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Bustamante-Rúa, Moisés Oswaldo; Naranjo-Gómez, Doria María; Daza-Aragón, Alan José; Bustamante-Baena, Pablo; Osorio-Botero, Julián David

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# Flash flotation of free coarse gold using dithiophosphate and dithiocarbamate as a replacement for traditional amalgamation

Moisés Oswaldo Bustamante-Rúa<sup>a</sup>, Doria Maria Naranjo-Gómez<sup>b</sup>, Alan José Daza-Aragón<sup>a</sup>,  
Pablo Bustamante-Baena<sup>a</sup> & Julián David Osorio-Botero<sup>a</sup>

<sup>a</sup> Facultad de Minas, Universidad Nacional de Colombia, Medellín, Colombia. [mobustam@unal.edu.co](mailto:mobustam@unal.edu.co), [ajdazaa@unal.edu.co](mailto:ajdazaa@unal.edu.co),  
[pabustamanteba@unal.edu.co](mailto:pabustamanteba@unal.edu.co), [judosoriobo@unal.edu.co](mailto:judosoriobo@unal.edu.co)

<sup>b</sup> Distribuidora de Químicos Industriales S.A, Medellín, Colombia. [dmmaranjog@unal.edu.co](mailto:dmmaranjog@unal.edu.co)

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## Abstract

This article develops the flash flotation as an alternative method to gravitational separation and amalgamation in order to achieve a greater recovery of coarse native gold through selective hydrophobicity. This method is directly applicated in the discharge of the mills and / or classifiers to recover free and coarse gold; which size is superior than approximately 150 micrometers. Physicochemical characterization of the free gold surfaces was carried out by the measurements of contact angle, Z potential (ZPC) and atomic force microscopy (AFM). The reagent was determined by specific conditions (basic pH, no requirements of an activator and a flotation time of the order of 2 minutes) achieving a superior recovery than 90%. In this case, the isoamyl dialkyl dithiophosphate collector (AERO 3501<sup>®</sup>) was the reagent which basically got the best results.

**Keywords:** flash flotation; free coarse gold; dithiophosphate; ditiocarbamate; amalgamation.

# Flotación flash de oro nativo grueso usando ditiofosfato y ditiocarbamato como remplazo de la amalgamación tradicional

## Resumen

En el presente trabajo se desarrolla la flotación flash como un método alternativo a la separación gravitacional y la amalgamación con el fin de alcanzar una mayor recuperación de oro nativo grueso a través de la hidrofobicidad selectiva. Esta puede ser usada directamente en la descarga de los molinos y/o clasificadores para recuperar oro libre y grueso, cuyo tamaño sea mayor a 150 micrómetros aproximadamente. Se realizó una caracterización fisicoquímica de las superficies de oro libre a partir de las mediciones de ángulo de contacto, potencial Z (ZPC) y microscopia de fuerza atómica (AFM), y se determinó el reactivo que bajo ciertas condiciones (pH básico, sin requerimientos de un activador y un tiempo de flotación del orden de 2 minutos) logró una recuperación mayor al 90%. En este caso, el colector dialquil ditiofosfato isoamilico (AERO 3501<sup>®</sup>) fue el reactivo que presentó los mejores resultados.


**Palabras clave:** flotación flash; oro nativo grueso; ditiofosfato; ditiocarbamato; amalgamación.

## 1. Introduction

The minerals concentration is the operation about the tenor or concentration rise (in percentage) of an ore or mineral rises, through the use of solid-solid separation equipment which allows the segregation of two or more mineralogical species, generating an enrichment of the aforementioned mineral in the stream [1]. The flash flotation

is a method that uses the minerals concentration by means of the physical-chemical mineral properties such as hydrophobicity and hydrophilicity [1-3]. This process is one of the most important for the recovery of gold and other valuable metals [4]. Its main restriction is to recover particles of thick material (range of 400-250  $\mu\text{m}$ ) by gravitational effects that interrupt the mechanism of the froth [5]. Flotation is a complex process which involve studies of hydrodynamic

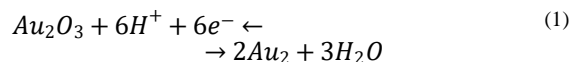
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phenomena and physicochemical surfaces [6]. In addition, the process considers the interaction between several factors, such as: the chemical nature of flotation reagents, equipment components and operation components [5].

