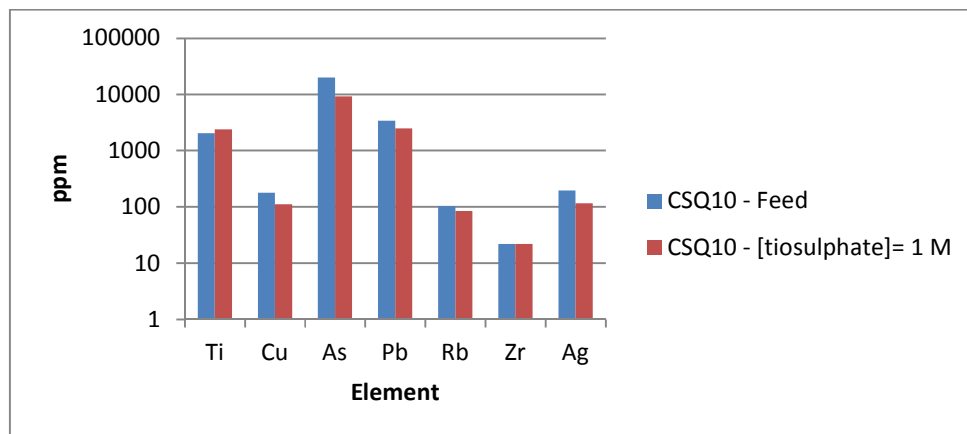


Figure 5.8 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the thiosulphate concentration to 1 M

This image indicates that some elements suffered leaching, since their concentration has decreased. However, the titanium concentration rose instead of decreasing.

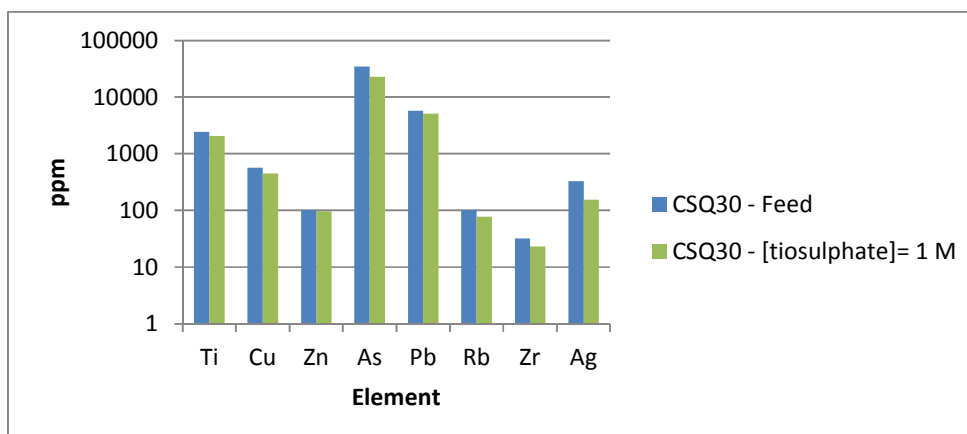


Figure 5.9 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the thiosulphate concentration to 1 M

For the CSQ30 sample it happened the same, since the concentrations diminished for each element.

The conclusion, for both cases, is that the leaching process was not selective for gold.

On the topic of the leaching yield the results for both samples are displayed on tables 33 and 34.

Table 33 - Leaching yield for the CSQ10 sample taking into consideration the influence of thiosulphate concentration (1 M)

[Thiosulphate] = 1 M - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	2.2
Yield (%)	60.00

Table 34 - Leaching yield for the CSQ30 sample taking into consideration the influence of thiosulphate concentration (1 M)

[Thiosulphate] = 1 M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	6.7
Yield (%)	25.39

This time, the CSQ10 sample provided much greater results than the CSQ30, on a 60% yield, contrarily to the expected.

• **Thiosulphate concentration: 2 M (assay 5)**

During this experiment, both pH and ORP were measured and then ORP was converted to Eh.

Table 35 - Varying thiosulphate concentration to 2 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	11.04
NaOH added:	---
Final pH:	---
ORP (mV):	-226.40
Eh potential (mV):	-15.40

pH 1h later:	10.20
NaOH added:	35 drops (20M)
Final pH:	11.10
ORP (mV):	-214.60
Eh potential (mV):	-3.60

pH 2h later:	10.20
NaOH added:	15 drops (20M)
Final pH:	11.00
ORP (mV):	-118.20
Eh potential (mV):	92.80

pH 3h later:	10.60
NaOH added:	7 drops (20M)
Final pH:	11.10
ORP (mV):	-135.30
Eh potential (mV):	75.70

pH 4h later:	10.50
NaOH added:	7 drops (20M)
Final pH:	11.00
ORP (mV):	-162.20
Eh potential (mV):	48.80

pH 5h later:	10.50
ORP (mV):	-153.50
Eh potential (mV):	57.50

Table 36 - Varying thiosulphate concentration to 2 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.80
NaOH added:	25 drops (20M)
Final pH:	10.98
ORP (mV):	-169.40
Eh potential (mV):	41.60

pH 1h later:	10.20
NaOH added:	45 drops (20M)
Final pH:	11.00
ORP (mV):	-140.50
Eh potential (mV):	70.50

pH 2h later:	10.50
NaOH added:	18 drops (20M)
Final pH:	11.00
ORP (mV):	-105.80
Eh potential (mV):	105.20

pH 3h later:	10.70
NaOH added:	7 drops (20M)
Final pH:	11.10
ORP (mV):	-111.80
Eh potential (mV):	99.20

pH 4h later:	10.50
NaOH added:	10 drops (20M)
Final pH:	11.00
ORP (mV):	-108.00
Eh potential (mV):	103.00

pH 5h later:	10.50
ORP (mV):	-106.10
Eh potential (mV):	104.90

For the oxidized sample, the pH varied more considerably, taking superior volumes of sodium hydroxide to keep it more a less constant.

The same happened for the redox potential, being these values higher for the CSQ30 ore sample.

The chemical analysis for both samples are displayed on figures 5.10 and 5.11 for CSQ10 and CSQ30 ores, respectively.

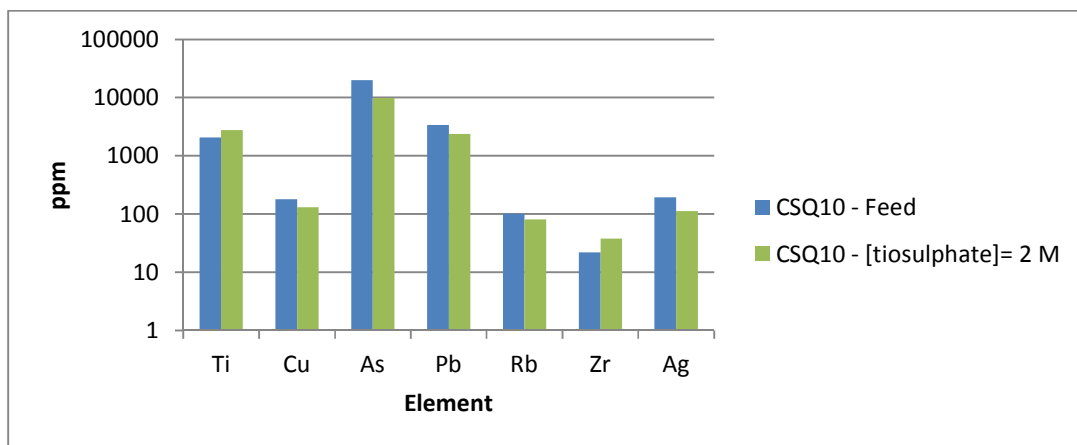


Figure 5.10 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the thiosulphate concentration to 2 M

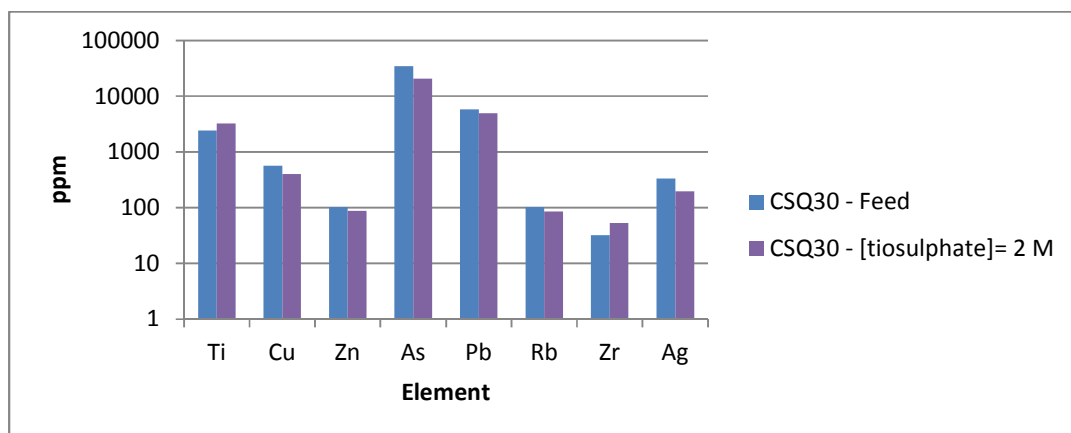


Figure 5.11 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the thiosulphate concentration to 2 M

Taking into consideration the previous images it is fair to say that the leaching experiment happened once it is notorious the element's concentration decrease for almost every element. Therefore it was not selective leaching for gold.

For this experiment, the data considering the CSQ10 sample could not be used as the residue concentration indicated by the laboratory that was higher than the feed content, an impossible situation since the yield cannot be higher than 100%. The only considered results are from the CSQ30 sample, on table 37.

Table 37 - Leaching yield for the CSQ30 sample taking into consideration the influence of thiosulphate concentration (2 M)

[Thiosulphate] = 2 M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	7.0
Yield (%)	22.05

- Performance of the ore samples regarding thiosulphate concentration variation**

Taking into consideration all of the data from assays 2 to 5 it was possible to study the behavior of each sample regarding the leaching process, when varying the thiosulphate concentration.

Despite pH and ORP were only measured from hour to hour and for that reason the behavior for the remaining time is unknown, both graphics display lines in order to simplify its reading.

Concerning the pH, figures 5.12 and figure 5.13 illustrate the behavior of the samples CSQ10 and CSQ30, respectively.

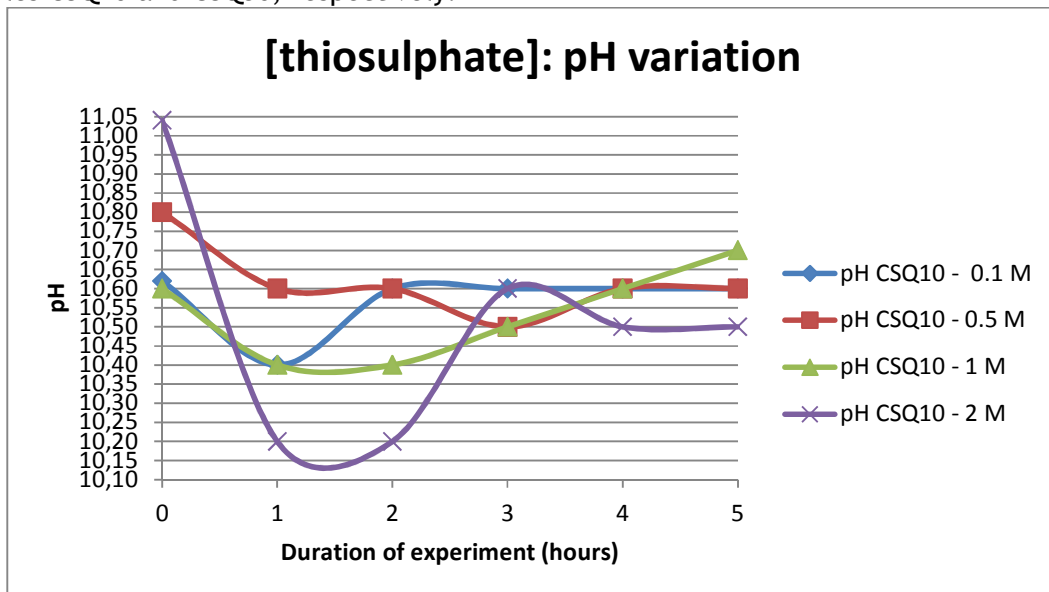


Figure 5.12 - pH behavior regarding the thiosulphate concentration variation, for the CSQ10 sample

In the beginning of every test, the pH was different, but when comparing its value by the end, they did not vary as much among each other.

Bearing in mind that the control assay is represented by the red line, it is observable that the pH did not vary much for this experiment. The higher the thiosulphate concentration, more unstable and variable is the pH.

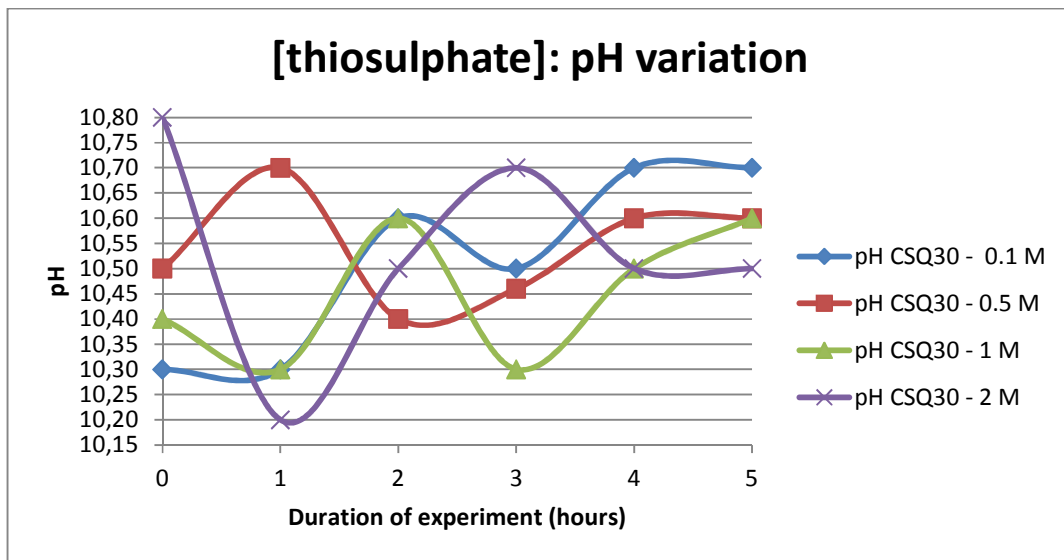


Figure 5.13 - pH behavior regarding the thiosulphate concentration variation, for the CSQ30 sample

When considering the oxidized sample, the pH varied more than on the experiments using the sulphides. However, the relation between pH and thiosulphate concentration is the same than for the previous series of tests.

Regarding Eh, figures 5.14 and 5.15 summarize the processes.

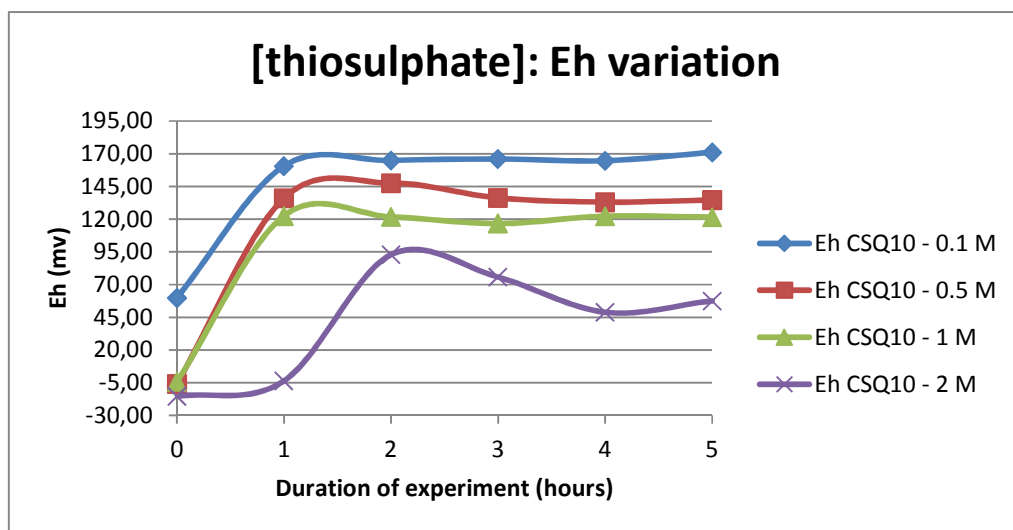


Figure 5.14 - Eh behavior regarding the thiosulphate concentration variation, for the CSQ30 sample

Every experiment had a similar behavior, however the leaching test which had a solution with higher content of thiosulphate varied a little more than the others. The higher the thiosulphate concentration, lower the redox potential.

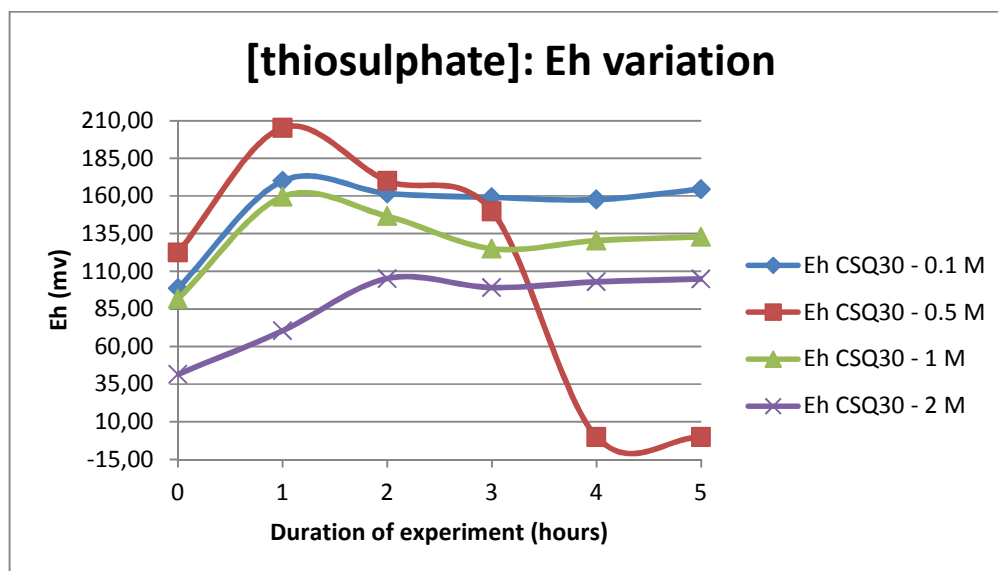


Figure 5.15 - Eh behavior regarding the thiosulphate concentration variation, for the CSQ30 sample

Apart from the control assay (red line), every other experiment exhibited a pattern behavior, the higher the thiosulphate concentration, the smaller the redox potential.

Finally, the figure 5.16 takes into consideration the yield calculated for each concentration of thiosulphate (0.1 M, 0.5 M, 1 M and 2 M).

