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## Differences in Etheramines Froth Properties and the Effects on Iron Ore Flotation. Part I: Two-Phase Systems

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### ABSTRACT

Etheramines are quartz collectors and also play a role as frothers in cationic reverse flotation of iron ores. While the adsorption of this surfactant on quartz surface has been widely investigated, the impact of its foam/froth properties on the selectivity in iron ore flotation has not yet been addressed in detail. The structural differences between ethermonoamine and etherdiamine result in distinct adsorption and frothing properties, offering versatility in the concentration of hematite. This study addressed the effect of the etheramine type on flotation selectivity, focused on the analysis of frothing properties. The characterization of the surfactants in the pure state and in two-phase systems (liquid/air) was conducted to investigate the behavior of etheramines concerning foam forming and stabilization. The surface tension measurements showed greater activity of etherdiamine at the liquid–air interface in comparison with ethermonoamine, which was more effective to prevent bubble coalescence and produce smaller bubbles, despite the reagent's higher critical coalescence concentration. Ethermonoamine presented faster foamability for concentrations greater than the observed critical coalescence concentration values as well as greater water recovery in foam, whereas the double CH<sub>2</sub> group and higher molecular weight imparted strong lipophilic character to etherdiamines, producing drier, voluminous, and more stable foams, with longer lifetime. These results are fundamental for the evaluation of three-phase systems, which will be presented in a subsequent paper.

### KEYWORDS

Etheramines; foam formation; foam stability; bubble size

### Introduction

Brazilian iron ore concentrates have a high price in the transoceanic market of direct reduction and blast furnace pellets. Reaching the required impurities grade results in low metallurgical recovery, which is an even greater challenge as the iron ore industry faces a steady decrease of iron content in the run of mine. In the concentration of iron ores, flotation is the most important technique, in which etheramines are widely used as quartz collectors, but also playing a role as frothers. Several studies explain the adsorption of the surfactant on quartz surface, whereas foam/froth properties of the reagent, also important for collision, adhesion, and transport probability in flotation (and consequently selectivity, grade, and recovery), have not been examined in detail.

The last two decades were rich in publications addressing characteristics and fundamental properties of foaming and frothing of different nonionic surfactants, basically alcohol and polyglycol types, in different flotation systems (Cho and Laskowsky 2002; Grau, Laskowski and Heiskanen 2005; Melo and Laskowski 2006; Zhang et al. 2012; Zhu et al. 2019). However, few investigations addressed properties of ionic surfactants, such as fatty acids (Atrafi and Pawlik 2016, 2017), and, according to our knowledge, references on foamability of etheramines are not available.

Amines are weak bases, which dissociate by protonation in aqueous medium under acidic and moderately alkaline pH ranges (Bulatovic, 2007). These surfactants are widely used in plant practice as quartz collectors in reverse flotation to concentrate iron ores (Araujo, Viana and Peres 2005; Filippov, Severov and Filippova 2014; Nakhaei and Irannajad 2018; Zhang et al. 2019). The ionic species adsorb onto the negatively charged quartz surface, whereas the molecular species adsorb at the air/liquid interface acting as a frother.

Fatty amines were used in the past (Uwadiale 1992). Smith and SCOTT (1990) stated that primary fatty amine adsorption on quartz is pH-dependent. In the pH range of full flotation, surface precipitation takes place when the amine solubility limit is exceeded within the quartz solution interface. The application of quaternary ammonium-based ionic liquids was reviewed by Sahoo, Rath and Das (2019). Aliquot 336 yielded 97% quartz recovery and 8% hematite recovery in tests with pure minerals.

Novel collectors, such as bio-collectors and innovative formulations, have been investigated and proposed as alternative reagents (Patra et al. 2018), however etheramines are still the most successful biodegradable collectors used in plant practice in the reverse cationic flotation of iron ores. Papini, Brandao and Peres (2001) concluded that etheramines perform better than fatty amines in the reverse flotation of an itabirite

Brazilian iron ore. They concluded that etheramines perform better than fatty amines, ethermonoamine yielded higher recovery than etherdiamine, and the performances were reverted when the amine is blended with fuel oil. Also, the functional ether group  $O(CH_2)_3$  represents an advantage by improving the solubility of these surfactants (Araujo, Viana and Peres 2005).