The native gold ores which are susceptible to flotation treatment have different mineralogical compositions ranging from free gold ore to metal ore, where the gold constitutes a secondary component recovered as a by-product [7]. The geological shapes of occurrences include placer deposits, hydrothermal and epithermal deposits, volcanic material, sedimentary rocks, metamorphic rocks, rusted and weathered material [8].

The recovering of relatively thick native gold (up to 250  $\mu\text{m}$ ) by means of froth flotation, has inaccuracies respecting to hydrophobic behavior of native gold and its relationship with the aspect ratio of some auriferous particles, their malleability have the tendency to be flattened (flakes), which provides the particles with enough large areas, to reach mechanical buoyancy in the recovery equipments [3,9]. Native gold is an alloy of non-specific composition and natural occurrence that contains around 80-99 % gold, 1-15 % silver and between 0-5 % copper [10]. In a non-complexed aqueous medium, gold is not reactive, since gold ions and oxides are thermodynamically unstable, requiring strongly oxidizing conditions for their formation, Eq. (1)



The oxygen chemisorption upon the gold surface starts at a potential above 1.4 Volts with coating monolayers reached at 2.0 Volts. This is attributed to the growth of an oxide layer ( $\text{Au}_2\text{O}_3$ ) in acid solutions and to the layer of hydroxide ( $\text{Au}(\text{OH})_3$ ) in basic solutions with high potentials. According to voltammetry, it has been determined that these reactions happen in ranges, depending on the orientation of the gold crystals and the presence of impurities [11].

Gold surfaces without patinas are naturally hydrophilic, however, if a monolayer of carbonaceous contaminants is adsorbed from the air or solution, those may be sufficient to produce a hydrophobic surface. As a result, gold can present natural hydrophobicity basically associated with its patinas [12]. The chemical composition of gold particles and their associations with other minerals affects flotation [13] when gold is associated with sulfides, its hydrophobicity is enhanced by the addition of flotation collectors such as xanthates, dithiophosphates and mercaptobenzothiolates [14]. The lack of a good release is a problem in the comminution circuits since it is a key element in the gold extraction operation [15]. Factors that probably affect the buoyancy of free gold are: particle size, absence of scale form, composition, surface contaminants, collector types and cell type [16].

### 1.1. Effect of particle size

The particle size has a great effect on the recovery of gold by flotation due to its high density. The flotation is effective for gold particles in the range of 20-200  $\mu\text{m}$ . For finer sizes, the selectivity of the gold decreases because of the floating of

ore gangue. For thicker particle sizes, flotation must be done at high densities of the mixture (35 % solids) since this reduces the particle size of sedimentation [8].

There is a range of particle size that presents a greater metallurgical recovery, generally the decrease of this could be observed for thicker and finer sizes of the ore [17]. Recovery decreases for small sizes, which is related to the difficulty of particle/bubble adhesion, because they do not acquire enough kinetic energy to produce a stable particle/bubble aggregate. On the other hand, in the optimum size range the particles are more easily dragged into the foam, since the drainage of the pulp is favored with the increase of the sedimentation rate [18]. It is important to note that, in the primary flotation stage ("rougher" stage) the flotation is carried out with an ore granulometry in which the total release of the gold particles is not necessary, however, in the cleaning stage where the selectivity of the particles is necessary, it is essential to carry out a regrind of the concentrate of the "rougher" stage for the release of the useful species of the ore [1,8].

### 1.2. Collector selection and dosage.

The function of the collector is to hydrophobize the surface of the desired mineral, this is the reason why it is the most important chemical reagent used in flotation [19]. The extensive experience in the flotation of minerals allows efficient use of certain types of collectors depending on the types of minerals and the mineralogical associations present where the collectors for the flotation of native gold usually prefer xanthates and dithiophosphates, or mixtures of them [11,20,21]. The xanthates used to oscillate from ethyl xanthate to amyl xanthate. In addition, with sodium dithiophosphate, in amounts from 50 g/t to 200 g/t, respectively [2,9]. In applications in which native gold is presented along with submicrometer gold and unreleased gold included in mineral sulfides, the combination of these reagents has been shown to be efficient for the recovery of sulfides and at the same way for the native gold; so later, cyanidation can be applied to the concentrates to obtain the gold.

The free gold particles can be recovered very selectively, specially against pyrite, keeping the gold surfaces clean from sludge and organic species, without the use of pH regulators, although with the use of small or zero doses of apolar collectors [20]. It should be taken into account that the flotation reagents require a certain conditioning time to be in contact with the pulp, so this way they could react efficiently on the interesting species of the ore [22]. Thus, the conditioning stage acquires great importance, since some reagents must be added in the grinding stage to have greater contact with the ore, while others are added directly to the discharge box of the ball mills or to the conditioner [14].