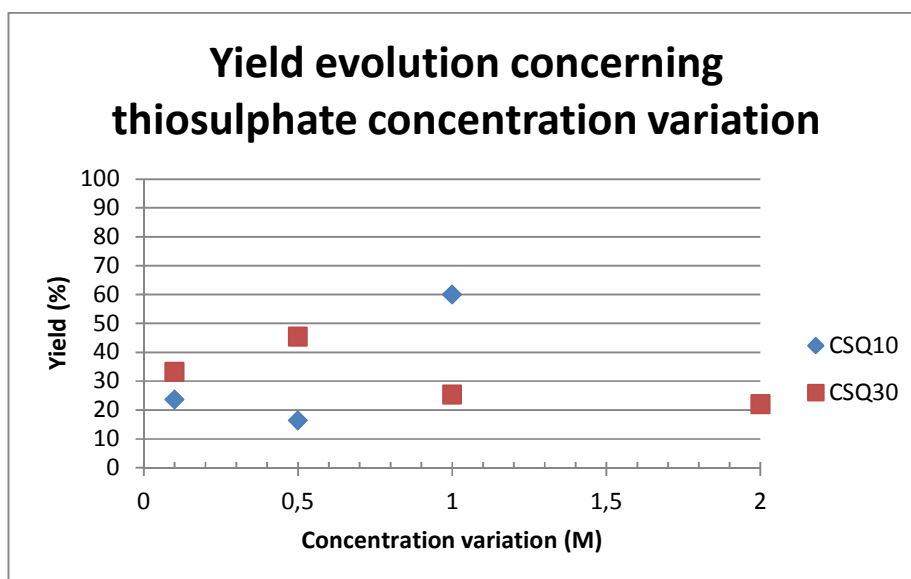


Figure 5.16 - Performance of the sample ores regarding thiosulphate concentration variation

As previously mentioned, for the CSQ10 ore sample, the result of thiosulphate 2 M was ignored. Concerning this ore sample, the optimal concentration was 1 M.

Regarding the oxidized sample, the optimal concentration value was 0.5 M. It cannot be concluded a pattern behavior because each sample has an optimal value within specific characteristics.

5.4 Varying copper concentration

The purpose of this set of experiments was to study the influence of the copper concentration on the leaching process. With this objective, two experiments were carried out: 0.0001 M and 0.01 M. The results considered for the 0.01 M were taken from the control assay.

- **Copper concentration: 0.0001 M (assay 6)**

During this experiment, both pH and ORP were measured and then ORP was converted to Eh, tables 38 and 39.

Table 38 - Varying copper concentration to 0.0001 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.72
NaOH added:	25 drops (20M)
Final pH:	11.01
ORP (mV):	-200.90
Eh potential (mV):	10.10

pH 1h later:	10.50
NaOH added:	20 drops (20M)
Final pH:	11.02
ORP (mV):	-77.30
Eh potential (mV):	133.70

pH 2h later:	10.70
NaOH added:	15 drops (20M)
Final pH:	11.10
ORP (mV):	-69.00
Eh potential (mV):	142.00

pH 3h later:	10.70
NaOH added:	10 drops (20M)
Final pH:	11.05
ORP (mV):	-73.80
Eh potential (mV):	137.20

pH 4h later:	10.70
NaOH added:	10 drops (20M)
Final pH:	11.10
ORP (mV):	-59.40
Eh potential (mV):	151.60

pH 5h later:	10.85
ORP (mV):	-71.50
Eh potential (mV):	139.50

Table 39 - Varying copper concentration to 0.0001 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.71
NaOH added:	45 drops (20M)
Final pH:	11.01
ORP (mV):	-87.10
Eh potential (mV):	123.90

pH 1h later:	10.60
NaOH added:	15 drops (20M)
Final pH:	11.10
ORP (mV):	-42.50
Eh potential (mV):	168.50

pH 2h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.05
ORP (mV):	-35.80
Eh potential (mV):	175.20

pH 3h later:	10.70
NaOH added:	10 drops (20M)
Final pH:	11.10
ORP (mV):	-62.10
Eh potential (mV):	148.90

pH 4h later:	10.70
NaOH added:	8 drops (20M)
Final pH:	11.01
ORP (mV):	-55.00
Eh potential (mV):	156.00

pH 5h later:	10.60
ORP (mV):	-59.40
Eh potential (mV):	151,60

The oxidized ore sample needed higher volumes of NaOH in order for its pH to remain more a less constant, than the sulphide sample. The pH for both samples varied a lot during this experiment.

The Eh was higher for the CSQ30 sample than it was for the CSQ10, varying not as much for this sample than for the CSQ30.

Regarding the chemical element's concentration figures 5.17 and 5.18 show the results for the CSQ10 and CSQ30 ore samples, respectively.

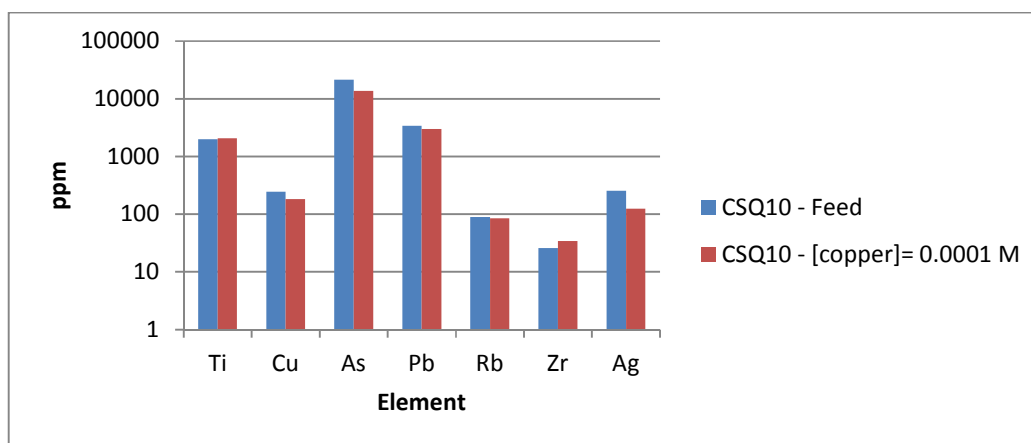


Figure 5.17 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the copper concentration to 0.0001 M

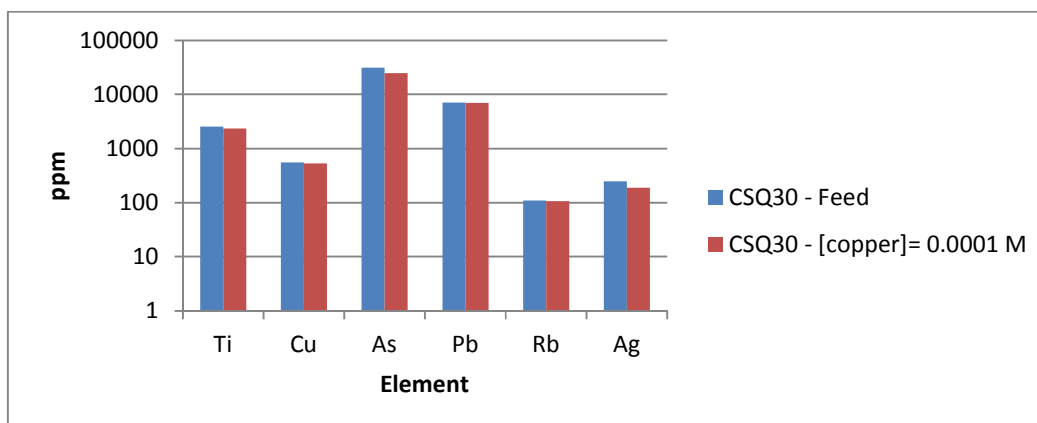


Figure 5.18 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the copper concentration to 0.0001 M

From the previous images, it is possible to sustain the hypothesis that some elements suffered leaching as their concentrations decreased, however it was not selective for gold.

Regarding the process yield, tables 40 and 41 show the obtained results.

Table 40 - Leaching yield for the CSQ10 sample taking into consideration the influence of copper concentration (0.0001 M)

[Copper] = 0.0001 M - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	2.1
Yield (%)	61.82

Table 41 - Leaching yield for the CSQ30 sample taking into consideration the influence of copper concentration (0.0001 M)

[Copper] = 0.0001 M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	4.4
Yield (%)	51.00

The yield for the CSQ10 sample was higher than it was for the CSQ30 this time, showing that the sulphides are more sensitive to copper concentration variation.

• **Copper concentration to 0.01 M (assay 7)**

Both pH and ORP were measured and ORP was then converted to Eh, tables 42 and 43.

Table 42 - Varying copper concentration to 0.01 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.59
NaOH added:	25 drops (20M)
Final pH:	11.01
ORP (mV):	-188.00
Eh potential (mV):	23.00

pH 1h later:	10.70
NaOH added:	15 drops (20M)
Final pH:	11.10
ORP (mV):	-43.20
Eh potential (mV):	167.80

pH 2h later:	10.70
NaOH added:	9 drops (20M)
Final pH:	11.08
ORP (mV):	-46.40
Eh potential (mV):	164.60

pH 3h later:	10.60
NaOH added:	7 drops (20M)
Final pH:	11.02
ORP (mV):	-46.00
Eh potential (mV):	165.00

pH 4h later:	10.70
NaOH added:	7 drops (20M)
Final pH:	11.10
ORP (mV):	-28.20
Eh potential (mV):	182.80

pH 5h later:	10.60
ORP (mV):	-36.00
Eh potential (mV):	175.00

Table 43 - Varying copper concentration to 0.01 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.40
NaOH added:	45 drops (20M)
Final pH:	11.01
ORP (mV):	-138.40
Eh potential (mV):	72.60

pH 1h later:	10.60
NaOH added:	20 drops (20M)
Final pH:	11.05
ORP (mV):	-40.20
Eh potential (mV):	170.80

pH 2h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	11.03
ORP (mV):	-15.00
Eh potential (mV):	196.00

pH 3h later:	10.70
NaOH added:	12 drops (20M)
Final pH:	11.05
ORP (mV):	-24.60
Eh potential (mV):	186.40

pH 4h later:	10,60
NaOH added:	10 drops (20M)
Final pH:	11,05
ORP (mV):	-0,20
Eh potential (mV):	210,80

pH 5h later:	10.70
ORP (mV):	-3.30
Eh potential (mV):	207.70

During these tests, the pH was more regular for both samples, but it needed to be fixed every hour by a sodium hydroxide solution.

The redox potential was higher for the CSQ30 sample than it was for the CSQ10, being more constant for this last one.

Concerning the chemical analysis of both samples, figures 5.19 and 5.20 summarize them.

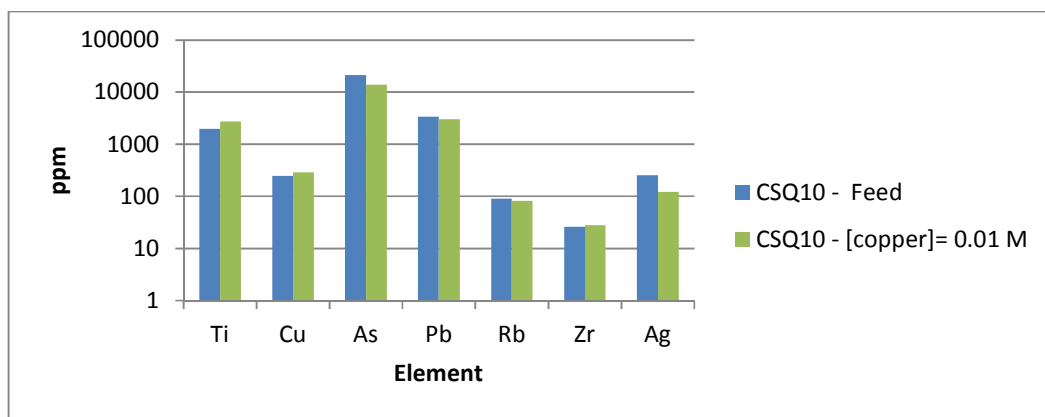


Figure 5.19 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the copper concentration to 0.01 M

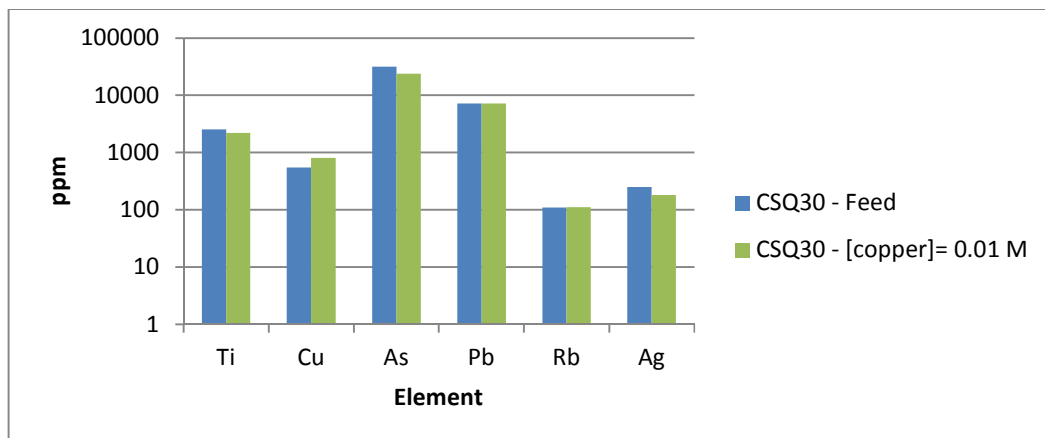


Figure 5.20 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the copper concentration to 0.01 M

Analyzing the previous images, it is observable a variation on the concentration, concordant with what would happened during the leaching process (concentration decrease). However this leaching process was not selective for gold (the main purpose).

Regarding the leaching yield, tables 44 and 45 provide the results.

Table 44 - Leaching yield for the CSQ10 sample taking into consideration the influence of copper concentration (0.01 M)

[Copper] = 0.01 M - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	0.96
Yield (%)	82.55

Table 45 - Leaching yield for the CSQ30 sample taking into consideration the influence of copper concentration (0.01 M)

[Copper] = 0.01 M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	1.4
Yield (%)	84.41

The yield was high for both cases (above 80%), despite it was greater for the CSQ30 sample.

- **Performance of the ore samples regarding copper concentration variation**

The pH variations for both samples are represented on the graphics that follow.

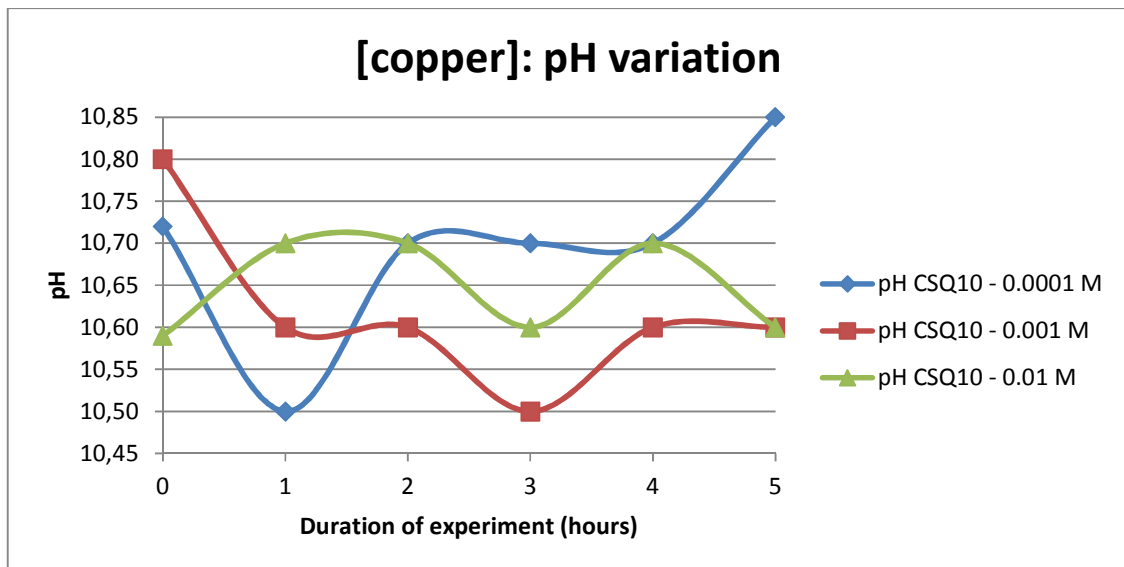


Figure 5.21 - pH behavior regarding the copper concentration variation, for the CSQ10 sample

The control assay is represented by the red line, which varied a little. The higher the copper concentration, lower the pH.

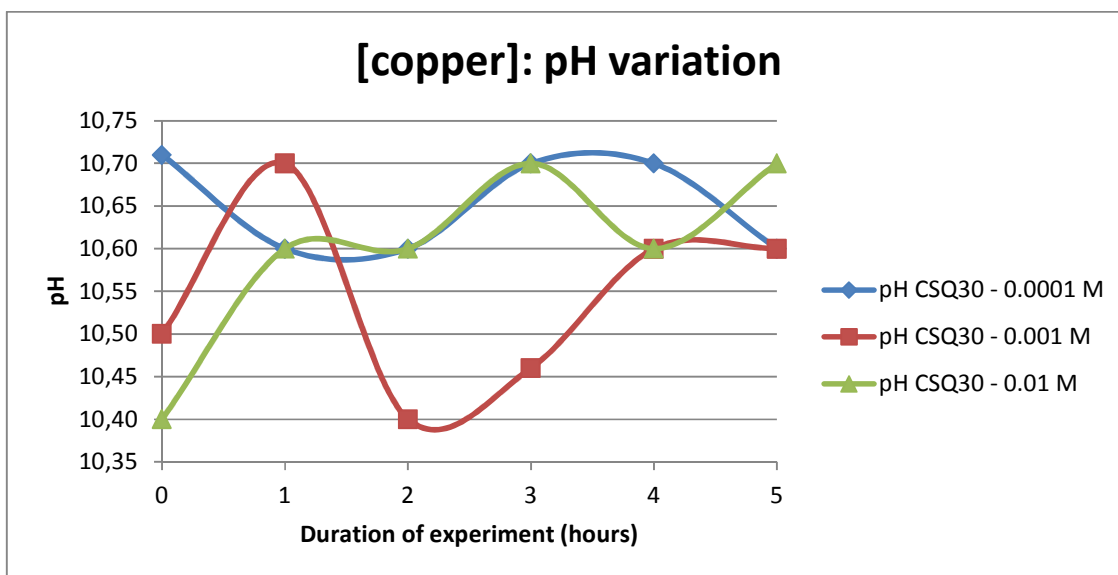


Figure 5.22 - pH behavior regarding the copper concentration variation, for the CSQ30 sample

Even though every experiment began with a very different pH value, the final measurement did not differ much. The control experiment (red line) was the one which varied the most. No conclusion can be made about the behavior of the sample regarding copper concentration variation as apparently there is no link between the pH values and the concentration.

For the redox potential, figures 5.23 and 5.24 illustrate all measures collected.

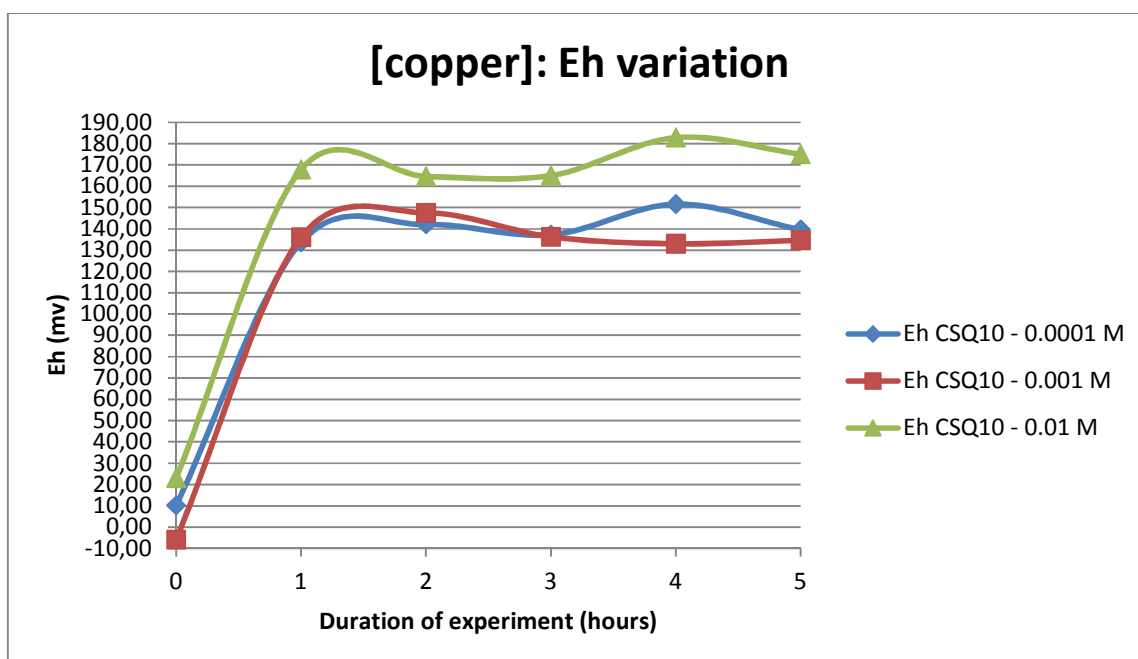


Figure 5.23 - Eh behavior regarding the copper concentration variation, for the CSQ10 sample

Observing the previous graphic it is possible to say that the samples behaved more or less in the same way. The experiment in which was used a higher copper concentration exhibited a higher redox potential.