Although ethermonoamines are generally used because of its low cost, understanding from use of reagents should consider overcoming challenges as reducing water consumption and tailing generation, starting with foam control. Etherdiamines have distinct effects on the flotation performance as a function of their structural characteristics (Matos et al. 2019). These effects depend on process variables and intrinsic characteristics of ores or minerals to be separated (Matos 2017; Vieira and Peres 2007). Etherdiamine, containing two polar sites, can adsorb onto more surface sites than ethermonoamine, which has similar molar weight, although just this is not sufficient to enhance the floatability. The characteristics of the hydrocarbon chain and their effects on the water/air interface, or froth phase, are as important as the ionic properties.

Vidyadhar, Kumari and Bhagat (2014) reported that mixed cationic/anionic collectors should present good results on flotation. Nakhaei and Irannajad (2018) stated that blending diamines with monoamines is a usual plant practice in a large Brazilian concentrator.

The performance of amines is impaired by the presence of  $Ca^{2+}$  in the flotation pulp. Good results were achieved by Lelis, da Cruz and Fernandes Lima (2019) by complexation of the interfering ions with EDTA.

All chemicals present affect collector adsorption and froth properties. Depressants, widely starches, complete the reagent's system in iron ore flotation (Rath and Sahoo 2020). One of the first studies on the interaction of amine and starch in quartz flotation was conducted by Hendriks and Smith (1972). Complex formation was suggested for the mutual adsorption effects of fatty primary amine and starch.

Pure liquids are not capable to form stable foam due to their high surface tension. The use of heteropolar molecules (surfactants) reduces the surface tension, resulting in the formation of more stable bubbles. The frother also contributes to bubble stabilization by strengthening the interfacial membrane which protects the wall of the bubble against hydrodynamic efforts. The surface tension decreases and foaming increases with the surfactant concentration and chain length, whereas the molecular structure defines the hydrophilic-lipophilic character (HLB) of the surfactant (Davies 1957; Tan, Zhang and Finch 2018).

The ability of a frother to form foam is a two-phase event, whereas the formation of froth during flotation is a three-phase phenomenon (Kowalczyk and Drzymala 2017). The effect of frothers on the foamability and foam stability is monitored by the bubble size, air dispersion, bubble half-life time, water recovery, and many other measurements.

This study compares the differences in performance of one ethermonoamine and one etherdiamine commonly used in iron ore flotation plant practice, comprising surfactants

characterization in the pure state and solutions, and foam properties in two-phase systems. Foam stability, bubble size, kinetics of foam formation, and wettability interfere directly with the selectivity of these surfactants in the reverse flotation of iron ores, which will be presented in Part II of this study.

## Materials and methods

### Materials

The surfactants ethermonoamine PA14F-30 (isodecyloxypropyl amine) and etherdiamine M73 (isotridecyloxypropyl-1,3-diaminopropane), supplied with the highest available purity ( $\geq 99.5$ ) by Air Products (actual Evonik, USA), were used in this study. These cationic surfactants are alkyl ethylene etheramine acetates with branched hydrocarbon chain and neutralization degree of 30%. Ethermonoamine PA14F-30 ( $C_{10}H_{21}$ ) has molecular weight of 225 g/mol whereas etherdiamine M73 ( $C_{13}H_{27}$ ) has molecular weight of 330 g/mol.

In the experiments, the pH was controlled by using caustic soda (NaOH). Deionized water (pH = 6.89; C = 1.23  $\mu$ S and T = 1.42 NTU) was used to prepare all 1% (w/v) reagents solutions and to conduct the two-phase tests.

### Methods

The total amine value (TAV) and the degree of neutralization were evaluated in duplicate, via titration with ethyl alcohol and KOH (potassium hydroxide). The total amine value is the number of milligrams of potassium hydroxide equivalent to the basicity of 1 g of sample.

The surface tension measurements of surfactant solutions were performed in a K10ST tensiometer ( $\pm 0.5$  mN/m) from Krüss, Germany, using the Du Nouy ring method. All tests were conducted at approximately  $21 \pm 2^\circ C$ , with 20 ml samples, measured five times after flame treatment of the platinum plate ring, in order to remove organic contamination.