### 1.3. Activators

For the flotation of native gold with clean surfaces it is not necessary to add activators, however activation may be required if there are coatings in the gold or if there are gold particles associated with coated pyrite. In such cases, copper

sulphate, sodium carbonate (which precipitates calcium and heavy metal ions) sodium sulphide (carefully dosed) and sulfur dioxide are implemented [7,10]. The activators include salts of base metals whose metallic ions are adsorbed on the surface of the mineral particles, modifying their superficial chemical properties; as a consequence, the pH range for mineral flotation can be extended, and the rate of flotation, recovery and selectivity are increased.

## 2. Methodology

### 2.1. Ore sample

The mineral used in the tests belongs to the San Nicolás mine in the municipality of Segovia-Antioquia-Colombia. We worked with a sulfide mineral that contained free gold, whose sample was taken at the discharge of the primary mill. The San Nicolás Mine corresponds to the mining district of Segovia - Remedios, an important mining district of veiniform gold and silver deposits inside of an intrusive granodioritic monolithic (Batolito de Segovia). Which is dated from Jurassic-Triassic [23]. The mineral moisture is 3.2 %, with an apparent density (measured with Burette) of 2.7 g/ml and a real density (measured with a Picnometer) of 2.6 g/ml. The granulometric analysis for the sample to be floated are described in the Fig. 1.

### 2.2. Chemical analysis

The chemical analysis for the sample studied can be seen in the Table 1.

Table 1.  
Chemical analysis of floated samples

Specie	(%)
FeS <sub>2</sub>	77.017
PbS	0.777
ZnS	0.418
CuFeS <sub>2</sub>	0.067
Insolubles	19.633
Total	97.012

Source: The authors.

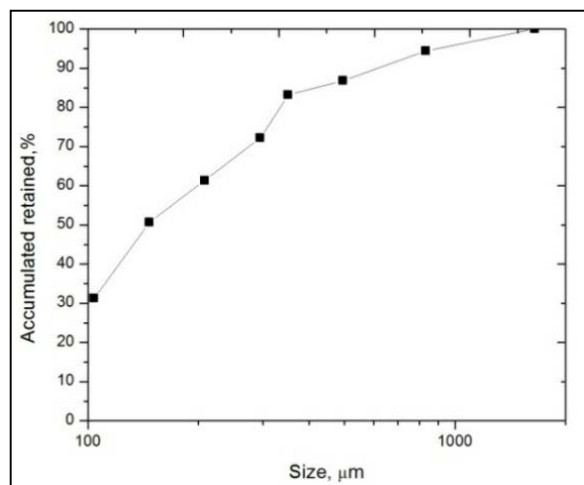


Figure 1. granulometric analysis for the sample.

Source: The authors.

Ignition losses are close to 41.39 % associated with sulfur, obtaining a higher percentage of pyrite, and in the case of galena, sphalerite and calcopitira as less contributors to the floated sample.

### 2.3. Determination of native gold susceptible to floating in a flash cell

The native gold that is susceptible to flotation is determined by means of an unconventional fire proof, which consists in avoid the generation of the famous "nugget effect", using the Colombian patent generated in the research group CIMEX of the National University of Colombia [24]. This method consists in the incorporation of the gravimetric concentration to the conventional tests, allowing to estimate with an accuracy between 1 % and 5%, a precision of 90%, even higher. The gold and silver levels in minerals of an alluvial or vein deposits, are discriminated if the concentration present, in the example, comes from the native element or the element associated with sulfides.

### 2.3. Flotation cell

To perform the flotation tests in the laboratory, it was required to manufacture a prototype flash flotation cell (Fig. 3 and Table 3). To arrive at the design of the optimal prototype, some variables are already established in the designs suggested by the industry were we are taken into account [25].

Based on the parameters previously established and knowing that a special condition is required in the internal geometry of the cell, an internal cone was designed inside the tank, in order to achieve the rises of the particles, therefore reducing the cells area and favoring the ascent of the heavier particles towards the mineralized foam.

(Bernoulli's Principle) The actual volume of the cell is 20 l and its design is shown in Fig. 2.

### 2.4. Reagents

The microflotation tests carried out in the Hallimond cell were previously determined in order the behavior in the selective gold flotation of the different preselected collectors (tionocarbamate and dithiophosphates).