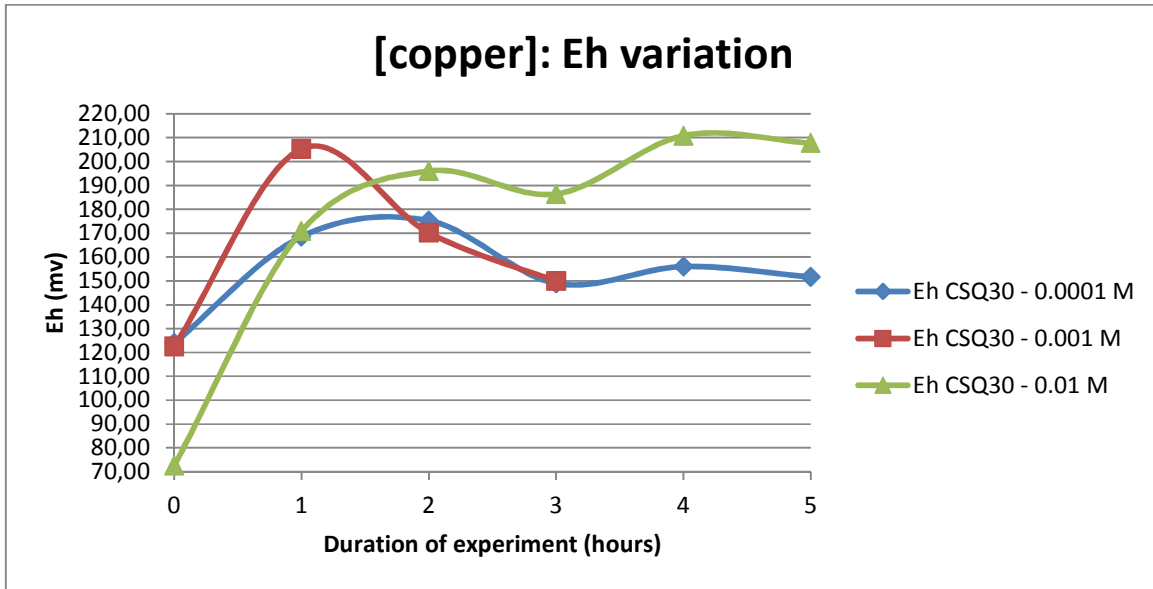


Figure 5.24 - Eh behavior regarding the copper concentration variation, for the CSQ30 sample

For this sample, the Eh behaved more widely and did not exhibit a specific behavior pattern.

The figure 5.25 takes into consideration the yield calculated for each concentration of copper (0.0001 M, 0.001 M and 0.01 M).

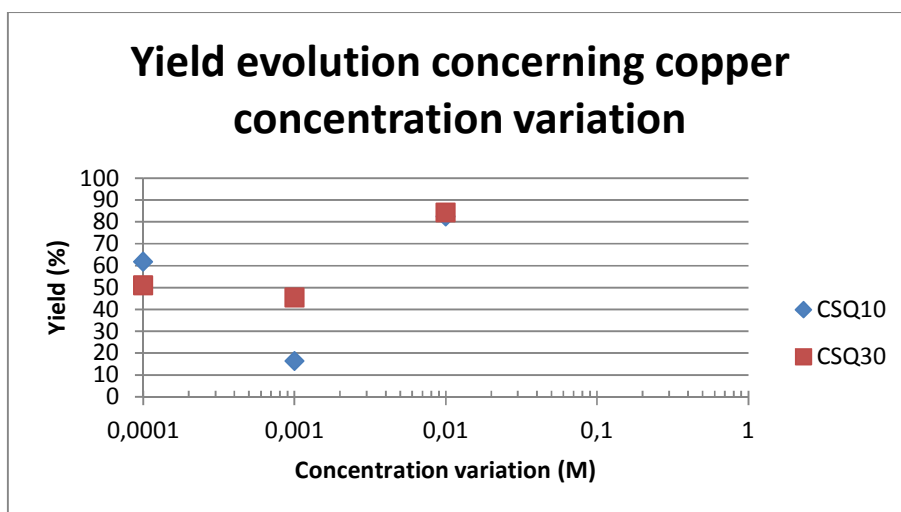


Figure 5.25 - Performance of the ore samples regarding copper concentration variation

For both samples, the optimal copper concentration was 0.01M.

5.5 Varying ammonia concentration

The purpose of this set of experiments was to study the influence of the ammonia concentration on the leaching process. With this objective, two experiments were carried out: 2 M and 3 M. The results considered for the 1 M were taken from the control assay.

- **Ammonia concentration: 2 M (assay 8)**

During this test, the ORP electrode was broken so that the only reads are from pH.

Table 46 - Varying ammonia concentration to 2 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.91
NaOH added:	---
Final pH:	---

pH 1h later:	10.50
NaOH added:	30 drops (20M)
Final pH:	10.93

pH 2h later:	10.60
NaOH added:	15 drops (20M)
Final pH:	10.98

pH 3h later:	10.43
NaOH added:	15 drops (20M)
Final pH:	10.95

pH 4h later:	10.50
NaOH added:	10 drops (20M)
Final pH:	11.20

pH 5h later:	10.58
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Table 47 - Varying ammonia concentration to 2 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.89
NaOH added:	---
Final pH:	---

pH 1h later:	10.44
NaOH added:	30 drops (20M)
Final pH:	10.89

pH 2h later:	10.50
NaOH added:	15 drops (20M)
Final pH:	11.01

pH 3h later:	10.51
NaOH added:	15 drops (20M)
Final pH:	10.95

pH 4h later:	10.48
NaOH added:	10 drops (20M)
Final pH:	11.01

pH 5h later:	10.61
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For these experiments, the pH was more regular for the CSQ10 ore sample, but it also needed to be fixed every hour by a sodium hydroxide solution, considering both samples.

Regarding the chemical elements concentration, the graphics that follow show the main results.

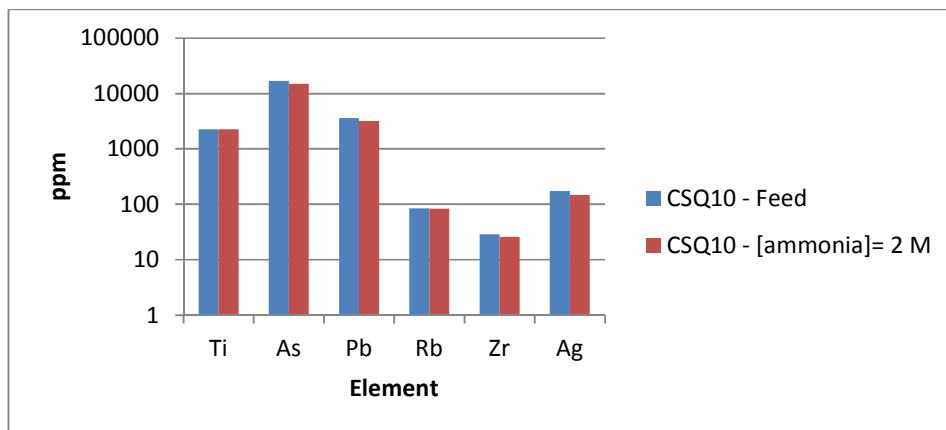


Figure 5.26 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the ammonia concentration to 2 M

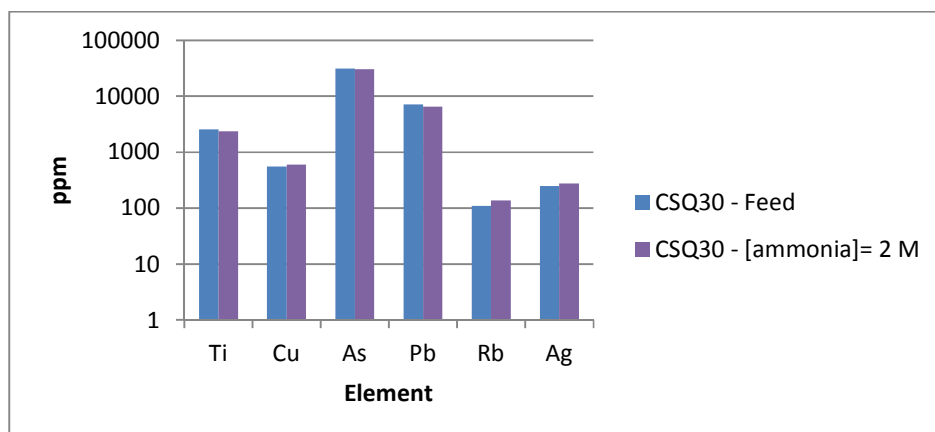


Figure 5.27 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the ammonia concentration to 2 M

Considering figures 5.26 and 2.57 it is possible to say that almost all of the elements suffered leaching, as their concentrations lowered. However, for the CSQ30 ore sample, copper, rubidium and silver concentration rose. These values are within the error of measurement.

The main conclusion is that the leaching process was not selective for gold.

Regarding the process yield, tables 48 and 49 show the results.

Table 48 - Leaching yield for the CSQ10 sample taking into consideration the influence of ammonia concentration (2 M)

[Ammonia] = 2 M - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	4.7
Yield (%)	14.55

Table 49 - Leaching yield for the CSQ30 sample taking into consideration the influence of ammonia concentration (2 M)

[Ammonia] = 2 M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	0.03
Yield (%)	99.67

The results for each sample are very different: while CSQ10 had a very poor yield, CSQ30 had an almost 100% leaching yield.

- **Ammonia concentration: 3 M (assay 9)**

During this experiment, only pH was measured for the same reason as in the previous assay.

Table 50 - Varying ammonia concentration to 3 M, measures for the CSQ10 sample

CSQ10	
Initial pH:	11.06
NaOH added:	---
Final pH:	---

pH 1h later:	10.66
NaOH added:	25 drops (20M)
Final pH:	10.94

pH 2h later:	10.67
NaOH added:	15 drops (20M)
Final pH:	10.94

pH 3h later:	10.58
NaOH added:	15 drops (20M)
Final pH:	10.91

pH 4h later:	10.52
NaOH added:	15 drops (20M)
Final pH:	11.01

pH 5h later:	10.51
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Table 51 - Varying ammonia concentration to 3 M, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.93
NaOH added:	---
Final pH:	---

pH 1h later:	10.61
NaOH added:	25 drops (20M)
Final pH:	10.93

pH 2h later:	10.64
NaOH added:	20 drops (20M)
Final pH:	11.03

pH 3h later:	10.64
NaOH added:	15 drops (20M)
Final pH:	10.98

pH 4h later:	10.61
NaOH added:	15 drops (20M)
Final pH:	11.02

pH 5h later:	10.51
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For both ore samples the first measure indicated that the pH needed no correction because the ammonia stabilizes pH.

Afterwards it remained more or less constant, being used practically identical hydroxide volumes for both ores to keep pH more or less constant.

The main chemical elements analysis for both samples are illustrated on figures 5.28 and 5.29.

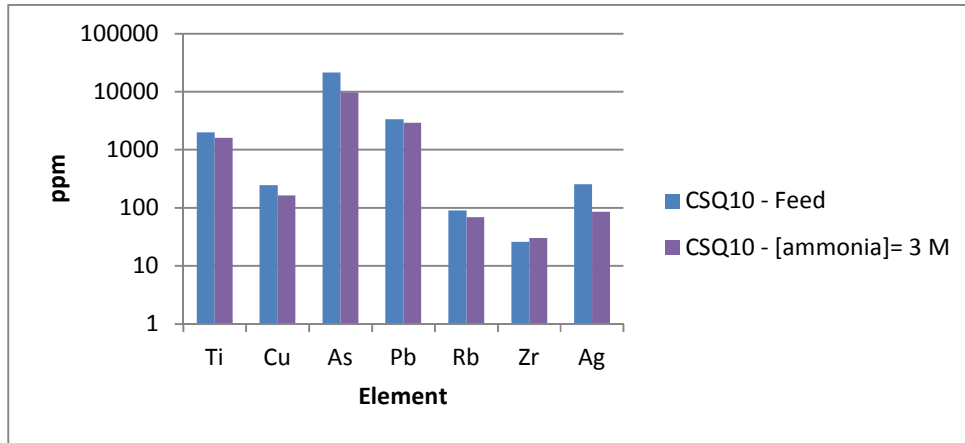


Figure 5.28 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the variation of the ammonia concentration to 3 M

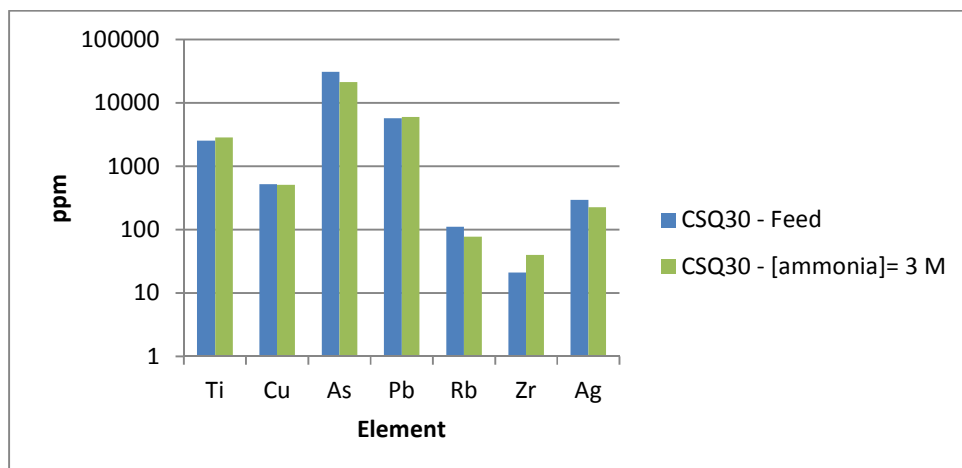


Figure 5.29 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the variation of the ammonia concentration to 3 M

Regarding the leaching process itself, it is possible to say that some chemical elements suffered leaching as their concentrations have decreased. However, some elements such as titanium and zirconium were found in higher concentrations than on the feed ore probably due to the uncertainty of the measure performed by the equipment. Therefore, the leaching process was not selective for gold.

Regarding the leaching yield, tables 52 and 53 provide the results.

Table 52 - Leaching yield for the CSQ10 sample taking into consideration the influence of ammonia concentration (3 M)

[Ammonia] = 3M - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	3.6
Yield (%)	34.55

Table 53 - Leaching yield for the CSQ30 sample taking into consideration the influence of ammonia concentration (3 M)

[Ammonia] = 3M - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	4.0
Yield (%)	55.46

Once again the samples did not behave the same way. However, the yield for the CSQ30 sample was higher than for the CSQ10.

- **Performance of the ore samples regarding ammonia concentration variation**

Regarding pH, figures 5.30 and 5.31 illustrate all the leaching tests carried out concerning the ammonia concentration variation.

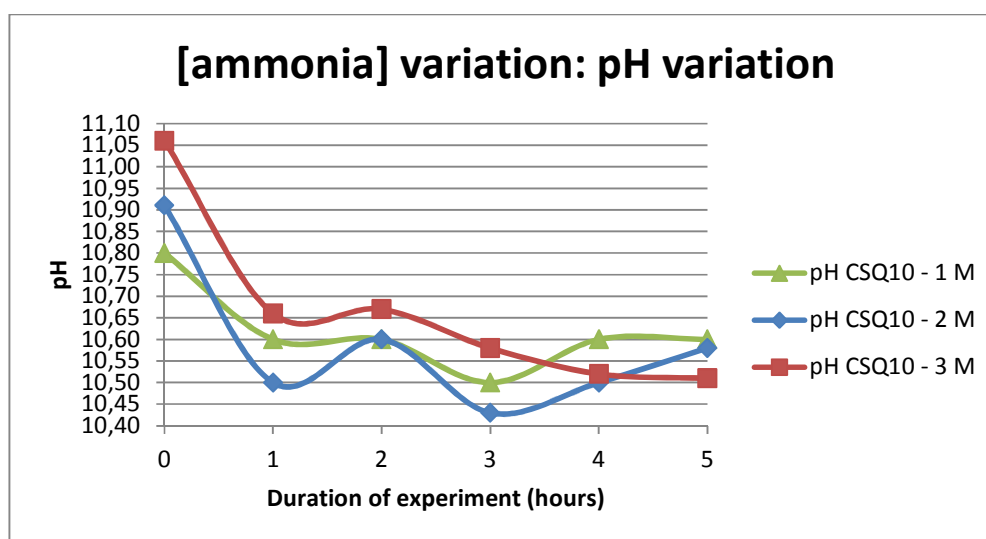


Figure 5.30 - pH behavior regarding the ammonia concentration variation, for the CSQ10 sample

For the experiment with higher ammonia concentration, pH values were also higher, at least for the major experiment duration. Despite the higher values, the increase of the ammonia concentration also turned the solution more unstable: the leaching experiment which was more stable was the one that used 1 M of ammonia (the control experiment).

The relation between ammonia concentration and pH that can be enlightened is: the higher the concentration, higher the initial pH values.

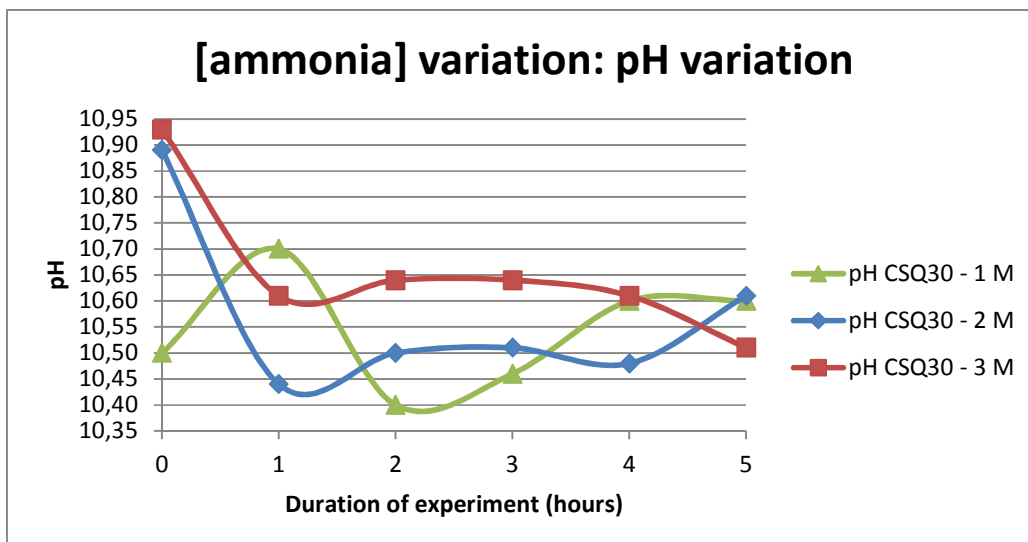


Figure 5.31 - pH behavior regarding the ammonia concentration variation, for the CSQ30 sample

The oxidized samples were more unstable than the sulphide ones. The same conclusions can be made for this set of leaching experiments: the higher the ammonia concentration, higher the initial pH values. The control sample (1 M of ammonia) was the sample that kept more constant values.