Bubble size measurements were conducted in an APBS MK4 Anglo Platinum Bubble Sizer apparatus connected to the flotation machine and under similar conditions of foam formation. For each test condition, 25–35 photographs were analyzed using image analysis software provided by Stone Three Mining (South Africa). This software performs the statistical analyses to calculate the mean Sauter diameter ( $d_{32}$ ).

Foam was generated in a 3.9 L cell of a laboratory Wemco flotation machine, using a 3 L pH adjusted aqueous solutions (water and reagents at pH = 10.5), at specific concentration. The solution was stirred at 1250 rpm during 1 min, then the air, at constant flow rate, was introduced and the foam was collected in individual recipients at subsequent 30 s intervals. Each foam aliquot was weighed in analytical balance ( $\pm 0.01$  mg) to generate kinetic curves of cumulative water retained in the floated fraction. Replicates of these tests were conducted to measure foam volume and its gradual extinction at time every 60 seconds, using a 2000 mL graduated glass cylinder ( $\pm 6$  mL) and a chronometer. The difference in half initial foam

**Table 1.** Design of experiments.

Seq Order	Etheramine Type	Concentration (mg/L)
1	Etherdiamine	10
2	Etherdiamine	15
3	Ethermonoamine	10
4	Etherdiamine	10
5	Ethermonoamine	10
6	Ethermonoamine	15
7	Etherdiamine	15
8	Ethermonoamine	15

volume ( $\text{Volume}_{50}$ ) and its time, here defined as  $\text{LF}_{50}$  determine the foam stability parameters.

The tests were designed according to a DOE  $2^2$  (Table 1), in which the defined variables and levels were amine type (ethermonoamine and etherdiamine) and frother concentration (10 and 15 mg/L). These concentrations were chosen because they are greater than CCC values for both etheramines, found from the bubble size measurements. Mass recovery, kinetic constant ( $k$ ), Volume 50, and Lifetime 50 were defined as response variable. The tests followed random sequence and the statistical analysis of data used the software Minitab 18.

## Results and discussion

### Amines characterization

The molecular weight (MW), hydrophilic–lipophilic balance (HLB) and activity at the water–air interface (surface tension) are relevant parameters to characterize the differences between surfactants and their effects on foam/froth formation, stabilization, and drainage during flotation.

**Table 2.** Etheramines properties: Molecular Weight (MW), Total Amine Value (TAV), density, pH and Hydrophilic Lipophilic Balance (HLB).

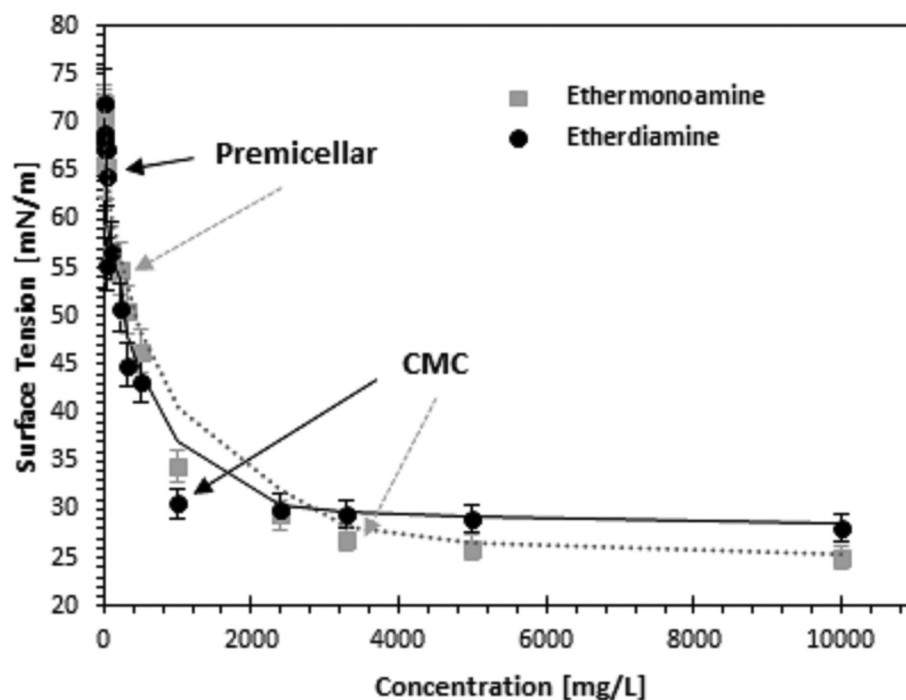
Amine type	MW(g/mol)	TAV	Density (kg/L)	pH	HLB
Ethermonoamine	225	$244 \pm 1$	0.85	10.25	6.87
Etherdiamine	330	$331.0 \pm 0.4$	0.87	9.97	5.45