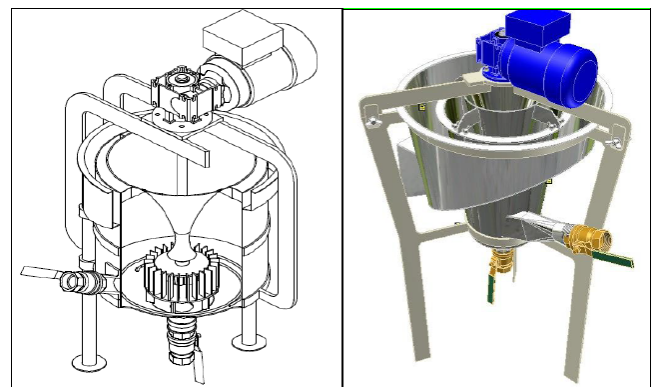


Figure 2. Flotation cell for the test in 3D.

Source: [26,25]

Table 2.  
Gold in samples of native gold susceptible to floating in flash cell.

ID	Total Au (g)			Total Au (%)		
	Sample	Concentrate	Tails	Sample	Concentrate	Tails
+48	0.0378	0.0081	0.0297	100	21.4	78.6
+48	0.0352	0.0081	0.0271	100	23	77
+48'	0.0206	0.0123	0.0083	100	59.8	40.2
+48'	0.0393	0.0123	0.0270	100	31.4	68.6
-48 / +100	0.0108	0.0069	0.0039	100	63.8	36.2
-48 / +100	0.0116	0.0069	0.0048	100	59.2	40.8
-48 / +100'	0.0151	0.0099	0.0052	100	65.8	34.2
-48 / +100'	0.0147	0.0099	0.0048	100	67.2	32.8

Source: The authors.

Table 3.  
Collectors used for mineral flotation.

	Tank volume [m <sup>3</sup> ]	A	B	C	D	E	Weight [kg]
SkimAir® -2400	120	9600	7200	4900	3800	6600	30600
SkimAir® -2400	49	8400	5700	4000	2800	4960	23000
SkimAir® -2400	23	6270	5000	3650		4040	8900
SkimAir® -2400	8	4520	3120	2280		2760	3800
SkimAir® -2400	2.2	3390	2400	1740		1900	1800
SkimAir® -2400	1.3	2920	2175	1575		1640	1260
SkimAir® -2400	0.3	1970	800	800		1290	350

Source: [25]

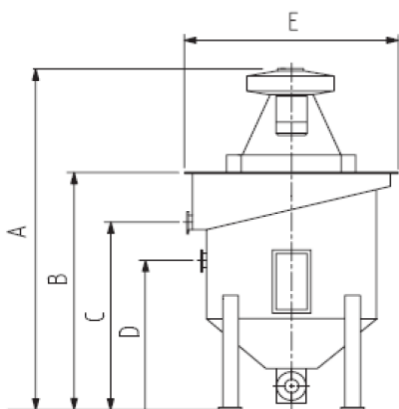


Figure 3. Flotation cell for the test.

Source: [25]

Among the used reagents were: monoalkyl dithionocarbamate (8474), isoamyl dialkyl dithiophosphate (Aero 3501), dithiophosphinate (AEROPHINE 3418 A), dithionocarbamate (S-9411), allyl thionocarbamates allyl thionocarbamates alkyls (5100), isoamyl dialkyl dithiophosphate plus amyl xanthate. (PAX) and allyl, a type dithionocarbamate alkyls plus dithionocarbamate, emphasizing the highest recovery of the first two (88-100%). The flotation conditions were as follows: collector conditioning time 10, 20 or 30 minutes, pH of the solution varying between 4 and 11, water volume of 30 ml, and a glycols type foaming agent.

The Thiol collector-type reagents were selected to favor the chemisorption on metal-type bonds, frequently used in the flotation of metal sulfides.

In Table 4, are present the general characteristics of the used collectors, whose were selected through the microflotation tests of free gold. Both collectors were used for the quantification of

gold hydrophobicity by measuring the contact angle, and at the same way to carrying out the flotation tests.

### 3. Results and discussion

#### 3.1. Contact angle

According to the results obtained from these measurements, could seen that the reagents induce greater hydrophobicity to the gold particles and a better conditioning time for the reagent. Additionally, it was possible to identify the variables to be taken into account in the design of experiments for the flash flotation tests.

