

# THE EFFECT OF ETHERAMINE TYPE ON THE HYDROPHOBICITY OF QUARTZ PARTICLES FROM IRON ORE

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## ABSTRACT

The cationic reverse flotation route is one of the main processes to remove silicates from iron ore. Hence, the etheramines are the most common surfactants used to modify the hydrophobicity of mineral surfaces. Moreover, the etheramines differentiate between ethermonoamines and etherdiamines, which have other peculiarities such as raw material sources, carbon chain length and type, and neutralization degree. The performance of three types of etheramines in the flotation of quartz was investigated in order to understand the influence of the chemical structure of these collectors on the floatability of silicate minerals. These studied etheramines were: MCM (medium chain ethermonoamine), MCD (medium chain etherdiamine) and LCD (long chain etherdiamine). Furthermore, their pKa values were measured to evaluate the influence of etheramine species distribution on the silicate flotation. The quartz flotation was performed at different pH conditions, collector concentration and type, in the presence and absence of an iron mineral depressant (starch).

Key words: etheramine, quartz, iron ore, flotation, pH, concentration

## 1. Introduction

Iron ore concentrators mainly process iron oxide and hydroxide minerals, such as magnetite ( $\text{Fe}_3\text{O}_4$ ), hematite ( $\text{Fe}_2\text{O}_3$ ), goethite ( $\text{FeO}(\text{OH})$ ) and limonite ( $\text{FeO}(\text{OH}) \cdot n\text{H}_2\text{O}$ ), however iron carbonates and sulphides can also be used as iron source for the industry (Filippov, 2014; Mhonde, 2016; Araujo et al., 2005). Once silicates are the main gangue minerals in iron ore deposits, the concentrators predominantly use flotation to concentrate iron ores in the fine size range ( $<149 \mu\text{m}$ ). Moreover, the technical literature described three main routes for iron ore concentration through flotation: a) reverse cationic flotation of silicates; b) direct anionic flotation of iron oxides; c) reverse anionic flotation of activated silicates (Araujo et al., 2005, Turrer and Peres, 2010, Lima et al., 2013).

Based on the mineralogy of deposits and operational characteristics of the concentrators, it is possible to define the reagents selections for reverse iron ore flotation. For example, etheramines ( $\text{R-O}-(\text{CH}_2)_3-\text{NH}_2$ ) have been widely used to remove silicate gangues from iron ores. In addition, processing of some types of iron ore can demand depressant reagents, among which the starch is the most commonly used to depress hematite. Starch consists of two fractions, a linear polymer “amylose” and a branched polymer “amylopectin” (Liu et al., 2000; Araujo et al., 2005; Lima et al., 2013; Mhonde et al., 2017; Peçanha et al., 2019). Furthermore, other amines have been also employed in reverse cationic flotation, such as fatty amines, quaternary ammonium salts and imidazoline, but they usually require

higher dosages than etheramines (Araujo et al., 2005; Filippov et al., 2010; Martins, 2012; Mhonde, 2017). Several mechanisms have been proposed for the surface charge generation of various systems. For oxide minerals such as hematite, quartz and alumina, the origin of the electrical charge at the oxide surface / aqueous phase can be described as protonation/deprotonation of the surface hydroxyls (Somasundaran, 1974; Fuerstenau, 2005; Laskowski, 1999; Crundwell, 2016; Huang et al., 2014; Filippov, 2012; Orhan and Bayraktar, 2006):



The mechanism of flotation of silicate and iron minerals is dependent on the electrical properties and the solubility of the mineral, the charge and chain length of the collector, and stability of the salt metal-collector. In addition, the depressant and dispersant adsorptions are also related to mineral surface characteristics, such as the chemical composition, the electrical charge distribution and solubility, the potential determining ions content in slurry, and the chemical and structural composition of modifier agent (Scott and Smith, 1991; Montes-Sotomayor et al., 1997; Pavlovic and Brandão, 2003; Fuerstenau, 2005; Laskowski, 1989 and 1999; Araujo et al., 2005; Filippov et al., 2010; Crundwell, 2016; Horta et al., 2016).

The objective of the present investigation was to evaluate the effect of the chemical structures of etheramines on the flotation performance of quartz in the presence and absence of starch, which was used as the depressant.

## 2. Experimental

### 2.1. Materials

The sample of crystalline quartz used in the study was purified from an iron ore sample, which was collected in the Brazilian Iron Quadrangle. The chemical assay of the quartz sample showed that it contains 98.95 % SiO<sub>2</sub>, 0.71 % Fe, 0.005 % P and 0.03 % loss on ignition (LOI) and the quantitative X-ray diffraction analysis identified that the sample contains 99.0% quartz and 1.0% hematite.

After crushing, grinding and wet screening, quartz and hematite minerals were purified by magnetic separation (8,000 Gauss and 21,000 Gauss). Quartz samples were wet-sieved to the size fraction of – 75+150 µm, which was used for microflotation in modified Hallimond tube.