The structural formula of etheramines allowed the calculation, as proposed by Davies (1957), of the HLB values of ethermonoamine (6.87) and etherdiamine (5.45). In the HLB range from 4 to 10, both reagents are classified as frothers (Drzymala and Kowalczyk 2018). The double  $\text{CH}_2$  group present in etherdiamine is responsible for its stronger lipophilic character (less affinity by water), showing better drainage in foam formation, for consequence, than ethermonoamine. Other physical chemical properties such as TAV (analyzed), density and acidity of etheramines (specified by the supplier) are essential for quantitative performance of frothers. These parameters are presented in Table 2.

Figure 1 shows that both reagents present strong tensoactive action, although etherdiamine is slightly more effective than ethermonoamine regarding the reduction of water surface tension. Surfactants with longer hydrocarbon chain present stronger surface activity than those with lower molecular weight, either in the case of ionic or nonionic molecules, as results found by Atrafi and Pawlik (2016).

As shown in Figure 1, the ethermonoamine and etherdiamine critical micelle concentration (CMC) are 3300 and 1000 mg/L, respectively.

Grau, Laskowski and Heiskanen (2005) stated that static surface tension should not be directly utilized in the analysis of the properties of dynamic systems, such as foams, due to the Marangoni effect (Tan et al. 2005a). However, it is important to characterize a surfactant action regarding foamability (Tan

**Figure 1.** Surface tension isotherms with indication of premicellar and CMC points, for ethermonoamine and etherdiamine at 21°C.

et al. 2005b). Lower surface tension increases the foamability of a solution from the perspective of surface energy (Wang et al. 2015).

## Two phase tests

### Bubble size

The increase in frother concentration reduces the average bubble size by coalescence prevention, up to a particular point, where a plateau is established. Above this point, coalescence is completely prevented. Cho and Laskowsky (2002) defined this point as critical coalescence concentration (CCC) and proposed drawing two asymptotes to define it, as shown in Figure 2(a).

Figure 2(b) presents the Normal Distribution Curves of mean Sauter diameters for etherdiamine and ethermonoamine at concentrations ranging from 10 to 25 mg/L (concentration after CCC). Ethermonoamine produces smaller ( $1.38 \pm 0.03$  mm) bubbles with less variability in size

distribution in comparison with etherdiamine ( $2.02 \pm 0.05$  mm), considering a 95% confidence interval. Finer bubbles increase the collision probability of particle/bubble, favoring mineral floatability. Also, the Figure 2 (b) presents a schematic representation of different etheramines adsorbed in air-water interface forming the bubbles.

### Water content

The measurement of water content in the floated flow allows the determination of the foamability and the foam stability in two-phase flotation tests. The DOE study compared the water mass recovery and its kinetic to evaluate foam formation, foam volume, and its half lifetime to define stability. The statistical analyses of results indicated that both variables (etheramine type and concentration), at tested levels were significant, considering 95% confidence interval.