No conclusions can be made about the redox potential, as there are no values for both ore samples.

Each leaching yield was then compiled on the figure 5.32.

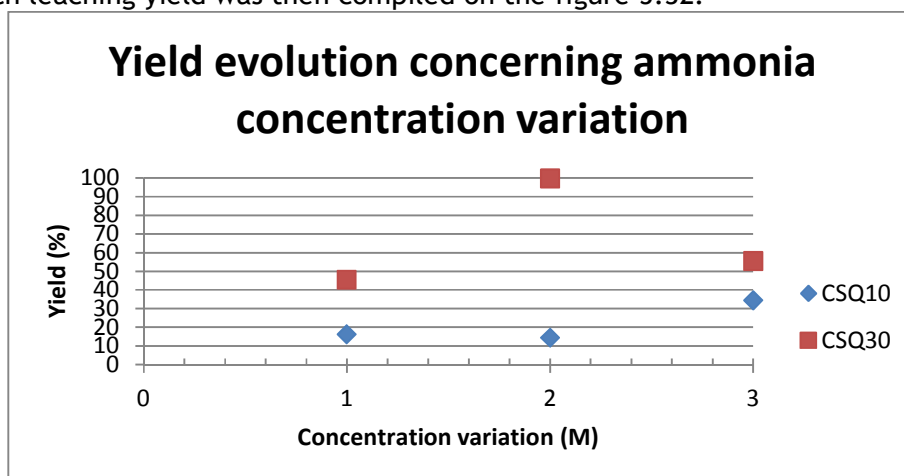


Figure 5.32 - Performance of the ore samples regarding ammonia concentration variation

The samples did not behave the same way. The optimal ammonia concentration for the CSQ10 sample was 3 M while for the CSQ30 it was 2 M, with an approximately 100% yield on the leaching process.

5.6 Varying temperature

The purpose of this set of experiments was to study the influence of the temperature on the leaching process. With this objective, two experiments were carried out: one with a range of temperatures from 60 to 70 °C and other between 40 and 45 °C. The results considered for the room temperature were taken from the control assay.

- **Temperature: between 60 and 70 °C (assay 10)**

Regarding this experimentation and having a new ORP electrode, three parameters were measured: pH, ORP and temperature (which had to be kept between 60 and 70 °C).

The readings are on tables 54 and 55.

Table 54 - Varying temperature between 60 and 70 °C, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.60
NaOH added:	15 drops (20M)
Final pH:	10.93
ORP (mV):	-220.30
Eh potential (mV):	-26.30
Temperature:	25.00

pH 1h later:	9.60
NaOH added:	70 drops (20M)
Final pH:	10.65
ORP (mV):	-181.50
Eh potential (mV):	12.50
Temperature:	65.00

pH 2h later:	10.60
NaOH added:	30 drops (20M)
Final pH:	10.90
ORP (mV):	-236.00
Eh potential (mV):	-42.00
Temperature:	67.00

pH 3h later:	10.63
NaOH added:	30 drops (20M)
Final pH:	11.01
ORP (mv):	-271.30
Eh potential (mV):	-77.30
Temperature:	63.00

pH 4h later:	11.20
NaOH added:	---
Final pH:	---
ORP (mv):	-273.50
Eh potential (mV):	-79.50
Temperature:	65.00

pH 5h later:	11.01
ORP (mv):	-265.50
Eh potential (mV):	-71.50
Temperature:	63.00

Table 55 - Varying temperature between 60 and 70 °C, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.30
NaOH added:	20 drops (20M)
Final pH:	10.89
ORP (mV):	-61.70
Eh potential (mV):	132.30
Temperature:	25.00

pH 1h later:	9.50
NaOH added:	75 drops (20M)
Final pH:	10.65
ORP (mv):	-135.60
Eh potential (mV):	58.40
Temperature:	61.00

pH 2h later:	10.50
NaOH added:	30 drops (20M)
Final pH:	11.09
ORP (mV):	-115.50
Eh potential (mV):	78.50
Temperature:	62.00

pH 3h later:	10.60
NaOH added:	30 drops (20M)
Final pH:	11.04
ORP (mv):	-102.50
Eh potential (mV):	91.50
Temperature:	62.00

pH 4h later:	10.90
NaOH added:	---
Final pH:	---
ORP (mv):	-130.30
Eh potential (mV):	63.70
Temperature:	62.00

pH 5h later:	10.60
ORP (mv):	-130.00
Eh potential (mV):	64.00
Temperature:	63.00

The pH during these assays floated considerably for both samples. This is due to the fact that pH is affected by temperature and its range was large.

However, this variance was greater for the CSQ30 sample.

The redox potential varied a lot during these experiments, being these measures considerably lower for the CSQ10 sample than for the CSQ30.

Considering the chemical element concentration variation, figures 5.33 and 5.34 illustrate the main tendency for the CSQ10 and CSQ30 ore samples, respectively.

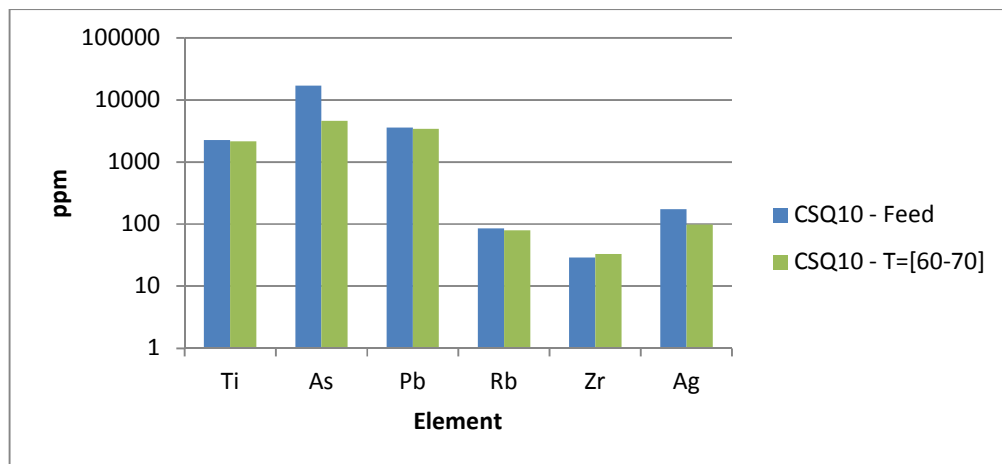


Figure 5.33 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the temperature variation between 60 and 70 °C

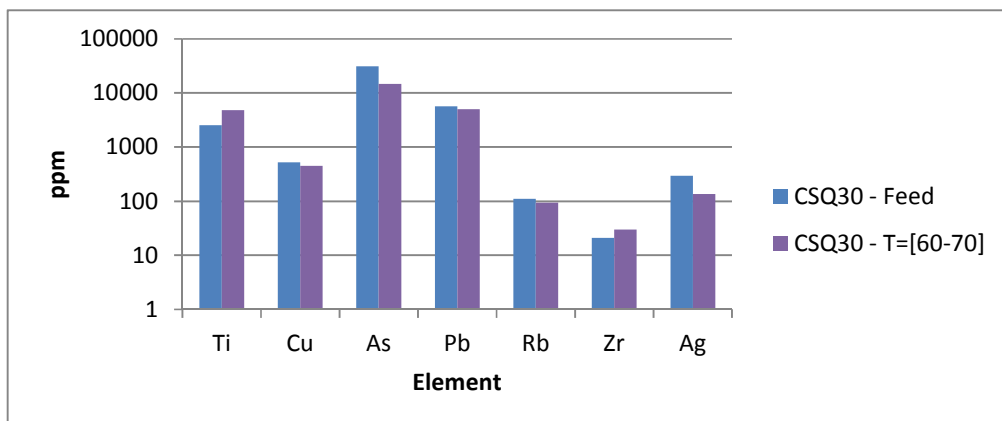


Figure 5.34 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the temperature variation between 60 and 70 °C

Considering the previous graphics, a plausible conclusion it that the leaching process occurred since the chemical element's concentrations, generally speaking, have decreased, except for zirconium (CSQ10) and titanium (CSQ30).

Therefore, it is possible to conclude that the leaching was not selective for gold.

Regarding the leaching yield, tables 56 and 57 provide the results.

Table 56 - Leaching yield for the CSQ10 sample taking into consideration the influence of temperature variation (60-70 °C)

Temperature: [60-70 °C] - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	3.9
Yield (%)	29.09

Table 57 - Leaching yield for the CSQ30 sample taking into consideration the influence of temperature variation (60-70 °C)

Temperature: [60-70°C] - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	4.5
Yield (%)	49.89

For the CSQ30 sample the yield was higher than for the CSQ10, as the samples behaved differently.

- **Temperature: between 40 and 45 °C (assay 11)**

Again, three parameters were measured: pH, ORP (which was converted to Eh later) and temperature (which had to be kept between 40 and 45 °C).

Table 58 - Varying temperature between 40 and 45 °C, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.75
NaOH added:	---
Final pH:	---
ORP (mV):	-228.30
Eh potential (mV):	-48.30
Temperature:	25.00

pH 1h later:	10.10
NaOH added:	50 drops (20M)
Final pH:	11.01
ORP (mV):	-186.90
Eh potential (mV):	-6.90
Temperature:	42.00

pH 2h later:	10.40
NaOH added:	15 drops (20M)
Final pH:	11.01
ORP (mV):	-190.53
Eh potential (mV):	-10.53
Temperature:	43.00

pH 3h later:	10.60
NaOH added:	10 drops (20M)
Final pH:	10.99
ORP (mV):	-250.30
Eh potential (mV):	-70.30
Temperature:	42.00

pH 4h later:	10.90
NaOH added:	---
Final pH:	---
ORP (mV):	-245.30
Eh potential (mV):	-65.30

Table 59 - Varying temperature between 40 and 45 °C, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.71
NaOH added:	---
Final pH:	---
ORP (mV):	-81.00
Eh potential (mV):	99.00
Temperature:	25.00

pH 1h later:	9.90
NaOH added:	55 drops (20M)
Final pH:	10.90
ORP (mV):	-59.40
Eh potential (mV):	120.60
Temperature:	42.00

pH 2h later:	10.30
NaOH added:	20 drops (20M)
Final pH:	10.98
ORP (mV):	-70.50
Eh potential (mV):	109.50
Temperature:	42.00

pH 3h later:	10.30
NaOH added:	15 drops (20M)
Final pH:	10.98
ORP (mV):	-90.50
Eh potential (mV):	89.50
Temperature:	42.00

pH 4h later:	10.70
NaOH added:	10 drops (20M)
Final pH:	10.99
ORP (mV):	-80.50
Eh potential (mV):	99.50

Temperature:	42.00
--------------	-------

Temperature:	44.00
--------------	-------

pH 5h later:	10.90
ORP (mV):	-177.90
Eh potential (mV):	2.10
Temperature:	42.00

pH 5h later:	10.77
ORP (mV):	-90.80
Eh potential (mV):	89.20
Temperature:	42.00

Again, the pH varied a lot, for the same reason stated for the earlier assay. The amount of sodium hydroxide need to keep pH constant was a little higher for the second sample, when analyzing the corresponding table.

About the redox potential it is fair to say that it was lower for the sulphide ore sample than it was for the oxidized sample, represented on the next figure.

Regarding the chemical element concentration variation, figures 5.35 and 5.36 illustrate the process.

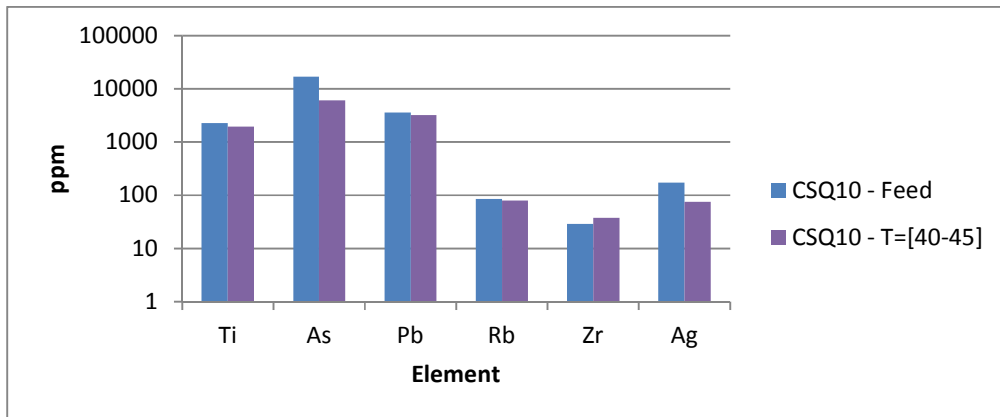


Figure 5.35 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the temperature variation between 40 and 45 °C

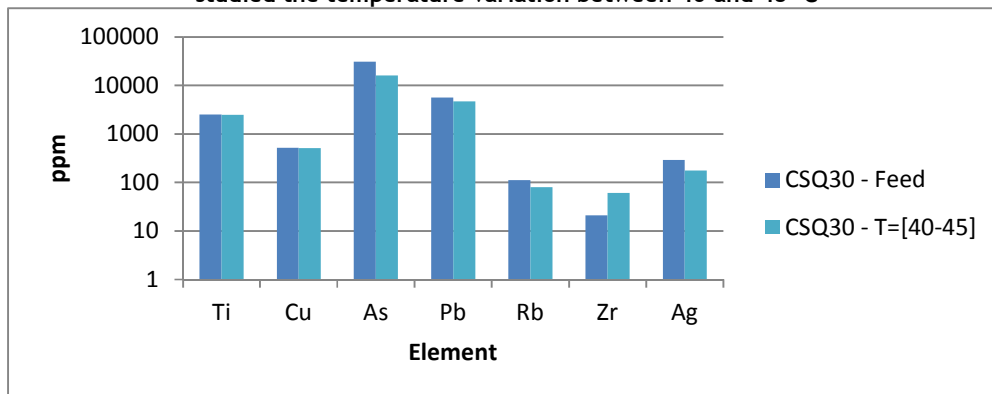


Figure 5.36 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the temperature variation between 40 and 45 °C

Analyzing the previous graphics it is possible to say that the leaching process occurred since the chemical element's concentrations, in general, have decreased, except for zirconium which increased in both samples. Therefore, this process was not selective for gold.

Regarding the leaching yield, tables 60 and 61 provide the results.

Table 60 - Leaching yield for the CSQ10 sample taking into consideration the influence of temperature variation (40-45 °C)

Temperature: [40-45] - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	0.72
Yield (%)	86.91

Table 61 - Leaching yield for the CSQ30 sample taking into consideration the influence of temperature variation (40-45 °C)

Temperature: [40-45] - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	0.08
Yield (%)	99.11

For the CSQ30 sample the yield was higher than for the CSQ10, as the samples behaved differently. For this range of temperature, the yield was higher than expected (since they were greater than on assay 10).

- **Performance of the ore samples regarding temperature variation**

Concerning pH, the figures that follow summarize the variation when studying the temperature.

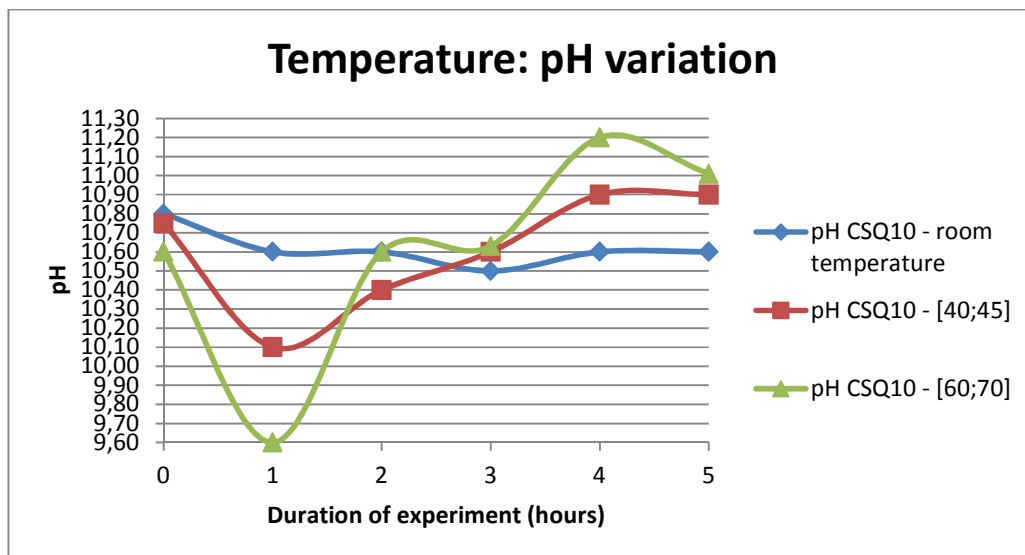


Figure 5.37 - pH behavior regarding the temperature variation, for the CSQ10 sample

Knowing that pH depends on temperature, and that the experiment represented by the green line had a greater temperature range it is normal that the pH variation was greater. The same happens with the assay in which the temperature was kept around 40 °C. The control assay was the one which had the more stable pH range as the temperature did not vary as much.

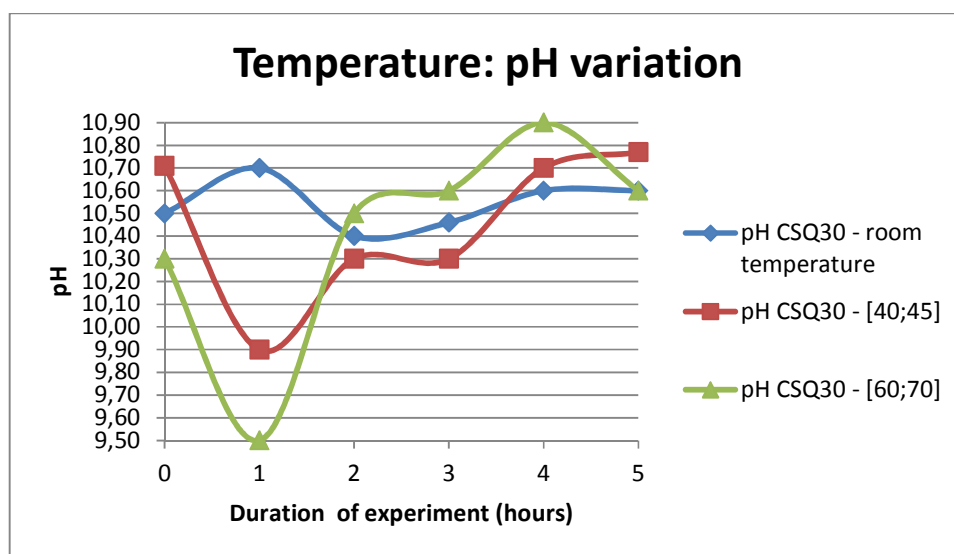


Figure 5.38 - pH behavior regarding the temperature variation, for the CSQ30 sample

The redox potential depends on pH, so this means that wider range of pH implies a wider range of Eh values which can be observable on the previous graphic.

The same conclusions for this set of experiments can be taken from the analysis of the figure 5.38.

The control assay (blue line) was the one which had the more stable pH and temperature range, being its Eh stable too.

Every leaching yield mentioned on the former assays is now summarized on the figure that follows.