Fig. 4 shows the results obtained in the contact angle measurements with the gold particle trough the collector AERO 3501®, selected for the flotation at different conditioning times.

The greater value obtained for the contact angle indicates that the collector induces greater selective hydrophobicity to the gold surface when there is a longer conditioning time and the pH is 11. On the other hand, the reagent S-8474®, provides selective hydrophobicity to the gold surface with a natural pH and a conditioning time of 10 min.

#### 3.2. Potential $\zeta$ and surface free energy

In Fig. 5 the measurement of the zeta potential for gold, pyrite and galena at different basic pH, are shown. A region of zeta potential is observed (between -25 and -50 mV), of which can be said that the gold particle, pyrite and galena are unstable and susceptible to be collected by a surfactant in the flotation process. When it is taken in place at a pH between 8-11. At pH 11, the zeta potential for gold, pyrite and galena, is close to -50 mV, meaning that at a pH of 11, there will be no separation of gold from its accompanying minerals. This result confirms that activators and depressants should be used.

Table 4.  
Collectors used for mineral flotation.

Chemical structure	Comercial reference	Name	Description
$\begin{array}{c} R \quad O \quad S \\ \quad \quad \parallel \\ \quad \quad P-SNa^{+} \\ \quad \quad \parallel \\ R \quad O \end{array}$	AERO 3501®	Isoamyl dialkyl dithiophosphate	Dosages of 5-25 gpt. Solutions of 5-20% or undiluted. Flotation of ZN, Cu and precious metals to float thick particles.
$\begin{array}{c} R \quad S \\ \quad \parallel \\ \quad C \\ \quad \parallel \\ R^1 \quad NH \end{array} \quad S^{\cdot}$	S-8474®	Dithiocarbamate (monoalkyl and dialkyl dithiocarbamates)	Dosages of 5-100 gpt. Solution at 5-20% or undiluted. Flotaminerals of Pb, Zn, Cu, Fe and precious metals. For the flotation of most sulfides and precious metals in neutral circuits and pH..

Source: The authors.

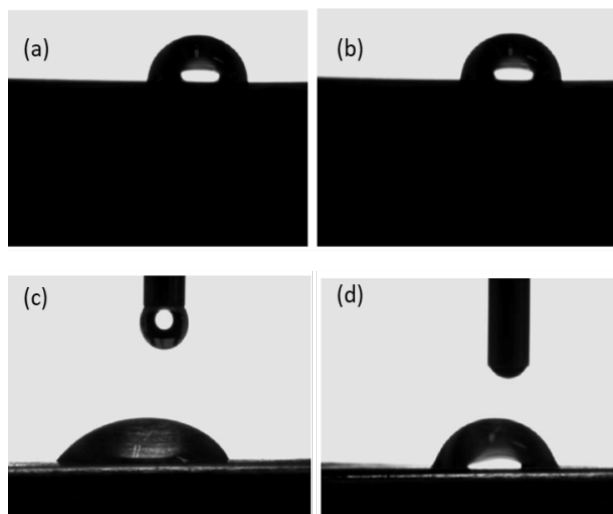


Figure 4. Contact angle for gold samples with 3501 collector (a) 30 minutes (b) 30 minutes and pH. (c) 10 minutes (d) 20 minutes.  
Source: The authors.

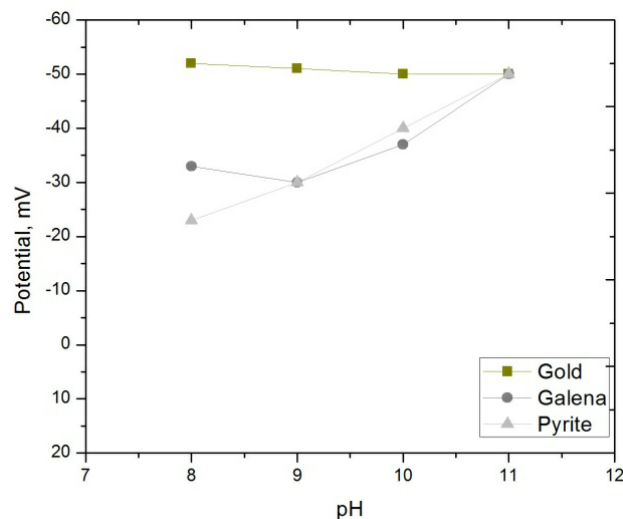


Figure 5. Z potential for gold, pyrite and galena at basic pH.  
Source: The authors.