Figure 3 presents water recovery as a function of etheramine type and concentration. The foam formed by ethermonoamine

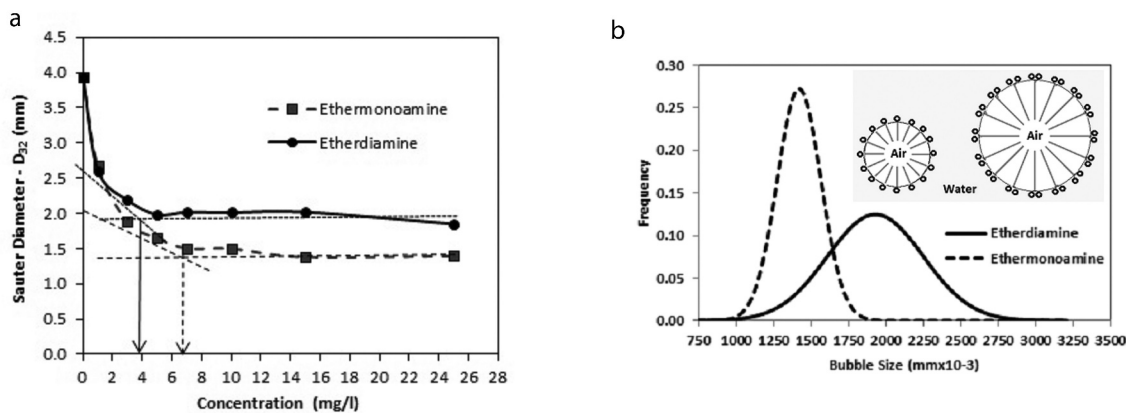


Figure 2. (a). CCC curves for ethermonoamine and etherdiamine at 10–25 mg/L. (b). Normal distribution of bubbles sizes at 10–25 mg/L for ethermonoamine and etherdiamine.

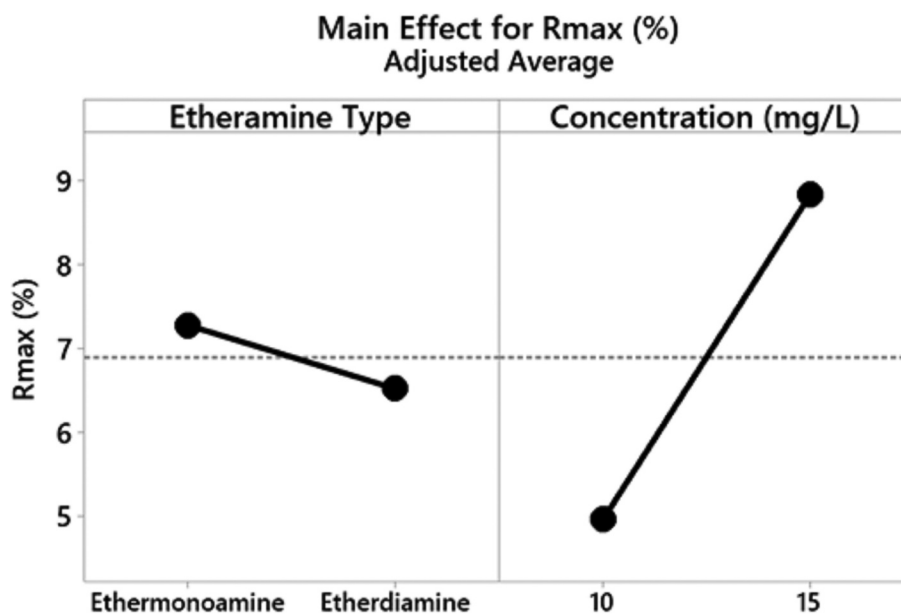


Figure 3. Cumulative water recovery in the foam ( $R_{max}$ ).

induces greater water recovery, which is potentialized by higher concentration. The water content in the floated fraction depends critically on the frother concentration (Malysa 1998), and an increased amount of water associated with the liquid/gas interface stabilizes the foam, which thus becomes more voluminous (Melo and Laskowski 2006).

The wet foam formed by ethermonoamine overdosage promotes hydrodynamic motion, dragging more water to the foam phase during the test whereas, for etherdiamine, the drainage of foam was more intense. This distinct and remarkable characteristic can be due to the hydrophilic–lipophilic balance and interfacial tension differences between each surfactant, previously discussed.

The differences between foam properties originated using ethermonoamine and etherdiamine are qualitatively illustrated in pictures in Figure 4. The assemblies of spherical bubbles, separated by thick liquid wall in ethermonoamine foam (Figure 4 right) are characteristic of wet and unstable foams (Leja 1982; Melo and Laskowski 2006). At picture, is possible see that this foam is less viscous and voluminous, and has smaller macro bubbles. In other hand, foams produced by etherdiamine (Figure 4 left) can be classified as metastable or dry, exemplified by bubbles with polyhedral shape and only slightly curved liquid film.