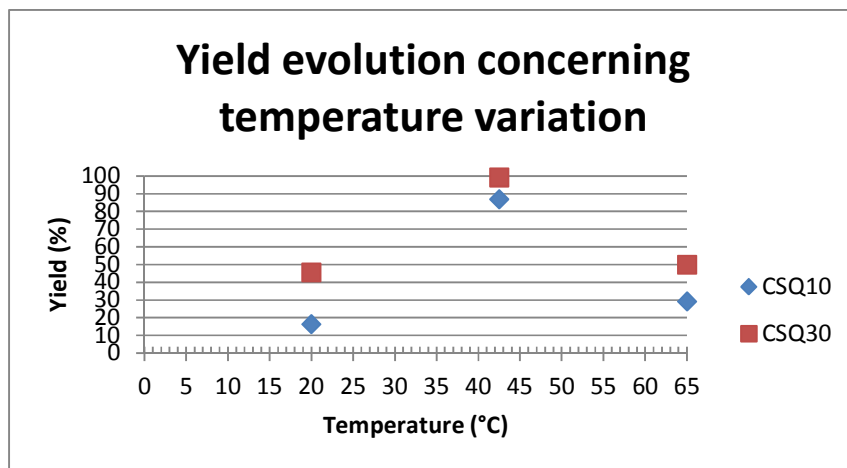


Figure 5.39 - Performance of the ore samples regarding temperature variation

Regarding the leaching yield when varying the temperature, the optimal temperature, for both ore samples was between 40 and 45 °C. On the CSQ30 sample this value was almost 100%.

5.7 Varying rotation speed

The purpose of this experiment was to study the influence of the rotation speed on the leaching process, taking into consideration that for the previous set of experiments, the rotation speed had to be kept at a minimum. With this objective, one experiment was carried out maintaining this velocity at the lowest possible. The results considered for the level 1 were taken from the control assay.

- **Rotation speed (assay 12): zero level**

According to the purpose of this experiment, only pH and ORP were measured (this latter parameter was then converted to Eh).

Table 62 - Varying rotation speed, measures for the CSQ10 sample

CSQ10	
Initial pH:	10.70
NaOH added:	10 drops (20M)
Final pH:	10.90
ORP (mV):	-190.00
Eh potential (mV):	21.00

pH 1h later:	10.20
NaOH added:	20 drops (20M)
Final pH:	11.01
ORP (mV):	-92.00
Eh potential (mV):	119.00

pH 2h later:	10.53
NaOH added:	15 drops (20M)
Final pH:	11.01
ORP (mV):	-50.20
Eh potential (mV):	160.80

pH 3h later:	10.60
NaOH added:	15 drops (20M)
Final pH:	11.10
ORP (mV):	-65.70
Eh potential (mV):	145.30

pH 4h later:	10.60
NaOH added:	15 drops (20M)
Final pH:	11.02
ORP (mV):	-51.00
Eh potential (mV):	160.00

pH 5h later:	10.65
ORP (mV):	-90.00
Eh potential (mV):	121.00

Table 63 - Varying rotation speed, measures for the CSQ30 sample

CSQ30	
Initial pH:	10.60
NaOH added:	20 drops (20M)
Final pH:	11.02
ORP (mV):	-63.60
Eh potential (mV):	147.40

pH 1h later:	10.40
NaOH added:	25 drops (20M)
Final pH:	10.90
ORP (mV):	-41.00
Eh potential (mV):	170.00

pH 2h later:	10.55
NaOH added:	15 drops (20M)
Final pH:	10.95
ORP (mV):	-25.30
Eh potential (mV):	185.70

pH 3h later:	10.57
NaOH added:	15 drops (20M)
Final pH:	10.96
ORP (mV):	-39.40
Eh potential (mV):	171.60

pH 4h later:	10.61
NaOH added:	20 drops (20M)
Final pH:	11.03
ORP (mV):	-25.00
Eh potential (mV):	186.00

pH 5h later:	10.63
ORP (mV):	-48.00
Eh potential (mV):	163.00

For these experiments, the pH was more regular for the CSQ10 ore sample, but it also needed to be fixed every hour by a sodium hydroxide solution, considering both samples.

The redox potential was higher for the second ore sample than for the first one, despite its more constant behavior for the CSQ30 sample.

The behavior of the chemical elements is represented on figures 5.40 and 5.41 for CSQ10 and CSQ30 samples, respectively.

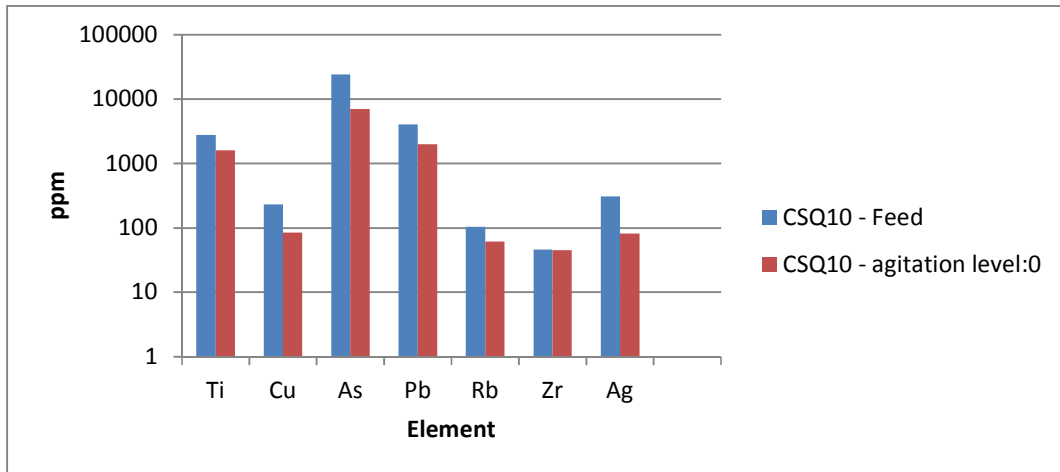


Figure 5.40 - Comparison of the chemical analysis for the CSQ10 sample before and after the test which studied the rotation speed variation to the zero level

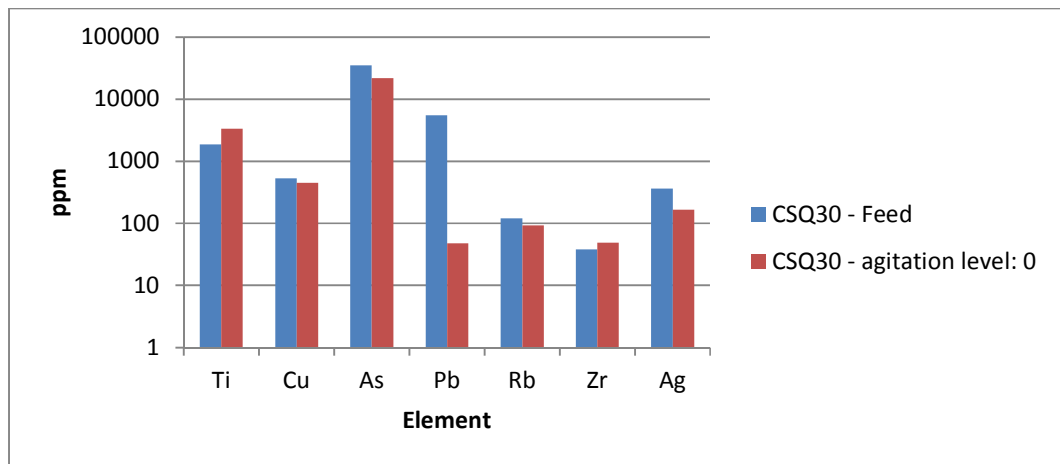


Figure 5.41 - Comparison of the chemical analysis for the CSQ30 sample before and after the test which studied the rotation speed variation to the zero level

Observing the previous images, it is possible to say that some leaching occur since the elements concentration decreased (generally speaking). The only exceptions are on the CSQ30 ore sample: titanium and zirconium. Therefore, the leaching process was not selective for gold.

Regarding the leaching yield, tables 64 and 65 provide the results.

Table 64 - Leaching yield for the CSQ10 sample taking into consideration the influence of speed rotation (level 0)

Rotation speed: minimum - CSQ10	
Au content - feeding (mg/kg)	5.5
Au content - residue (mg/kg)	1.9
Yield (%)	65.45

Table 65 - Leaching yield for the CSQ30 sample taking into consideration the influence of speed rotation (level 0)

Rotation speed: minimum - CSQ30	
Au content - feeding (mg/kg)	8.98
Au content - residue (mg/kg)	0.2
Yield (%)	97.77

The ore samples behaved differently. However, the CSQ30 exhibited an extremely high yield.

- **Performance of the ore samples regarding rotation speed variation**

Concerning pH, figures 5.42 and 5.43 illustrate the behavior during the different leaching tests.

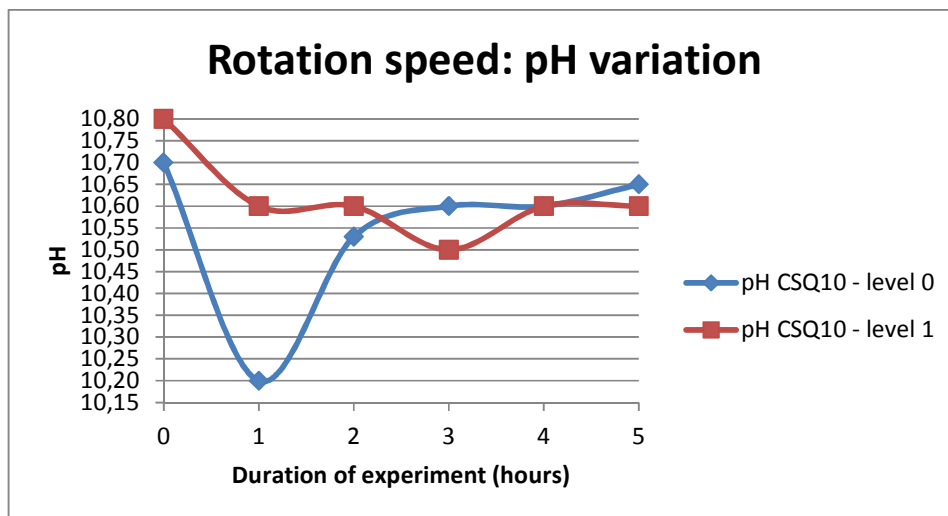


Figure 5.42 - pH behavior regarding the rotation speed variation, for the CSQ10 sample

The pH varied more significantly for the leaching experiment which had a minimal rotation speed. For the control assay, pH did not vary as much.

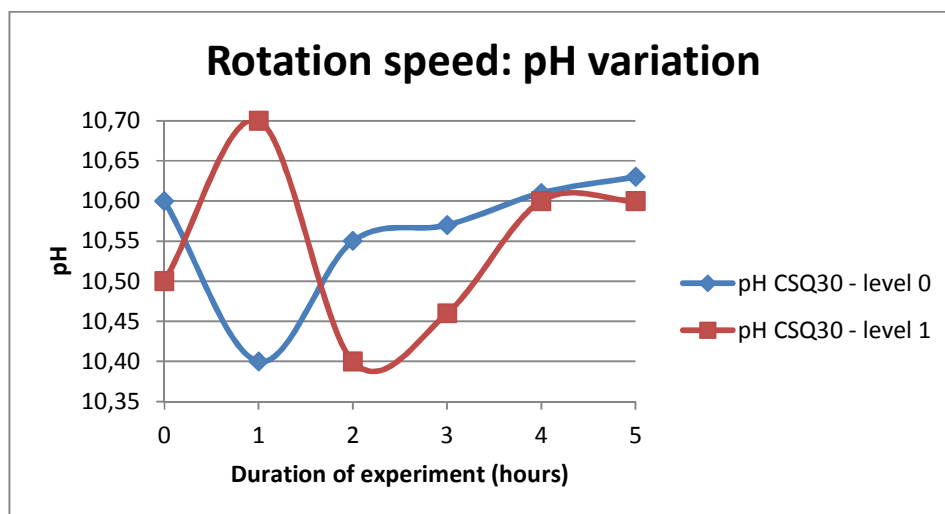


Figure 5.43 - pH behavior regarding the rotation speed variation, for the CSQ30 sample

For this sample the pH varied more considerably for this ore is more unstable. However, this time, the control assay (red line) was the one which varied the most.

Concerning Eh the graphics from figures 5.44 and 5.45 summarize what happened during the leaching experiments for the CSQ10 and CSQ30 ore samples, respectively.

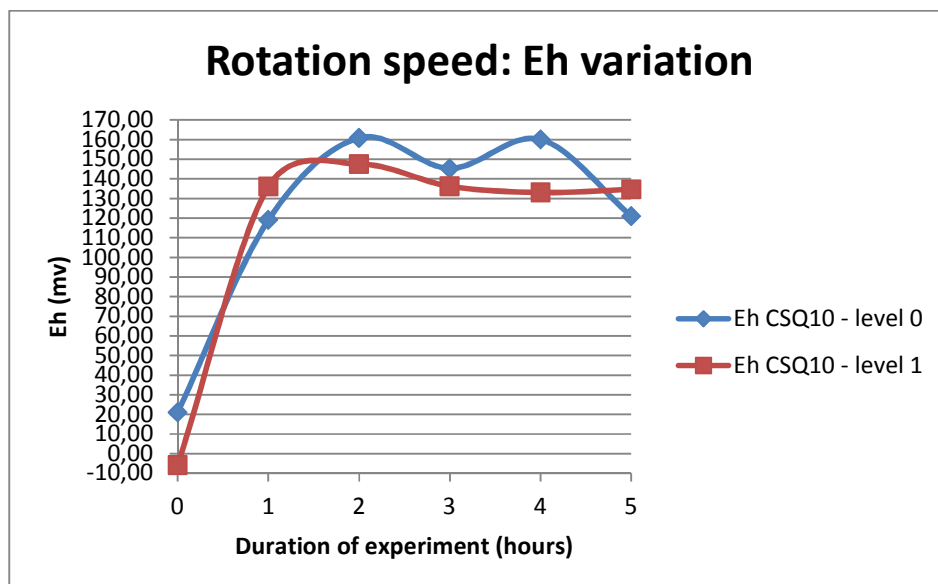


Figure 5.44 - Eh behavior regarding the rotation speed variation, for the CSQ10 sample

For both ore samples the redox potential behavior was similar. However, for the control assay (red line) it was more constant.

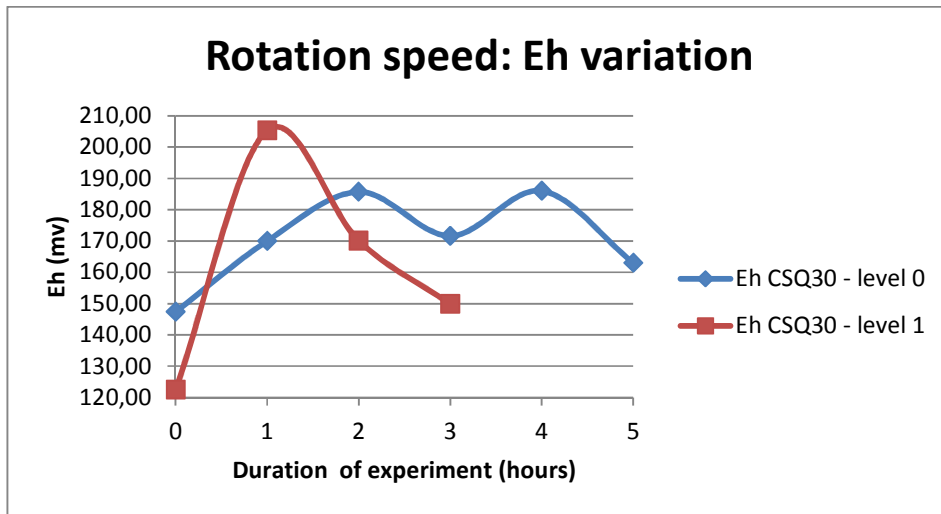


Figure 5.45 - Eh behavior regarding the rotation speed variation, for the CSQ30 sample

For this ore sample Eh had higher values (more positive) than for the sulphides. The materials did not behave the same way as the one represented by the blue line was more constant.

Every leaching yield mentioned on the former assays is now summarized on the figure that follows.

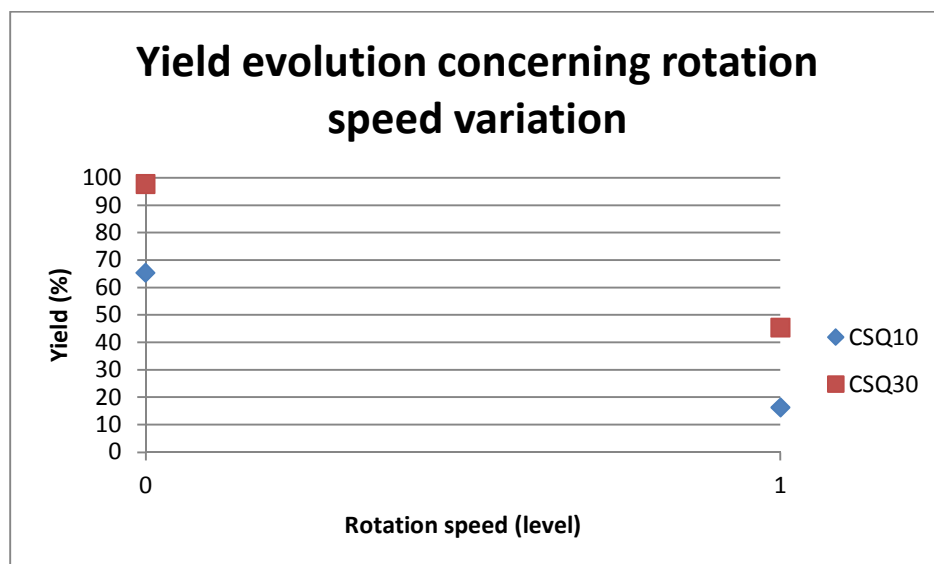


Figure 5.46 - Performance of the ore samples regarding speed rotation variation

Both ore samples exhibited better leaching yield when the speed rotation was at the minimum.

6. CONCLUSIONS

There is a growing need of substituting cyanide as a leaching agent. The ammoniacal-thiosulphate system is one of them.

In this work, several experiments were carried out in order to study what the optimal concentrations for the sodium thiosulphate, copper and ammonia were, as well as the influence of the temperature and speed rotation on the leaching process.

The results differed from one sample to another as they had different characteristics: CSQ10 was a “sulphide” sample while CSQ30 was an “oxidized” sample (both from the Castromil Mines, on Portugal). However, it is important to bear in mind that the analysis of the collected samples had a processing error of 25-30%, so the results are affected by that uncertainty. All yields calculated derived from the residue concentrations as the liquids did not reveal viable results.

During each leaching experiment, pH and redox potential were measured, enabling the following conclusions:

- The CSQ30 (oxidized sample) pH was more unstable, probably because the ore reacted more with the solution.
- The redox potential for the CSQ30 sample was higher, enlightening the tendency of this substance to gain electrons and be reduced.

The procedure consisted of mixing 250g of ore sample with a 375mL of solution composed by thiosulphate, ammonia and copper during 5 hours.

The first experiment was the standard test, where the leaching yield was tested with a solution composed only by sodium thiosulphate. For both ores the results were not very high (as expected): 16.36% for the CSQ10 and 38.75% for the CSQ30.

The second experiment was the control test: all the other assays are variations from this one. This test was also used to study the influence of time on the leaching process, showing that the yield varied differently for each ore sample. Even though it was a comparison from the liquids test, the values used were merely suggestive.

The next stage of the project was to study the sodium thiosulphate concentration variation. The results show that for the sulphide sample the **optimal concentration of thiosulphate is 1 M while for the oxide is 0.5 M.**

The study of the copper concentration was the second stage. For this assembly of experiments, both samples behaved the same way: the **optimal copper concentration was, in both cases, 0.01M.**

The ammonia influence was the last stage of the concentration study and the results show that the **optimal ammonia concentration for the CSQ10 is 3 M while for the CSQ30 was 2 M.**

The temperature study had not expected results: supposedly, the increase of temperature increases the leaching yield. However, **for both cases the optimal temperature was from between 40 and 45 °C.**

During the last set of experiments, the speed rotation had to be reduced because the equipment became unstabilized. For that reason, a final test was taken in order to study the effect of this speed rotation decrease.