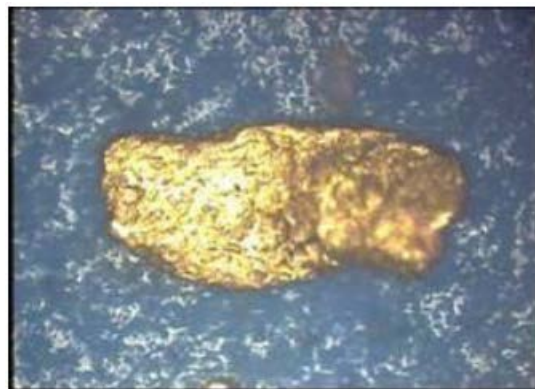


Figure 6. Gold particle.  
Source: The authors.

For the measurement of surface free energy, Atomic Force Microscopy (AFM), we made some measurements to a gold particle without a collector and to a gold particle with a collector (Fig. 6).

With the AFM measurements, it is sought to establish if the surfactants are decreasing the superficial tension in the surface of the gold particle.

The results obtained from the first measurements are shown in Fig. 7.

Comparing both graphs, it can be determined that the energy of the surface with the presence of the collector decreased substantially, which indicates a process of adsorption of the surfactant in the gold-water interface.

With the measurements in the AFM, it is not quantitatively determined which collector induces greater hydrophobicity to the gold surface, but at the same, the effect of different collectors on the surface free energy, as well can be determined.

### 3.4. Operational aspects

The flash cell can be used in the discharge of primary mill, in a small mining and/or in the discharge of classifiers in the return, when grinding circuits are closed (see Fig. 8.). This process competes with the use of mercury and the use of jig and centrifugal gravimetric concentrations, whose are currently used.



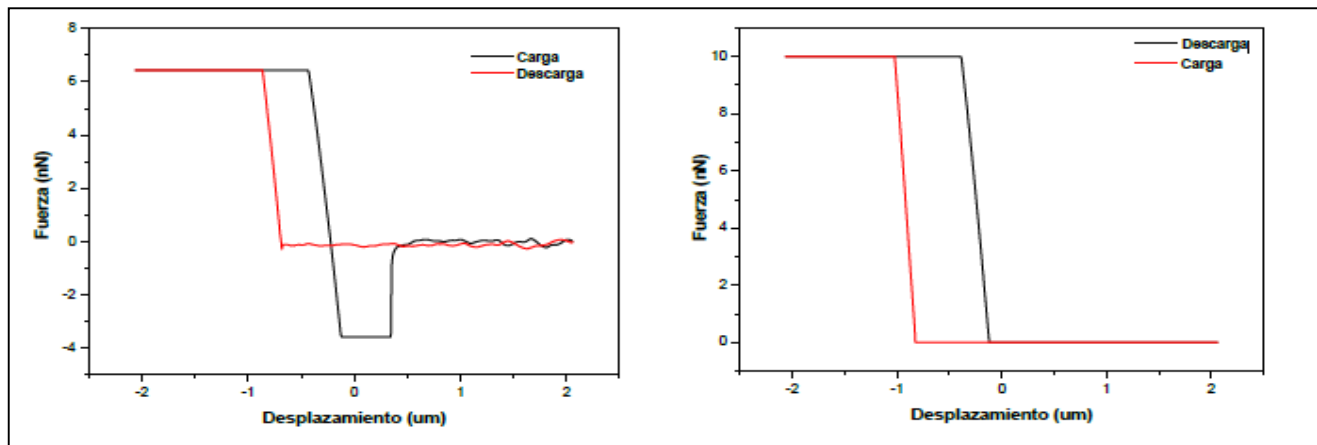


Figure 7. AFM measurements to a gold particle without a collector (left) and to a gold particle with a collector (right).

Source: The authors.

Table 5.

Results of the flotations with the collectors AERO 3501® and S-8474®

Collector	Collector Dosage (g/t)	pH	collector conditioning time (min)	Flotation time (min)	Recovery (%)
3501	50	10.5	10	5	84.56
8474	50	10.5	10	5	97.22
8474	50	10.5	10	2	95.58
3501	50	10.5	10	2	99.28
8474	50	10.5	10	2	95.55
3501	50	10.5	10	2	94.92
3501	50	10.5	10	2	79.10
3501	50	11	10	2	52.20
3501	50	11	10	1	98.21

Source: The authors.

### 3.3. Flotation tests

After the results described above, the best upshots of the tests in Flash flotation are shown in Table 5.