Atrafi and Pawlik (2017) reached similar results evaluating fatty acid solutions, quantified by TOC (total organic carbon). These authors observed that shorter chain surfactants (hexanoate and laurate) produced wet foams with very low concentration of surfactant accumulated in the foam phase, in contrast with sodium oleate, which produced stable dry foams, attributed to greater adsorption at the gas-liquid interface.

Neethling, Lee and Cilliers (2003) developed a fundamental model to predict water recovery from unstable ( $\alpha < 0.5$ ) and stable ( $\alpha \geq 0.5$ ) foams, in which  $\alpha$  represents the recovery of air by uninterrupted bubbles. The equations depend on rheological parameters such as kinetic viscosity, liquid density, gravitational constant, in addition to the bubble diameter, which square is inversely proportional to the water recovery. The evaluation of the equations confirms the results found, since the greater recovery of water from the unstable foam of ethermonoamine presented smaller bubbles and requires an additional multiplier parameter as a function of  $\alpha$ .

### Kinetics of foamability

Most flotation-related processes, including foam formation, follow the so-called first order kinetic equation:

$$R = R_{max}[1 - \exp(-kt)] \quad (1)$$

where,  $R$  is the cumulative mass recovery in the froth overflow (%),  $R_{max}$  is the ultimate cumulative flotation recovery (%),  $k$  is the first order kinetic constant ( $s^{-1}$ ) and  $t$  is the flotation time (s). Applying the method of local efficiency (Drzymala, Ratajczak and Kowalczyk 2017; Ratajczak, Drzymala and Kowalczyk 2016) at  $t = 0$  and applying the derivative (eq. 2), we can obtain the kinetic constant values shown in Figure 5:

$$\left. \frac{dR}{dt} \right|_{t=0} = R_{max} \cdot k \quad (2)$$

Figure 5 shows that ethermonoamine presented faster kinetics in comparison with etherdiamine, and that greater constants are observed at higher amine concentration.

### Foam stability

The foamability is a dynamic process and part of the water that stabilizes the foam suffers drainage. The action of specific surfactants could either hold the bubbles structure or later, collapse the foam, which determines the life time or foam stability.

Figures 6 and 7 show the difference in half initial foam volume ( $\text{Volume}_{50}$ ) and its time, here defined as  $LF_{50}$ , between ethermonoamine and etherdiamine and the considered concentrations. This concept can be used for any foam volume reduction. Etherdiamine forms greater volume of foam with longer lifetime.

According to Tan et al. (2005b), the foamability increases with the frother concentration, then reaches a maximum plateau region, and decreases above this well-defined concentration range. It explains why the higher tested concentration reached higher volume with lower lifetime. The foamability and water content are also function of the gas flow rate, which was held constant in this study.

As expected, the faster and wetter foam from ethermonoamine have shorter lifetime. The foam produced by ethermonoamine extinguishes completely at 180 s, whereas remaining foam is present after 600 s with etherdiamine as shown in Figure 8. The higher hydrophilic character of

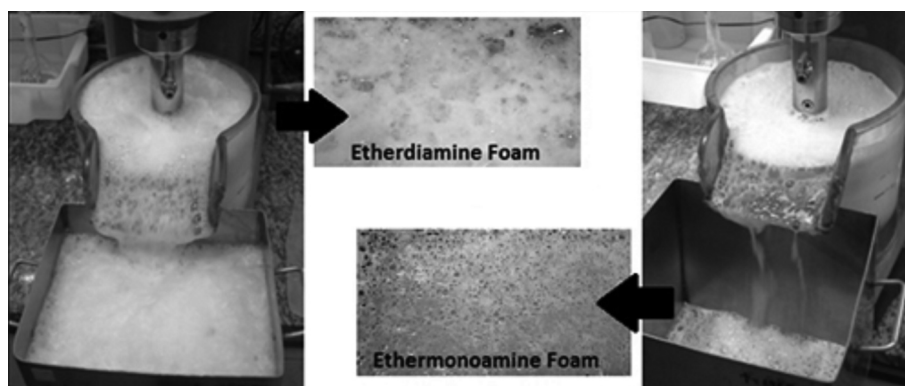


Figure 4. Example of stable and dry foam formed by etherdiamine and wet and unstable formed by ethermonoamine (15 mg/L).