Surprisingly, both ore samples reacted really well to this **speed decrease and the leaching yield increased for both cases.** The main reason for keeping the system in rotation is to prevent the solids from settling so it would think that level 1 were the best to do it. However, the almost 100% of the CSQ30 sample says otherwise.

The table that follows condenses the efficiencies obtained for the different leaching experiments.

Concentrations (M)			Temperature (°C)	Speed rotation (level)	CSQ10	CSQ30
thiosulphate	copper	ammonia			yield (%)	
0.5	0	0	room temperature	1	16.36	38.75
0.5	0.001	1	room temperature	1	16.36	45.43
0.1	0.001	1	room temperature	1	23.64	33.18
1	0.001	1	room temperature	1	60.00	25.39
2	0.001	1	room temperature	1	---	22.05
0.5	0.0001	1	room temperature	1	61.82	51.00
0.5	0.01	1	room temperature	1	82.55	84.41
0.5	0.001	2	room temperature	1	14.55	99.67
0.5	0.001	3	room temperature	1	34.55	55.46
0.5	0.001	1	60-70	0	29.09	49.89
0.5	0.001	1	40-45	0	86.91	99.11
0.5	0.001	1	room temperature	0	65.45	97.77

Summarizing, the optimal conditions for each sample are:

	Concentrations (M)			Temperature (°C)	Speed rotation (level)
	thiosulphate	copper	ammonia		
CSQ10	1	0.01	2	40-45	0
CSQ30	0.5	0.01	3	40-45	0

7. LIMITATIONS AND FUTURE WORK

The main limitations are related with the results of the gold concentrations. The applied method (flame atomic absorption spectrometer) might not be the most suitable, since the majority of the results which came back from the laboratory did not make much sense, particularly the liquids. On top of that, this method has a 25-30% error associated to it.

Another difficulty held by this assembly of experiments is the filtration/washing duration. In some cases, these procedures went over 15 hours, even when using a vacuum pump. The filters used were not the most suited as well and probably were the main cause of the procedures duration. The incongruity of the liquids concentrations could be due to this fact. Taking plenty of time to filtrate, the gold has time precipitate from the solution onto the residue anew.

In a future work, a leaching experiment should take place for each one of the two Castromil samples, considering their optimal conditions determined in this work. The reason that did not occur during this assembly of experiments was that LNEG's method took very long, so all of the work was based on the procedures done in Akashi and Hishikari mines. The results obtained came to prove that each sample has its unique optimal conditions.

Also a study using the same ore samples and a cyanide solution must be performed as the leaching processes depend on the ore mineralogy, so each case must be analyzed separately. It is relevant to know whether thiosulphate is a more effective alternative to cyanide as the latter substance is harmful and highly toxic, since this set of experiments only serve the purpose of studying the behavior of the ore samples to the ammoniacal-thiosulphate system.

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APPENDIX A

Sub-sample preparation

On this appendix, every Mastersizer 2000 result as well as the chemical analysis performed by the portable X-ray fluorescence analyzer is going to be displayed.

Sub-sample 2

Concerning the CSQ10 ore sample, table 66 and figure A.1 illustrate the Mastersizer results.

Table 66 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ10 sub-sample 2

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	6.40	11.482	61.48
0.011	0.00	0.120	0.00	1.259	7.84	13.183	66.08
0.013	0.00	0.138	0.00	1.445	9.43	15.136	70.62
0.015	0.00	0.158	0.00	1.660	11.21	17.378	75.08
0.017	0.00	0.182	0.00	1.905	13.20	19.953	79.45
0.020	0.00	0.209	0.00	2.188	15.44	22.909	83.66
0.023	0.00	0.240	0.00	2.512	17.94	26.303	87.63
0.026	0.00	0.275	0.00	2.884	20.71	30.200	91.24
0.030	0.00	0.316	0.00	3.311	23.77	34.674	94.34
0.035	0.00	0.363	0.01	3.802	27.11	39.811	96.82
0.040	0.00	0.417	0.18	4.365	30.74	45.709	98.58
0.046	0.00	0.479	0.57	5.012	34.65	52.481	99.65
0.052	0.00	0.550	1.17	5.754	38.79	60.256	99.99
0.060	0.00	0.631	1.94	6.607	43.13	69.183	100.00
0.069	0.00	0.724	2.86	7.586	47.63	79.433	100.00
0.079	0.00	0.832	3.92	8.710	52.21	91.201	100.00
0.091	0.00	0.955	5.10	10.000	56.85	104.713	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 69.183 µm
- D_{80} is 19.953 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The graphic that follows shows the particle size distribution obtained from the previous table.

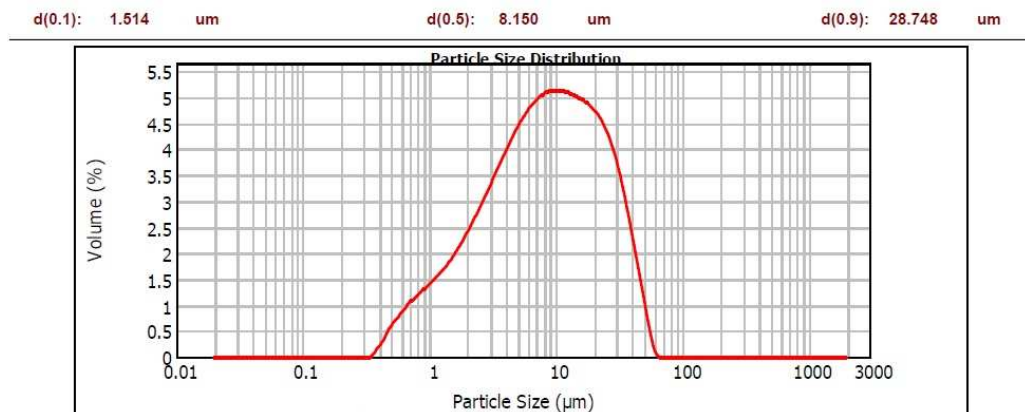


Figure A.1 - Particle size distribution, using Mastersizer 2000 for the CSQ10 sub-sample 2

Regarding the CSQ30 sub-sample, table 67 and figure A.2 illustrate the results obtained from the Mastersizer 2000.

Table 67 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ30 sub-sample 2

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	10.60	11.482	56.48
0.011	0.00	0.120	0.00	1.259	12.02	13.183	60.33
0.013	0.00	0.138	0.00	1.445	13.49	15.136	64.14
0.015	0.00	0.158	0.00	1.660	15.07	17.378	67.92
0.017	0.00	0.182	0.00	1.905	16.79	19.953	71.66
0.020	0.00	0.209	0.00	2.188	18.69	22.909	75.36
0.023	0.00	0.240	0.00	2.512	20.79	26.303	79.03
0.026	0.00	0.275	0.03	2.884	23.10	30.200	82.64
0.030	0.00	0.316	0.20	3.311	25.62	34.674	86.15
0.035	0.00	0.363	0.70	3.802	28.36	39.811	89.47
0.040	0.00	0.417	1.46	4.365	31.32	45.709	92.51
0.046	0.00	0.479	2.46	5.012	34.48	52.481	95.13
0.052	0.00	0.550	3.65	5.754	37.83	60.256	97.22
0.060	0.00	0.631	4.96	6.607	41.35	69.183	98.71
0.069	0.00	0.724	6.35	7.586	45.02	79.433	99.60
0.079	0.00	0.832	7.77	8.710	48.79	91.201	99.97
0.091	0.00	0.955	9.19	10.000	52.62	104.713	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 104.713 µm
- D₈₀ is 26.303 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The figure that follows illustrate the results from the previous table.

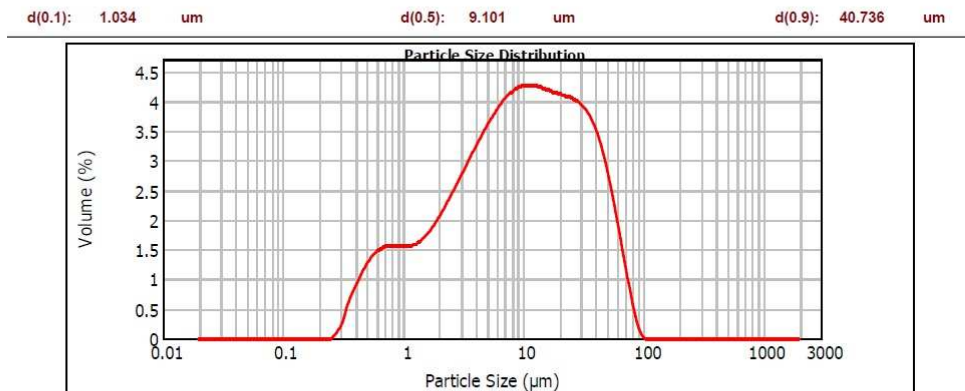


Figure A.2 - Particle size distribution, using Mastersizer 2000 for the CSQ30 sub-sample 2

The chemical compositions for both ore samples are presented on tables 68 and 69.

Table 68 - Chemical composition for the CSQ10 sub-sample

CSQ10	ppm	+/-
S	91582	9564
Ti	2067	288
Fe	>10%	1%
Cu	180	17
As	19945	282
Pb	3411	59
Rb	102	4
Zr	22	3
Ag	196	18
I	<661	
Ca	<923	
K	<6413	
Cl	<9159	
P	<90854	
Ba	<373	
Cr	<190	
Mn	<124	
Co	<547	

Table 69 - Chemical composition for the CSQ30 sub-sample

CSQ30	ppm	+/-
Ti	2433	370
Fe	>10%	1%
Cu	569	37
Zn	102	21
As	34747	681
Pb	5742	128
Rb	102	6
Zr	32	5
Ag	331	25
I	<818	
Ca	<980	
K	<5971	
Cl	<9126	
S	<22252	
Pb	<95930	
Ba	<491	
Cr	<233	
Mn	<168	

Ni	<95
Zn	<29
Hg	<56
Se	<20
Sr	<7
Mo	<12
Cd	<61
Sn	<101
Sb	<107

Co	<1497
Ni	<219
Hg	<101
Se	<34
Sr	<11
Mo	<18
Cd	<85
Sn	<142
Sb	<152

Sub-sample 3

Concerning the CSQ10 ore sample, table 70 and figure A.3 illustrate the Mastersizer results.

Table 70 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ10 sub-sample 3

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.106	0.00	1.096	3.19	11.482	48.20	120.226	97.72
0.011	0.00	0.120	0.00	1.259	4.18	13.183	49.94	138.038	98.85
0.013	0.00	0.138	0.00	1.445	5.37	15.136	53.73	158.489	99.60
0.015	0.00	0.158	0.00	1.660	6.78	17.378	57.55	181.970	99.93
0.017	0.00	0.182	0.00	1.905	8.44	19.953	61.38	208.930	100.00
0.020	0.00	0.209	0.00	2.188	10.34	22.909	65.16	239.893	100.00
0.023	0.00	0.240	0.00	2.512	12.48	26.303	68.87	275.423	100.00
0.026	0.00	0.275	0.00	2.884	14.84	30.200	72.47	316.228	100.00
0.030	0.00	0.316	0.00	3.311	17.38	34.674	75.91	363.079	100.00
0.035	0.00	0.363	0.00	3.802	20.09	39.811	79.16	416.969	100.00
0.040	0.00	0.417	0.00	4.365	22.93	45.709	82.20	478.630	100.00
0.046	0.00	0.479	0.11	5.012	25.91	52.481	85.03	549.541	100.00
0.052	0.00	0.550	0.33	5.754	29.00	60.256	87.66	630.957	100.00
0.060	0.00	0.631	0.67	6.607	32.21	69.183	90.10	724.436	100.00
0.069	0.00	0.724	1.12	7.598	35.54	79.433	92.36	831.784	100.00
0.079	0.00	0.832	1.68	8.710	38.98	91.201	94.42	954.993	100.00
0.091	0.00	0.956	2.37	10.000	42.54	104.713	96.22	1096.478	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 208.930 µm
- D₈₀ is 39.811 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The figure that follows illustrate the results from the previous table.

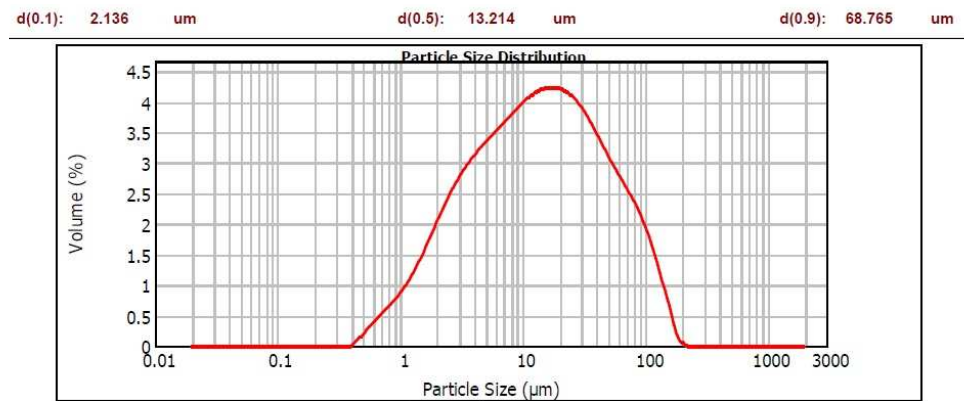


Figure A.3 - Particle size distribution, using Mastersizer 2000 for the CSQ10 sub-sample 3

Regarding the CSQ30 sub-sample, figure A.4 and table 71 illustrate the results obtained from the Mastersizer 2000.

Table 71 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ30 sub-sample 3

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	10.12	11.482	54.94	120.226	98.93
0.011	0.00	0.120	0.00	1.259	11.58	13.183	58.58	138.038	99.58
0.013	0.00	0.138	0.00	1.445	13.10	15.136	62.19	158.489	99.90
0.015	0.00	0.158	0.00	1.660	14.74	17.378	65.75	181.970	100.00
0.017	0.00	0.182	0.00	1.905	16.53	19.953	69.21	208.930	100.00
0.020	0.00	0.209	0.00	2.188	18.49	22.909	72.56	239.883	100.00
0.023	0.00	0.240	0.00	2.512	20.64	26.303	75.78	275.423	100.00
0.026	0.00	0.275	0.02	2.884	22.98	30.200	78.85	316.228	100.00
0.030	0.00	0.316	0.17	3.311	25.52	34.674	81.76	363.078	100.00
0.035	0.00	0.363	0.63	3.802	28.24	39.811	84.51	418.969	100.00
0.040	0.00	0.417	1.33	4.365	31.14	45.709	87.09	478.630	100.00
0.046	0.00	0.479	2.25	5.012	34.20	52.481	89.48	549.541	100.00
0.052	0.00	0.550	3.36	5.754	37.41	60.256	91.66	630.957	100.00
0.060	0.00	0.631	4.80	6.607	40.74	69.183	93.62	724.436	100.00
0.069	0.00	0.724	5.93	7.586	44.19	79.433	95.34	831.764	100.00
0.079	0.00	0.832	7.31	8.710	47.72	91.201	96.81	954.993	100.00
0.091	0.00	0.955	8.71	10.000	51.31	104.713	98.01	1096.478	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 181.970 µm
- D_{80} is 34.674 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The graphic that follows shows the particle size distribution obtained from the previous table.

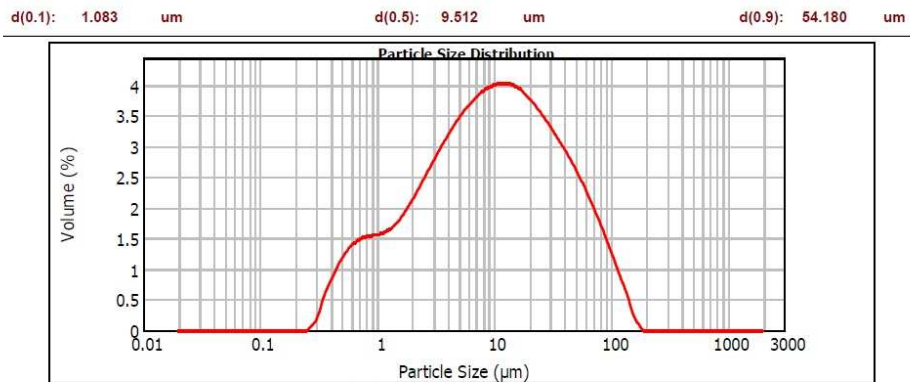


Figure A.4 - Particle size distribution, using Mastersizer 2000 for the CSQ30 sub-sample 3

The chemical compositions for both ore samples are presented on tables 72 and 73.

Table 72 - Chemical composition for the CSQ10 sub-sample

CSQ10	ppm	+/-
S	>10%	1%
Ti	1977	293
Mn	195	47
Fe	>10%	1%
Co	744	194
Cu	246	19
As	21332	310
Pb	3377	61
Rb	90	4
Zr	26	3
Ag	254	19
I	<688	
Ca	<925	
K	<6114	
Cl	<9446	
P	<94369	
Ba	<385	
Cr	<216	
Ni	<103	
Zn	<31	
Hg	<61	
Se	<21	
Sr	<7	
Mo	<12	
Cd	<64	
Sn	<107	
Sb	<114	

Table 73 - Chemical composition for the CSQ10 sub-sample

CSQ30	ppm	+/-
Ti	2531	374
Fe	>10%	1%
Cu	553	36
Zn	123	21
As	31529	602
Pb	7155	151
Rb	110	6
Sr	12	4
Zr	24	5
Mo	29	6
Ag	249	24
Sn	146	46
I	<816	
Ca	<932	
K	<6085	
Cl	<9109	
S	<22052	
P	<102115	
Ba	<497	
Cr	<230	
Mn	<168	
Co	<1451	
Ni	<210	
Hg	<99	
Se	<33	
Cd	<83	
Sb	<148	

Sub-sample 4

Concerning the CSQ10 ore sample, table 74 and figure A.5 illustrate the Mastersizer results.

Table 74 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ10 sub-sample 4

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	3.19	11.482	45.81	120.226	97.15
0.011	0.00	0.120	0.00	1.259	4.18	13.183	49.28	138.038	98.34
0.013	0.00	0.138	0.00	1.445	5.36	15.136	52.79	158.489	99.20
0.015	0.00	0.158	0.00	1.660	6.76	17.378	56.33	181.970	99.76
0.017	0.00	0.182	0.00	1.905	8.40	19.953	59.88	208.930	100.00
0.020	0.00	0.209	0.00	2.188	10.28	22.909	63.42	239.883	100.00
0.023	0.00	0.240	0.00	2.512	12.42	26.303	66.93	275.423	100.00
0.026	0.00	0.275	0.00	2.884	14.78	30.200	70.39	316.228	100.00
0.030	0.00	0.316	0.00	3.311	17.36	34.674	73.79	363.078	100.00
0.035	0.00	0.363	0.00	3.802	20.12	39.811	77.12	416.869	100.00
0.040	0.00	0.417	0.00	4.365	23.03	45.709	80.34	478.630	100.00
0.046	0.00	0.479	0.11	5.012	26.07	52.481	83.44	549.541	100.00
0.052	0.00	0.550	0.32	5.754	29.20	60.256	86.38	630.957	100.00
0.060	0.00	0.631	0.66	6.607	32.41	69.183	89.11	724.436	100.00
0.069	0.00	0.724	1.11	7.586	35.68	79.433	91.59	831.764	100.00
0.079	0.00	0.832	1.68	8.710	39.01	91.201	93.77	954.993	100.00
0.091	0.00	0.955	2.37	10.000	42.38	104.713	95.63	1096.478	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 208.903 µm
- D_{80} is 45.709 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The graphic that follows shows the particle size distribution obtained from the previous table.