Analyzing the recovery of free gold from the flotation tests carried out with the AERO 3501® and S-8474® collectors, it is observed that the best results were achieved by implementing a 10-min conditioning time and 2-min flotation times, with recoveries of free gold particles of 99.28 % and a conditioning time of 10 min and a flotation times of 5 min., with recoveries of free gold particles of 97.22 %, respectively.

Because of the characteristics of the stream in which a Flash cell is installed, the float gold is thick (greater than 150 μm), therefore this technique is frequently used for the rapid concentration of minerals that have reached their releases in the discharges of the classifiers, allowing to increase the global recovery of the plants, whose main objectives are to avoid the overmoulding of released minerals and at the same way in the recirculation of the stream in the mill.

The main benefits with the use of flash flotation Cells are:

- Lower stock market
- Increase in typical recovery
- Better humidity of concentrates "
- High grade final concentrates are recovered only in a single stage
- Minor volumes in conventional cells.
- 

### 3.4. Environmental impact of the project

The use of mercury in Colombian mining has generated serious environmental and technological impacts, which historically make a detrimental association between gold mining and the soil contamination, the surface water and other actors in the environments around the mines [27].

There is a good correlation between artisanal mining and the increase of mercury losses in the enrichment processes of gold-bearing minerals. While the large-scale mining reports orders of 0.001 g of mercury per gram of gold produced, small-scale mining reports losses of up to 14.5 g of mercury per gram of produced gold on average, whose can amount to 66.5 g of mercury per gram of gold with the minimum values of 7.5 g of mercury per gram of the produced gold [28], which implies global losses of Mercury around 55.1 t/year just in 5 municipalities of Northeast Antioquia: (Segovia, remedies, Nechí, El Bagre, Zaragoza).

It is estimated from the 100 % of the lost, 34 % is emitted to the atmosphere and 66 % goes to the water streams [28]. The technology, traditionally used by miners in these regions, consists of the use of amalgamating coconuts, in which it is estimated that around 50% of the mercury is lost in the process. This project is basically aimed at the elimination of a historical problem, which has been the use of mercury in gold mining, to search the developing of the least environmental impact and to generate conditions that allow improving the relations between mining and the environment in pro of the sustainability of the exploitation of gold.

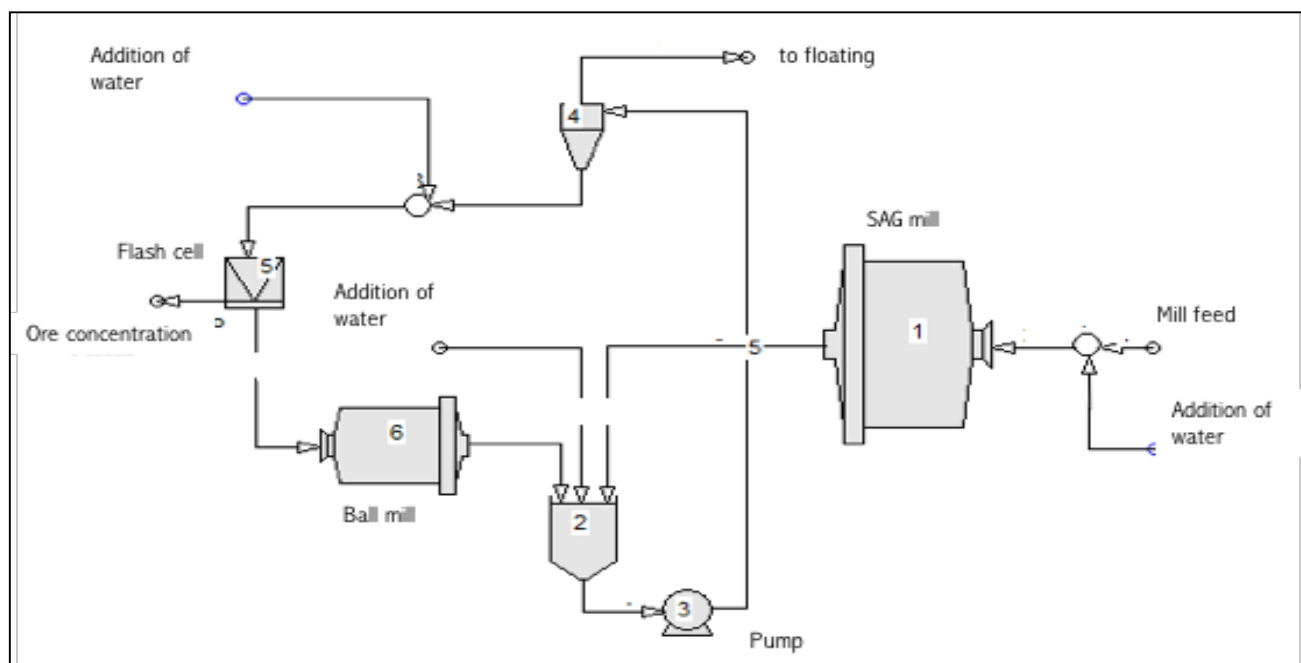


Figure 8. Scheme flash flotation in grinding circuit.