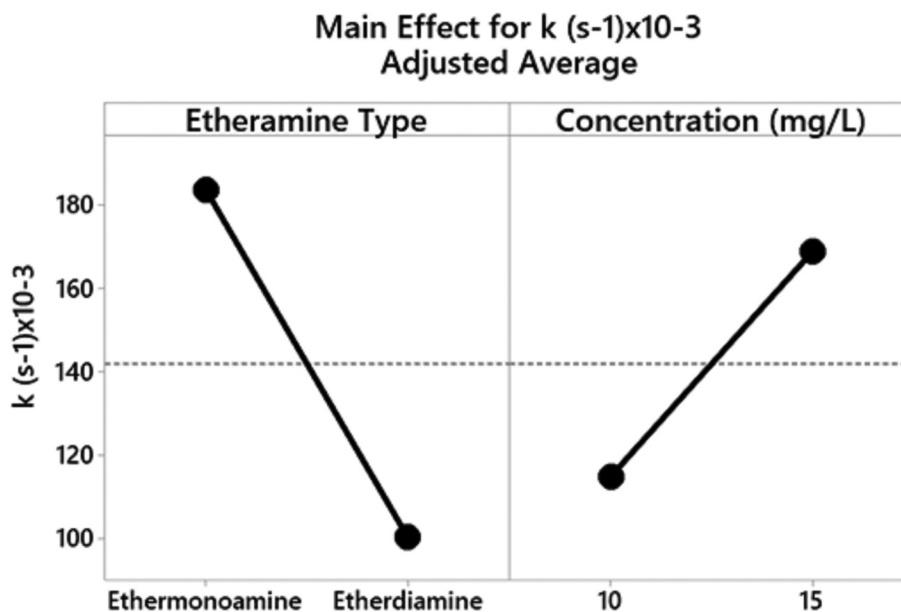


Figure 5. Kinetics of foamability for etheramine type and concentration.

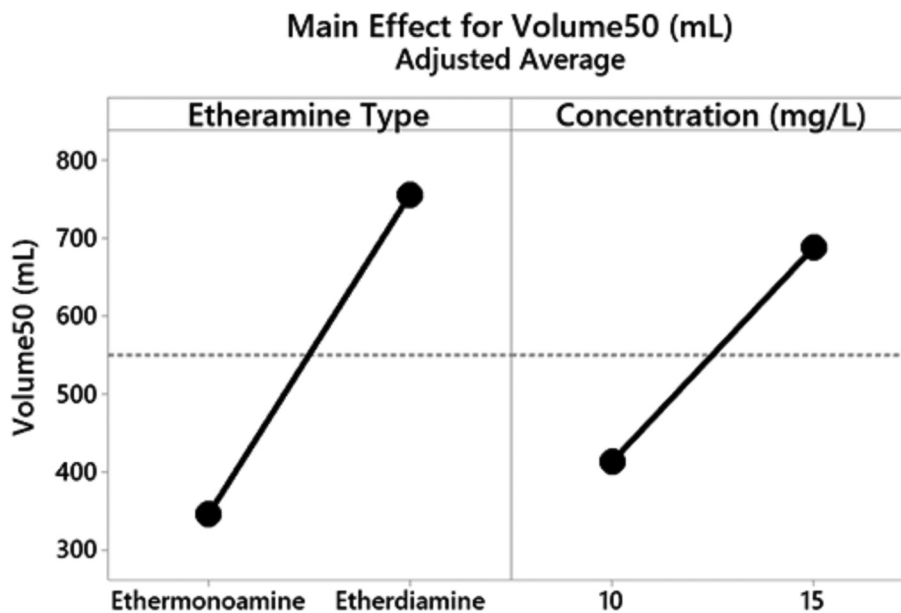


Figure 6. Half foam volume (Volume<sub>50</sub>).

ethermonoamine promotes premature collapse of bubbles, reducing the stability of foam in opposition to the etherdiamine behavior, which promotes greater elasticity in its bubbles lamellar layers and Plateau Borders generated.