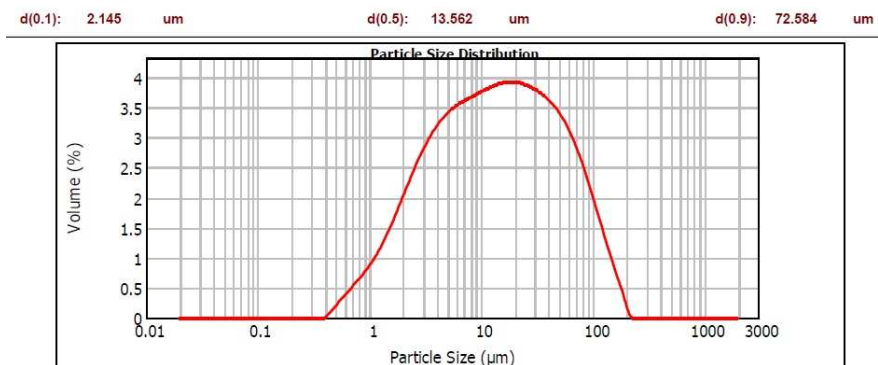


Figure A.5 - Particle size distribution, using Mastersizer 2000 for the CSQ10 sub-sample 4

Regarding the CSQ30 sub-sample, table 75 and figure A.6 illustrate the results obtained from the Mastersizer 2000.

Table 75 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ30 sub-sample 4

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	15.48	11.482	72.53
0.011	0.00	0.120	0.00	1.259	17.38	13.183	76.61
0.013	0.00	0.138	0.00	1.445	19.34	15.136	80.66
0.015	0.00	0.158	0.00	1.660	21.46	17.378	84.62
0.017	0.00	0.182	0.00	1.905	23.79	19.953	88.40
0.020	0.00	0.209	0.00	2.188	26.39	22.909	91.89
0.023	0.00	0.240	0.00	2.512	29.28	26.303	94.92
0.026	0.00	0.275	0.04	2.884	32.48	30.200	97.33
0.030	0.00	0.316	0.47	3.311	35.96	34.674	99.03
0.036	0.00	0.363	1.26	3.802	39.69	39.811	99.84
0.040	0.00	0.417	2.43	4.365	43.61	45.709	100.00
0.046	0.00	0.479	3.94	5.012	47.68	52.481	100.00
0.052	0.00	0.550	5.69	5.754	51.82	60.256	100.00
0.060	0.00	0.631	7.61	6.607	56.00	69.183	100.00
0.069	0.00	0.724	9.61	7.586	60.17	79.433	100.00
0.079	0.00	0.832	11.61	8.710	64.32	91.201	100.00
0.091	0.00	0.955	13.57	10.000	68.44	104.713	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 45.709 µm
- D₈₀ is 15.136 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The figure that follows illustrate the results from the previous table.

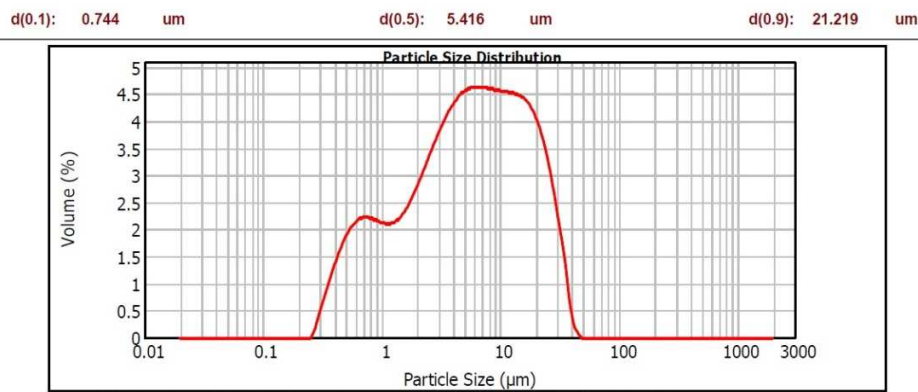


Figure A.6 - Particle size distribution, using Mastersizer 2000 for the CSQ30 sub-sample 4

The chemical compositions for both ore samples are presented on tables 76 and 77.

Table 76 - Chemical composition for the CSQ10 sub-sample

CSQ10	ppm	+/-
S	>10%	1%
Ti	2284	315
Fe	188	50%
Cu	>10%	1%
Zn	224	18
As	16945	242
Pb	3619	62
Rb	85	42
Sr	8	3
Zr	29	18
Ag	174	
I	<718	
Ca	<991	
K	<6563	
Cl	<10210	
P	<110393	
Ba	<409	
Cr	<230	
Co	<544	
Ni	<94	
Zn	<29	
Hg	<53	
Se	<19	
Mo	<12	
Cd	<61	
Sn	<103	
Sb	<110	

Table 77 - Chemical composition for the CSQ30 sub-sample

CSQ30	ppm	+/-
Ti	2517	344
Fe	>10%	1%
Co	1568	448
Cu	524	33
Zn	97	18
As	30791	559
Pb	5658	1147
Rb	111	6
Sr	11	5
Zr	21	20
Ag	294	
I	<748	
Ca	<876	
K	<5565	
Cl	<8081	
S	<20839	
P	<93208	
Ba	<454	
Cr	<219	
Mn	<154	
Ni	<195	
Hg	<87	
Se	<31	
Mo	<17	
Cd	<79	
Sn	<130	
Sb	<141	

Sub-sample 5

Concerning the CSQ10 ore sample, table 78 and figure A.7 illustrate the Mastersizer results.

Table 78 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ10 sub-sample 5

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	6.18	11.482	61.01
0.011	0.00	0.120	0.00	1.259	7.49	13.183	65.37
0.013	0.00	0.138	0.00	1.445	8.95	15.136	69.61
0.015	0.00	0.158	0.00	1.660	10.58	17.378	73.78
0.017	0.00	0.182	0.00	1.905	12.44	19.953	77.91
0.020	0.00	0.209	0.00	2.188	14.56	22.909	82.00
0.023	0.00	0.240	0.00	2.512	16.97	26.303	86.02
0.026	0.00	0.275	0.00	2.884	19.70	30.200	89.86
0.030	0.00	0.316	0.00	3.311	22.76	34.674	93.35
0.035	0.00	0.363	0.01	3.802	26.17	39.811	96.27
0.040	0.00	0.417	0.20	4.365	29.90	45.709	98.41
0.046	0.00	0.479	0.60	5.012	33.95	52.481	99.65
0.052	0.00	0.550	1.19	5.754	38.25	60.256	99.99
0.060	0.00	0.631	1.95	6.607	42.74	69.183	100.00
0.069	0.00	0.724	2.85	7.586	47.34	79.433	100.00
0.079	0.00	0.832	3.87	8.710	51.96	91.201	100.00
0.091	0.00	0.955	4.98	10.000	56.54	104.713	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 69.183 µm
- D_{80} is 19.953 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The figure that follows illustrate the results from the previous table.

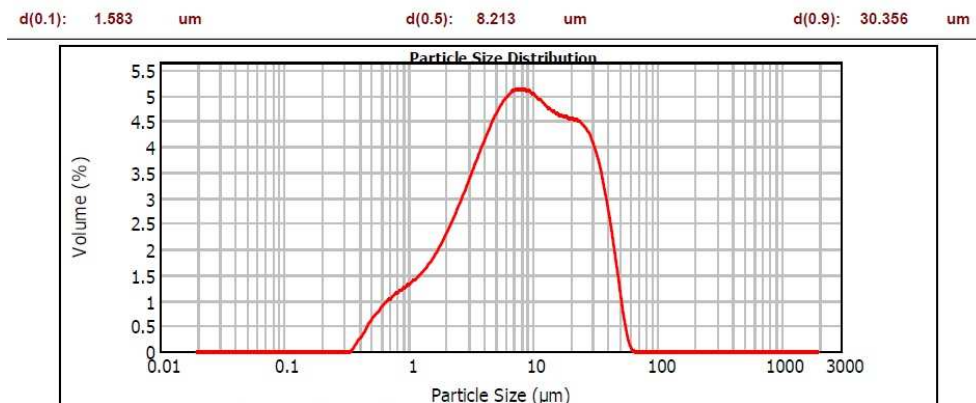


Figure A.7 - Particle size distribution, using Mastersizer 2000 for the CSQ10 sub-sample 5

Regarding the CSQ30 sub-sample, table 79 and figure A.8 illustrate the results obtained from the Mastersizer 2000.

Table 79 - Particle size analysis performed by Mastersizer 2000 with reference to CSQ30 sub-sample 5

Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.105	0.00	1.096	7.84	11.482	53.97	120.226	98.08
0.011	0.00	0.120	0.00	1.259	8.99	13.183	57.98	138.038	98.91
0.013	0.00	0.138	0.00	1.445	10.20	15.136	61.89	158.489	99.51
0.015	0.00	0.158	0.00	1.660	11.52	17.378	65.65	181.970	99.86
0.017	0.00	0.182	0.00	1.905	13.01	19.953	69.25	208.930	100.00
0.020	0.00	0.209	0.00	2.188	14.71	22.909	72.66	239.883	100.00
0.023	0.00	0.240	0.00	2.512	16.65	26.303	75.87	275.423	100.00
0.026	0.00	0.275	0.00	2.884	18.85	30.200	78.87	316.228	100.00
0.030	0.00	0.316	0.01	3.311	21.34	34.674	81.65	363.078	100.00
0.035	0.00	0.363	0.31	3.802	24.13	39.811	84.23	416.869	100.00
0.040	0.00	0.417	0.82	4.365	27.19	45.709	86.61	478.630	100.00
0.046	0.00	0.479	1.54	5.012	30.53	52.481	88.79	549.541	100.00
0.052	0.00	0.550	2.42	5.754	34.10	60.256	90.80	630.957	100.00
0.060	0.00	0.631	3.42	6.607	37.87	69.183	92.63	724.436	100.00
0.069	0.00	0.724	4.50	7.586	41.80	79.433	94.28	831.764	100.00
0.079	0.00	0.832	5.61	8.710	45.83	91.201	95.75	954.993	100.00
0.091	0.00	0.955	6.72	10.000	49.91	104.713	97.02	1096.478	100.00

From the previous table it possible to conclude that:

- 100% of the material is under 208.930 µm
- D₈₀ is 34.674 µm, which means that it is enough for the experiment and there is no need of reducing the particle size even more.

The graphic that follows shows the particle size distribution obtained from the previous table.

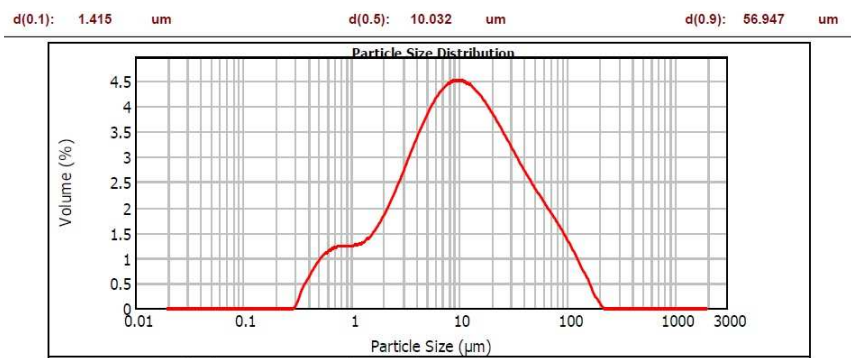


Figure A.8 - Particle size distribution, using Mastersizer 2000 for the CSQ30 sub-sample 5

The chemical compositions for both ore samples are presented on tables 80 and 81.

Table 80 - Chemical composition for the CSQ10 sub-sample

CSQ10	ppm	+/-
S	>10%	1%
Ti	2796	354
Fe	>10%	1%
Co	825	217
Cu	232	20
As	24203	369
Pb	4087	74
Rb	104	5
Zr	46	4
Ag	306	20
I	<788	
Ca	<1073	
K	<7806	
Cl	<11623	
P	<127857	
Ba	<448	
Cr	<231	
Mn	<158	
Ni	<111	
Zn	<34	
Hg	<66	
Se	<23	
Sr	<8	
Mo	<13	
Cd	<66	
Sn	<112	
Sb	<118	

Table 81 - Chemical composition for the CSQ10 sub-sample

CSQ30	ppm	+/-
S	24922	7767
Ti	1861	363
Ba	568	165
Mn	440	58
Fe	>10%	1%
Co	1769	453
Cu	532	34
Zn	73	19
As	35035	652
Pb	5519	118
Rb	120	6
Zr	38	5
Mo	22	6
Ag	363	25
I	<835	
Ca	<959	
K	<5996	
Cl	<8529	
Pb	<105556	
P	<226	
Ni	<195	
Hg	<98	
Se	<32	
Sr	<11	
Cd	<81	
Sn	<137	
Sb	<146	

APPENDIX B

Chemical analysis - residues

In this appendix, the chemical compositions for the CSQ10 and CSQ30 samples after each leaching are going to be displayed.

Assay 2: Influence of time on the leaching process

Table 82 - Chemical analysis for the CSQ10 sample after leaching, influence of time test

CSQ10	ppm	+/-
S	>10%	1%
Ti	3785	441
Mn	204	62%
Fe	>10%	1%
Cu	194	18
As	12574	187
Pb	3167	57
Rb	122	5
Zr	44	4
Ag	123	18
I	<949	
Ca	<1444	
K	<9970	
Cl	<13996	
P	<131877	
Ba	<526	
Cr	<264	
Co	<539	
Ni	<95	
Zn	<28	
Hg	<48	
Se	<17	
Sr	<7	
Mo	<12	
Cd	<63	

Table 83 - Chemical analysis for the CSQ30 sample after leaching, influence of time test

CSQ30	ppm	+/-
Ti	2933	386
Fe	>10%	1%
Cu	577	33
Zn	97	17
As	19231	341
Pb	5197	105
Rb	110	6
Sr	12	3
Zr	45	5
Mo	18	5
Ag	201	22
Sb	162	45
I	<828	
Ca	<940	
K	<6091	
Cl	<9253	
S	<23721	
P	<108644	
Ba	<504	
Cr	<235	
Mn	<172	
Co	<1252	
Ni	<183	
Hg	<70	
Se	<24	

Sn	<106
Sb	<113

Cd	<76
Sn	<127

Assay 3: Varying thiosulphate concentration to 0.1 M

Table 84 - Chemical analysis for the CSQ10 sample after leaching, variation of thiosulphate concentration to 0.1 M test

CSQ10	ppm	+/-
S	87857	10199
Ti	2686	320
Mn	144	47
Fe	>10%	1%
Cu	287	20
Zn	32	10
As	16965	247
Pb	3332	59
Rb	125	5
Zr	53	4
Ag	217	18
I	<729	
Ca	<1045	
K	<7715	
Cl	<10057	
P	<105586	
Ba	<399	
Cr	<199	
Co	<563	
Ni	<97	
Hg	<53	
Se	<19	
Sr	<7	
Mo	<12	
Cd	<63	
Sn	<106	
Sb	<113	

Table 85 - Chemical analysis for the CSQ30 sample after leaching, variation of thiosulphate concentration to 0.1 M test

CSQ30	ppm	+/-
Ti	3094	381
Fe	>10%	1%
Co	2440	457
Cu	601	35
Zn	80	18
As	25791	473
Pb	5687	118
Rb	103	6
Zr	45	5
Mo	18	6
Ag	251	23
I	<855	
Ca	<971	
K	<5774	
Cl	<9219	
S	<23763	
P	<109455	
Ba	<491	
Cr	<232	
Mn	<171	
Ni	<196	
Hg	<82	
Se	<29	
Sr	<10	
Cd	<79	
Sn	<134	
Sb	<143	

Assay 4: Varying thiosulphate concentration to 1 M

Table 86 - Chemical analysis for the CSQ10 sample after leaching, variation of thiosulphate concentration to 1 M test

CSQ10	ppm	+/-
S	84544	8755
Ti	2399	263
Fe	>10%	0%
Cu	112	13
As	9223	123
Pb	2493	42
Rb	85	3
Zr	22	3
Ag	117	16
I	<588	
Ca	<861	
K	<6314	
Cl	<8198	
P	<86325	
Ba	<327	
Cr	<170	
Mn	<115	
Co	<414	
Ni	<72	
Zn	<21	
Hg	<21	
Se	<36	
Sr	<13	
Mo	<6	
Cd	<11	
Sn	<92	
Sb	<99	

Table 87 - Chemical analysis for the CSQ30 sample after leaching, variation of thiosulphate concentration to 1 M test

CSQ30	ppm	+/-
S	47404	11413
Ti	2062	466
Fe	>10%	1%
Cu	445	34
Zn	96	19
As	22791	463
Pb	5108	118
Rb	78	6
Sr	13	4
Zr	23	5
Ag	154	25
I	<1054	
Ca	<1280	
K	<7773	
Cl	<13710	
P	<133849	
Ba	<633	
Cr	<291	
Mn	<209	
Co	<1406	
Ni	<198	
Hg	<81	
Se	<29	
Mo	<18	
Cd	<84	
Sn	<144	
Sb	<151	

Assay 5: Varying thiosulphate concentration to 2 M

Table 88 - Chemical analysis for the CSQ10 sample after leaching, variation of thiosulphate concentration to 2 M test

CSQ10	ppm	+/-
S	77163	9047
Ti	2757	291
Fe	>10%	0%
Co	483	141
Cu	132	14
As	9750	131
Pb	2382	41
Rb	81	3
Zr	38	3
Ag	112	16
Sn	118	31
I	<664	
Ca	<904	
K	<6445	
Cl	<9320	
P	<86216	
Ba	<353	
Cr	<183	
Mn	<123	
Ni	<74	
Zn	<22	
Hg	<39	
Se	<14	
Sr	<6	
Mo	<11	
Cd	<56	
Sb	<98	

Table 89 - Chemical analysis for the CSQ30 sample after leaching, variation of thiosulphate concentration to 2 M test

CSQ30	ppm	+/-
Ti	3216	388
Fe	>10%	1%
Cu	400	28
Zn	88	16
As	20563	360
Pb	4945	99
Rb	86	5
Zr	53	5
Ag	195	22
I	<837	
Ca	<940	
K	<5641	
Cl	<9327	
S	<23809	
P	<101074	
Ba	<497	
Cr	<229	
Mn	<163	
Co	<1183	
Ni	<173	
Hg	<71	
Se	<25	
Sr	<10	
Mo	<19	
Cd	<75	
Sn	<124	
Sb	<135	

Assay 6: Varying copper concentration to 0.0001 M

Table 90 - Chemical analysis for the CSQ10 sample after leaching, variation of copper concentration to 0.0001 M test

CSQ10	ppm	+/-
S	>10%	1%
Ti	2047	323
Fe	>10%	1%
Cu	183	17
As	13582	192
Pb	2989	52
Rb	84	4
Zr	34	3
Ag	124	17
I	<726	
Ca	<1012	
K	<6522	
Cl	<10772	
P	<108960	
Ba	<425	
Cr	<232	
Mn	<152	
Co	<525	
Ni	<90	
Zn	<25	
Hg	<47	
Se	<16	
Sr	<6	
Mo	<12	
Cd	<60	
Sn	<101	
Sb	<107	

Table 91 - Chemical analysis for the CSQ30 sample after leaching, variation of copper concentration to 0.0001 M test

CSQ30	ppm	+/-
Ti	2321	372
Fe	>10%	1%
Cu	530	34
Zn	78	18
As	24564	454
Pb	6989	142
Rb	107	6
Sr	20	4
Zr	37	5
Mo	18	6
Ag	187	23
Cd	80	25
I	<797	
Ca	<904	
K	<5870	
Cl	<8763	
S	<22184	
P	<100864	
Ba	<498	
Cr	<229	
Mn	<161	
Co	<1364	
Ni	<198	
Hg	<80	
Se	<29	
Sn	<134	
Sb	<142	

Assay 7: Varying copper concentration to 0.01 M

Table 92 - Chemical analysis for the CSQ10 sample after leaching, variation of copper concentration to 0.01 M test