Source: The authors.

#### 4. Conclusion

The surfactant substances adhered to the surface of the particle modify the ZPC, a condition that can favor the adherence or non-adherence of the particles to the air bubbles to guarantee a selective foaming flotation, also an important measure for the flotation of minerals is the contact angle that according to the experimental data of the average value of the contact angle measured on a gold surface, without applying collector is  $43.70^\circ$  and the contact angle for gold can be favorably modified, by means of the addition of different manifolds and collector mixtures, as is the case for mixtures with isoamyl dialkyl dithiophosphate (AERO 3501®), which reached values of up to 96. The reagent used allows to obtain selective hydrophobicity of gold in auriferous ores accompanied by sulphides, such as: pyrites, chalcopyrites, galena and sphalerite, since at no time did the native gold present natural drophobicity. The grain size of the gold particles obtained in the flotation using flash cell is relatively thick, greater than 100 mesh (Tyler, approximately 150 microns). The residence time in the flash flotation as its name indicates is suggested to be short of the order of up to 2 min to 5 min of flotation in the flash cell, depending on which type of collector is used. Times greater than this compromise the selectivity of the flotation and generate a decrease in the gold content in the concentrates that come out in the flotation foam. A prototype of the Flash cell was designed and manufactured, considering hydrodynamic parameters to avoid the sedimentation of too thick gold particles, since the tendency of this type of particles (coarse gold) is to sediment quickly and avoid being collected in mineralized foams and having relatively thick native gold buoyancy in a flash cell designed by this research and that can be an alternative for the direct elimination of mercury.

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**M.O. Bustamante-Rúa**, is BSc. in Mining and Metallurgy Eng. at the Universidad Nacional de Colombia, MSc. in Sciences of Engineering with a Mention in Extractive Metallurgy - Mention - Mineralurgy of the University of Concepción -Chile, DSc. in Engineering Sciences with a Mention Extractive Metallurgy - Mining of the Universidad de Concepción - Chile. Is full professor of the Universidad Nacional de Colombia, Medellín, director and researcher of the Mineral Institute CIMEX. ORCID: 0000-0002-1692-991X

**D.M. Naranjo-Gómez**, is BSc. in Mining and Metallurgy Eng. with a MSc. degree in Materials and Processes. Currently he works in the company Distribuidora de Químicos Industriales S.A. which provides flotation reagents for the mining industry. ORCID: 0000-0002-9052-5788

**P. Bustamante-Baena**, is BSc. in Mining and Metallurgy Eng. at the Universidad Nacional de Colombia. MSc. in Mineral Resources of the same university. Has winner for best undergraduate work and Otto de Greiff National Contest. Belongs to the research group CIMEX Institute of minerals. He is currently the winner of the "Joven Investigador" for Colciencias. Has worked in the industry in the underground coal sector, open pit coal, mineral processing and transformation. ORCID: 0000-0002-8409-5531

**A.J. Daza-Aragón**, is BSc. in Mining and Metallurgy Eng. of the Universidad Nacional de Colombia, finished his studies in 2015, MSc. in Mineral Resources of the Universidad Nacional de Colombia., He was an

auxiliary student of the CIMEX Mineral Institute, where he participated in two seedbeds in association with the National Program of Seeds of Research, Creation and Innovation of the Universidad Nacional de Colombia, 2013-2015.

ORCID: 0000-0001-7596-9666

**J.D. Osorio-Botero**, is BSc. in Mining and Metallurgy Eng. from the Universidad Nacional de Colombia, completed his studies in 2015. He is currently a student of the MSc. in Engineering - Mineral Resources of the Universidad Nacional de Colombia. He works as a support engineer in the project flotation and separation of magnetic minerals that realized between the Universidad Nacional de Colombia and the company AngloGold Ashanti. ORCID: 0000-0002-2845-937X



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