Finally, the ability of etherdiamines to form bubbles with thinner, well-drained, and better elasticity walls, makes them more resilient to the three stages of particle collection (collision, adhesion, and transport) inherent to the flotation process, compared to smaller bubbles formed by ethermonoamine.

## Conclusion

The results in this study indicate that the different structural characteristics of ethermonoamine and etherdiamine define their effect on foamability and foam stability. Etherdiamine presents stronger lipophilic character and higher molecular weight than ethermonoamine, whereas the latter produces smaller bubbles at faster formation rates and presents higher critical coalescence concentration than etherdiamine. The higher molecular weight, lipophilic character, and surficial activity of etherdiamine generates, at greater intensity and

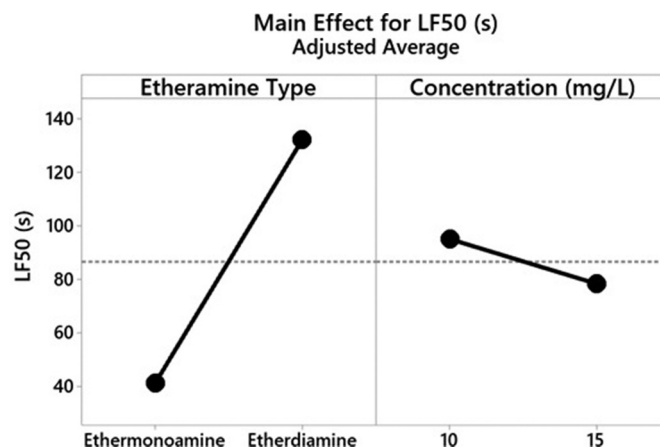


Figure 7. Lifetime of foam volume (LF<sub>50</sub>).

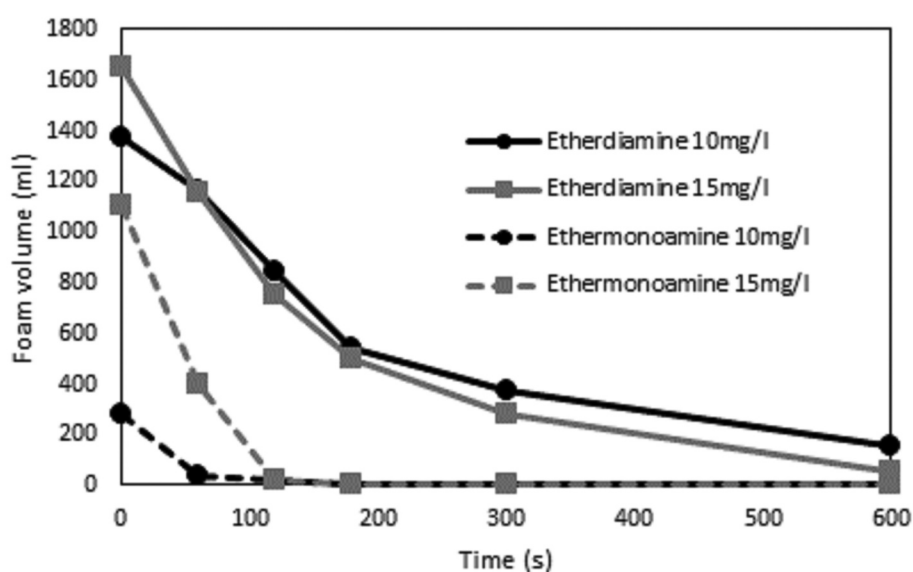


Figure 8. Foam stability for ethermonoamine and etherdiamine.

slower kinetics, more stable, drier, and persistent foams compared to ethermonoamine. In the next step of this study (Part II), these remarkable differences in the stability of foams produced by ethermonoamine and etherdiamine will be evaluated in three-phase tests to clarify their impact on the selectivity in the reverse cationic flotation of iron ore.

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## Disclosure statement

No potential conflict of interest was reported by the author(s).

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