42C10L	ppm	+/-
S	>10%	1%
Ti	2745	359
Fe	>10%	1%
Co	850	176
Cu	288	19
As	13861	197
Pb	3005	53
Rb	82	4
Zr	28	3
Ag	122	17
I	<789	
Ca	<1120	
K	<7193	
Cl	<11652	
P	<128541	
Ba	<456	
Cr	<261	
Mn	<168	
Ni	<89	
Zn	<26	
Hg	<47	
Se	<17	
Sr	<7	
Mo	<12	
Cd	<60	
Sn	<101	
Sb	<107	

Table 93 - Chemical analysis for the CSQ30 sample after leaching, variation of copper concentration to 0.01 M test

42C30L	ppm	+/-
Ti	2197	367
Fe	>10%	1%
Cu	804	40
Zn	88	18
As	23874	442
Pb	7207	146
Rb	111	6
Sr	19	4
Zr	41	5
Mo	19	6
Ag	181	23
I	<816	
Ca	<1011	
K	<5923	
Cl	<9197	
S	<23216	
P	<99443	
Ba	<493	
Cr	<232	
Mn	<162	
Co	<1383	
Ni	<198	
Hg	<78	
Se	<29	
Cd	<80	
Sn	<133	
Sb	<141	

Assay 8: Varying ammonia concentration to 2 M

Table 94 - Chemical analysis for the CSQ10 sample after leaching, variation of ammonia concentration to 2 M test

CSQ10	ppm	+/-
S	>10%	1%
Ti	2260	339
Mn	312	56
Fe	>10%	1%
Co	702	179
Cu	162	16
As	15038	213
Pb	3180	55
Rb	84	7
Zr	26	4
Ag	149	26
I	<789	
Ca	<1045	
K	<7078	
Cl	<11018	
P	<119689	
Ba	<443	
Cr	<247	
Ni	<92	
Zn	<28	
Hg	<49	
Se	<17	
Sr	<7	
Mo	<12	
Cd	<61	
Sn	<102	
Sb	<108	

Table 95 - Chemical analysis for the CSQ30 sample after leaching, variation of ammonia concentration to 2 M test

CSQ30	ppm	+/-
S	32010	9712
Ti	2365	436
Fe	>10%	1%
Cu	594	39
Zn	91	21
As	30384	608
Pb	6490	145
Rb	136	7
Sr	22	4
Mo	21	6
Ag	277	26
I	<989	
Ca	<1125	
K	<7238	
Cl	<11452	
P	<119560	
Ba	<506	
Cr	<272	
Mn	<190	
Co	<1653	
Ni	<235	
Hg	<93	
Se	<33	
Zr	<15	
Cd	<87	
Sn	<146	
Sb	<156	

Assay 9: Varying ammonia concentration to 3 M

Table 96 - Chemical analysis for the CSQ10 sample after leaching, variation of ammonia concentration to 3 M test

CSQ10	ppm	+/-
S	98762	9570
Ti	1606	256
Fe	>10%	0%
Co	619	155
Cu	163	15
As	9778	134
Pb	2909	49
Rb	69	3
Zr	30	3
Ag	86	16
I	<579	
Ca	<832	
K	<5225	
Cl	<8780	
Pb	<88954	
Ba	<339	
Cr	<185	
Mn	<122	
Ni	<83	
Zn	<22	
Hg	<38	
Se	<14	
Sr	<6	
Mo	<11	
Cd	<56	
Sn	<95	
Sb	<101	

Table 97 - Chemical analysis for the CSQ30 sample after leaching, variation of ammonia concentration to 3 M test

CSQ30	ppm	+/-
Ti	2854	368
Fe	>10%	1%
Cu	509	32
Zn	84	17
As	21153	380
Pb	5924	119
Rb	77	5
Zr	40	5
Mo	28	5
Ag	227	23
I	<789	
Ca	<965	
K	<5231	
Cl	<9291	
S	<22439	
P	<102817	
Ba	<476	
Cr	<228	
Mn	<163	
Co	<1221	
Ni	<178	
Hg	<74	
Se	<26	
Sr	<10	
Cd	<77	
Sn	<131	
Sb	<138	

Assay 10: Varying temperature between 60 and 70 °C

Table 98 - Chemical analysis for the CSQ10 sample after leaching, variation of temperature between 60 and 70 °C

CSQ10	ppm	+/-
S	>10%	1%
Ti	2170	286
Mn	169	46
Fe	>10%	1%
Co	743	157
Cu	200	16
As	4600	70
Pb	3441	55
Rb	80	3
Zr	33	3
Ag	98	16
I	<626	
Ca	<904	
K	<5778	
Cl	<9426	
P	<10861	
Ba	<368	
Cr	<200	
Ni	<81	
Zn	<21	
Hg	<31	
Se	<12	
Sr	<6	
Mo	<11	
Cd	<57	
Sn	<96	
Sb	<102	

Table 99- Chemical analysis for the CSQ30 sample after leaching, variation of temperature between 60 and 70 °C

CSQ30	ppm	+/-
S	67527	20420
Ti	4755	894
Fe	>10%	4%
Cu	450	37
Zn	99	20
As	14780	343
Pb	5067	131
Rb	94	6
Zr	30	6
Mo	26	7
Ag	136	28
I	<1984	
Ca	<2337	
K	<14627	
Cl	<27150	
P	<240249	
Ba	<1127	
Cr	<541	
Mn	<383	
Co	<1536	
Ni	<209	
Hg	<74	
Se	<28	
Sr	<12	
Cd	<97	
Sn	<160	
Sb	<172	

Assay 11: Varying temperature between 40 and 45 °C

Table 100 - Chemical analysis for the CSQ10 sample after leaching, variation of temperature between 40 and 45 °C

CSQ10	ppm	+/-
S	>10%	1%
Ti	1944	330
Fe	>10%	1%
Cu	152	15
As	6110	88
Pb	3214	53
Rb	80	3
Sr	7	2
Zr	38	3
Ag	75	16
I	<747	
Ca	<1086	
K	<6807	
Cl	<11004	
P	<116826	
Ba	<437	
Cr	<231	
Mn	<151	
Co	<454	
Ni	<79	
Zn	<20	
Hg	<32	
Se	<13	
Mo	<11	
Cd	<56	
Sn	<96	
Sb	<101	

Table 101 - Chemical analysis for the CSQ30 sample after leaching, variation of temperature between 40 and 45 °C

CSQ30	ppm	+/-
S	31116	10105
Ti	2486	470
Ba	680	211
Mn	613	77
Fe	>10%	1%
Cu	509	32
Zn	101	17
As	16204	300
Pb	4745	100
Rb	80	5
Zr	61	5
Ag	177	23
Sb	264	47
I	<1129	
Ca	<1306	
K	<6960	
Cl	<12765	
P	<130878	
Cr	<292	
Co	<1222	
Ni	<181	
Hg	<66	
Se	<24	
Sr	<10	
Mo	<16	
Cd	<78	
Sn	<131	

Assay 12: Varying rotation speed

Table 102 - Chemical analysis for the CSQ10 sample after leaching, variation of rotation speed to zero level

CSQ10	ppm	+/-
S	>10%	1%
Ti	1604	258
Fe	>10%	0%
Co	925	146
Cu	84	13
As	7010	97
Pb	1986	36
Rb	62	3
Zr	45	3
Ag	82	16
I	<594	
Ca	<848	
K	<5412	
Cl	<9794	
P	<103568	
Ba	<338	
Cr	<167	
Mn	<120	
Ni	<73	
Zn	<21	
Hg	<33	
Se	<12	
Sr	<6	
Mo	<11	
Cd	<56	
Sn	<93	
Sb	<100	

Table 103 - Chemical analysis for the CSQ30 sample after leaching, variation of rotation speed to zero level

CSQ30	ppm	+/-
Ti	3350	464
Fe	>10%	1%
Cu	453	30
As	21842	388
Pb	4771	98
Rb	93	5
Zr	49	5
Ag	167	22
Sb	164	45
I	<1069	
Ca	<1266	
K	<7564	
Cl	<12024	
S	<29191	
Pb	<129992	
Ba	<592	
Cr	<278	
Mn	<202	
Co	<1130	
Ni	<162	
Zn	<44	
Hg	<71	
Se	<25	
Sr	<10	
Mo	<16	
Cd	<75	
Sn	<127	

APPENDIX C

Leaching yield - calculations

In this appendix, all the yield calculations regarding liquids are going to be displayed.

Through liquid examination, the expression that shows the yield of the process is:

$$\begin{aligned} \text{yield} &= \frac{\text{Au mass in solution}}{\text{Au mass in the feed material}} \\ &= \frac{\text{concentration of Au} \times \text{volume of solution}}{\text{Au mass in the feed material}} \end{aligned}$$

For each experiment, the solution volume was 375mL and the ore weight 250g; a sample was collected after the leaching experiment. After the filtration and washing of the material, the volumes collected were measured and a sample was taken from each.

Assay 1: Standard Test

Table 104 - Leaching yield for the CSQ10 sample on the standard test

Standard Test - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.067
Yield (%)	1.34
Filtrated solution volume (ml)	204
Concentration (mg/L Au)	0.19
Yield (%)	2.82
Washing solution volume (ml)	325
Concentration (mg/L Au)	0.049
Yield (%)	1.16

Table 105 - Leaching yield for the CSQ30 sample on the standard test

Standard Test - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0.054
Yield (%)	0.66
Filtrated solution volume (ml)	200
Concentration (mg/L Au)	0.32
Yield (%)	2.85
Washing solution volume (ml)	278
Concentration (mg/L Au)	0.34
Yield (%)	4.21

Assay 2: Influence of time on the leaching process

This experiment served to purposes: it was the control assay and it studied the influence of time on the leaching yield.

Table 106 - Leaching yield for the CSQ10 sample taking into consideration the influence of time

Influence of time - CSQ10	
Initial volume (ml)	375
Concentration after 1 hour (mg/L Au)	0.66
Yield (%)	18.00
Volume after 3 hours (ml)	345
Concentration after 3 hours (mg/L Au)	0.41
Yield (%)	10.29
Volume after 5 hours (ml)	315
Concentration after 5 hours (mg/L Au)	1.6
Yield (%)	36.65
Volume after 8 hours (ml)	275
Concentration after 8 hours (mg/L Au)	0.22
Yield (%)	4.40
Filtrated solution volume (ml)	167
Concentration (mg/L Au)	0.068
Yield (%)	0.83
Washing solution volume (ml)	320
Concentration (mg/L Au)	<0.025
Yield (%)	---

Table 107 - Leaching yield for the CSQ30 sample taking into consideration the influence of time

Influence of time - CSQ30	
Initial volume (ml)	375
Concentration after 1 hour (mg/L Au)	2.3
Yield (%)	38.42
Volume after 3 hours (ml)	345
Concentration after 3 hours (mg/L Au)	1.9
Yield (%)	29.20
Volume after 5 hours (ml)	315
Concentration after 5 hours (mg/L Au)	1.8
Yield (%)	25.26
Volume after 8 hours (ml)	275
Concentration after 8 hours (mg/L Au)	0.87
Yield (%)	10.66
Filtrated solution volume (ml)	178
Concentration (mg/L Au)	0.56
Yield (%)	4.44
Washing solution volume (ml)	305
Concentration (mg/L Au)	0.36
Yield (%)	4.89

For the CSQ10 sample the yield of the washing process could not be determined because the laboratory did not give back a precise value.

Assay 3: Varying thiosulphate concentration to 0.1 M

Table 108 - Leaching yield for the CSQ10 sample taking into consideration the influence of thiosulphate concentration (0.1 M)

[Thiosulphate] = 0.1 M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.68
Yield (%)	18.55
Filtrated solution volume (ml)	260
Concentration (mg/L Au)	1.2
Yield (%)	22.69
Washing solution volume (ml)	290
Concentration (mg/L Au)	0.25
Yield (%)	5.27

Table 109 - Leaching yield for the CSQ30 sample taking into consideration the influence of thiosulphate concentration (0.1 M)

[Thiosulphate] = 0.1 M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0.42
Yield (%)	7.02
Filtrated solution volume (ml)	245
Concentration (mg/L Au)	0.26
Yield (%)	2.84
Washing solution volume (ml)	290
Concentration (mg/L Au)	0.15
Yield (%)	1.94

Assay 4: Varying thiosulphate concentration to 1 M

Table 110 - Leaching yield for the CSQ10 sample taking into consideration the influence of thiosulphate concentration (1 M)

[Thiosulphate] = 1 M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.25
Yield (%)	6.82
Filtrated solution volume (ml)	240
Concentration (mg/L Au)	0.04
Yield (%)	0.70
Washing solution volume (ml)	345
Concentration (mg/L Au)	0.11
Yield (%)	2.76

Table 111 - Leaching yield for the CSQ30 sample taking into consideration the influence of thiosulphate concentration (1 M)

[Thiosulphate] = 1 M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0,33
Yield (%)	5.51
Filtrated solution volume (ml)	248
Concentration (mg/L Au)	0.21
Yield (%)	2.32
Washing solution volume (ml)	252
Concentration (mg/L Au)	0.25
Yield (%)	2.81

Assay 5: Varying thiosulphate concentration to 2 M

Table 112 - Leaching yield for the CSQ10 sample taking into consideration the influence of thiosulphate concentration (2 M)

[Thiosulphate] = 2 M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.008
Yield (%)	0.22
Filtrated solution volume (ml)	243
Concentration (mg/L Au)	0.027
Yield (%)	0.48
Washing solution volume (ml)	330
Concentration (mg/L Au)	0.12
Yield (%)	2.88

Table 113 - Leaching yield for the CSQ30 sample taking into consideration the influence of thiosulphate concentration (2 M)

[Thiosulphate] = 2 M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0.21
Yield (%)	3.51
Filtrated solution volume (ml)	265
Concentration (mg/L Au)	0.3
Yield (%)	3.54
Washing solution volume (ml)	340
Concentration (mg/L Au)	0.2
Yield (%)	3.03

Assay 6: Varying copper concentration to 0.0001 M

Table 114 - Leaching yield for the CSQ10 sample taking into consideration the influence of copper concentration (0.0001 M)

[Copper] = 0.0001 M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.2
Yield (%)	5.45
Filtrated solution volume (ml)	248
Concentration (mg/L Au)	0.5
Yield (%)	9.02
Washing solution volume (ml)	325
Concentration (mg/L Au)	0.15
Yield (%)	3.55

Table 115 - Leaching yield for the CSQ30 sample taking into consideration the influence of copper concentration (0.0001 M)

[Copper] = 0.0001 M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0.86
Yield (%)	14.37
Filtrated solution volume (ml)	258
Concentration (mg/L Au)	0.73
Yield (%)	8.39
Washing solution volume (ml)	342
Concentration (mg/L Au)	0.32
Yield (%)	4.87

Assay 7: Varying copper concentration to 0.01 M

Table 116 - Leaching yield for the CSQ10 sample taking into consideration the influence of copper concentration (0.01 M)

[Copper] = 0.01 M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	1.8
Yield (%)	49.09
Filtrated solution volume (ml)	248
Concentration (mg/L Au)	1.8
Yield (%)	32.47
Washing solution volume (ml)	305
Concentration (mg/L Au)	0.52
Yield (%)	11.53

Table 117 - Leaching yield for the CSQ30 sample taking into consideration the influence of copper concentration (0.01 M)

[Copper] = 0.01 M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	12
Yield (%)	---
Filtrated solution volume (ml)	245
Concentration (mg/L Au)	3
Yield (%)	32.74
Washing solution volume (ml)	325
Concentration (mg/L Au)	1.4
Yield (%)	20.27

Assay 8: Varying ammonia concentration to 2 M

Table 118 - Leaching yield for the CSQ10 sample taking into consideration the influence of ammonia concentration (2 M)

[Ammonia] = 2 M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	1.5
Yield (%)	40.91
Filtrated solution volume (ml)	215
Concentration (mg/L Au)	0.28
Yield (%)	4.38
Washing solution volume (ml)	332
Concentration (mg/L Au)	0.11
Yield (%)	2.66

Table 119 - Leaching yield for the CSQ30 sample taking into consideration the influence of ammonia concentration (2 M)

[Ammonia] = 2 M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	2.6
Yield (%)	43.43
Filtrated solution volume (ml)	209
Concentration (mg/L Au)	2
Yield (%)	18.62
Washing solution volume (ml)	340
Concentration (mg/L Au)	0.47
Yield (%)	7.12

Assay 9: Varying ammonia concentration to 3 M

Table 120 - Leaching yield for the CSQ10 sample taking into consideration the influence of ammonia concentration (3 M)

[Ammonia] = 3M - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	1.4
Yield (%)	38.18
Filtrated solution volume (ml)	203
Concentration (mg/L Au)	0.37
Yield (%)	5.46
Washing solution volume (ml)	305
Concentration (mg/L Au)	0.1
Yield (%)	2.22

Table 121 - Leaching yield for the CSQ30 sample taking into consideration the influence of ammonia concentration (3 M)

[Ammonia] = 3M - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	3.5
Yield (%)	58.46
Filtrated solution volume (ml)	205
Concentration (mg/L Au)	2.6
Yield (%)	23.74
Washing solution volume (ml)	300
Concentration (mg/L Au)	0.56
Yield (%)	7.48

Assay 10: Varying temperature between 60 and 70 °C

Table 122 - Leaching yield for the CSQ10 sample taking into consideration the influence of temperature variation (60-70 °C)

Temperature: [60-70] - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	<0.05
Yield (%)	---
Filtrated solution volume (ml)	200
Concentration (mg/L Au)	<0.04
Yield (%)	---
Washing solution volume (ml)	350
Concentration (mg/L Au)	<0.04
Yield (%)	---

Table 123 - Leaching yield for the CSQ30 sample taking into consideration the influence of temperature variation (60-70 °C)

Temperature: [60-70] - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0.63
Yield (%)	10.52
Filtrated solution volume (ml)	204
Concentration (mg/L Au)	0.26
Yield (%)	2.36
Washing solution volume (ml)	345
Concentration (mg/L Au)	0.57
Yield (%)	8.76

Assay 11: Varying temperature between 40 and 45 °C

Table 124 - Leaching yield for the CSQ10 sample taking into consideration the influence of temperature variation (40-45 °C)

Temperature: [40-45] - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.03
Yield (%)	0.82
Filtrated solution volume (ml)	217
Concentration (mg/L Au)	0.13
Yield (%)	2.05
Washing solution volume (ml)	349
Concentration (mg/L Au)	0.03
Yield (%)	0.76

Table 125 - Leaching yield for the CSQ30 sample taking into consideration the influence of temperature variation (40-45 °C)

Temperature: [40-45] - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	<0.05
Yield (%)	---
Filtrated solution volume (ml)	208
Concentration (mg/L Au)	1.00
Yield (%)	9.27
Washing solution volume (ml)	350
Concentration (mg/L Au)	0.05
Yield (%)	0.78

Assay 12: Varying rotation speed

Table 126 - Leaching yield for the CSQ10 sample taking into consideration the influence of speed rotation (level 0)

Rotation speed: minimum - CSQ10	
Solution volume (ml)	375
Concentration (mg/L Au)	0.04
Yield (%)	1.09
Filtrated solution volume (ml)	199
Concentration (mg/L Au)	0.73
Yield (%)	10.57
Washing solution volume (ml)	318
Concentration (mg/L Au)	0.27
Yield (%)	6.24

Table 127 - Leaching yield for the CSQ30 sample taking into consideration the influence of speed rotation (level 0)

Rotation speed: minimum - CSQ30	
Solution volume (ml)	375
Concentration (mg/L Au)	0.051
Yield (%)	0.85
Filtrated solution volume (ml)	205
Concentration (mg/L Au)	0.04
Yield (%)	0.37
Washing solution volume (ml)	270
Concentration (mg/L Au)	0.44
Yield (%)	5.29