### 2.2. Zeta potential

The zeta potential was measured using the Zeta-Meter 4.0 (Zeta Meter System). Quartz suspensions of 0.5 g/L (100% < 20 µm) were prepared in 0.001 mol/L of KNO<sub>3</sub> electrolyte solutions, conditioned for 1 h at room temperature (22 °C) in the presence of predetermined concentration of reagents and pH. The pH of the suspension at the time of measurement was reported in the results.

### 2.3. Acid dissociation constant (pKa)

The dissociation constants of these etheramines were determined using the titration technique described by Mhonde (2016) and Fernandes (2017). The concentrations of etheramine species in solution change with variation in pH according to the reactions 3 - 5 (Laskowski, 1989; Matulis and Bloomfield, 2001; Ari, 2001).

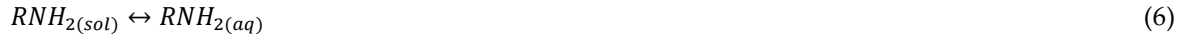


$$K_{acid} = \frac{[RNH_{2(aq)}].[H^+]}{[RNH_3^+_{(aq)}]} \quad (5)$$

pKa measurements were carried out in order to obtain the pH at which the amine collectors would be 50% dissociated.

The measurements were conducted in 808 Titrand (Metrohm). A solution of 0.05 mol/L of HCl was gradually added to 25 mL of a 1 % amine collector solution until the solution's pH was at pH 1.5. At this point, 0.05 mol/L of NaOH solution was titrated into the collector solution in aliquots of about 0.1 mL noting the new pH after the addition of each aliquot of NaOH. When the pH remained constant, 1 mL aliquots of the 0.05 mol/L NaOH solution were added, once again checking the pH of the amine solution. As the pH began to rise, aliquots of 0.5 mL were once again added up until a pH value of 12 was reached.

Moreover, in saturated systems (Matulis and Bloomfield, 2001; Laskowski, 1989):



The concentration of soluble amine can be written as (Matulis and Bloomfield, 2001):

$$RNH_{2(aq)} = fS = S \frac{[RNH_{2(sol)}]}{C} \quad (7)$$

where S is the limiting solubility of RNH<sub>2</sub>, f is the fraction of RNH<sub>2</sub> in precipitated form, and C is the total concentration of alkylamine in all forms (Laskowski, 1989; Matulis and Bloomfield, 2001; Ari, 2001):

$$C = [RNH_3^+_{(aq)}] + [RNH_{2(aq)}] + [RNH_{2(sol)}] \quad (8)$$

## 2.4. Reagents

Three different etheramines with 30% neutralization degree from Clariant were employed in the microflotation tests, in the absence and presence of starch, which was composed of approximately 73% amylopectin and 27% amylose. The reagents suite for microflotation tests is shown in Table 1.

**Table 1. The reagents suite used in the experimental work**

Reagent name	Chemical group	Reagent type
Flotigam™ EDA-3	Alky ethermonoamine – Medium chain length (MCM)	Collector
Flotigam™ 3135-3	Alky etherdiamine – Medium chain length (MCD)	Collector
Flotigam™ 2835-3	Alky etherdiamine – Long chain length (LCD)	Collector
Amidex 3001	Starch	Depressant
HCl (10%)	Inorganic acid	pH modifier
NaOH (10%)	Inorganic base	pH modifier

## 2.5. Microflotation procedures

The objective of these tests was to evaluate the flotation performance of pure quartz, without a possible influence of iron oxides (hematite and magnetite). Quartz microflotation tests were carried out in duplicate using the modified Hallimond tube of 280 mL, as described by Fernandes (2017). A magnetic stirrer was used to promote the complete mixture between reagents and mineral suspensions.

Quartz sample (1 g) was conditioned in 50 mL collector solution of pre-adjusted pH and concentration for 1 min. The volume of the tube was completed to 280 mL with a pH-adjusted solution for microflotation and after 1 minute of collector conditioning time, air was introduced in the system at a rate of 40 mL/min during 1 minute of flotation time. After each microflotation test, the sunk and floated products were filtered, dried and weighed. The flotation response (recovery) was calculated in percent basis by dividing the mass of the floated product by the mass of the feed.

For microflotation tests with the presence of depressant, 1 g of mineral sample was also conditioned in 25 mL depressant solution of pre-adjusted pH and concentration for 5 min, then 50 mL of collector solution, which also had its pH and concentration pre-adjusted. The volume was also completed to 280 mL and with 1 min of conditioning quartz samples were floated for 1 min at aeration rate of 40 mL/min.

### 3. Results and discussion

#### 3.1. Quartz sample characterization

In order to determine the sign and magnitude of the surface charge on the quartz particles, zeta potential tests were performed at different pH. The results are shown in Figure 1. The isoelectric point (IEP) of quartz in the absence of flotation reagents is below pH 2.0, which indicates that the mineral samples are negatively charged at all flotation pH.

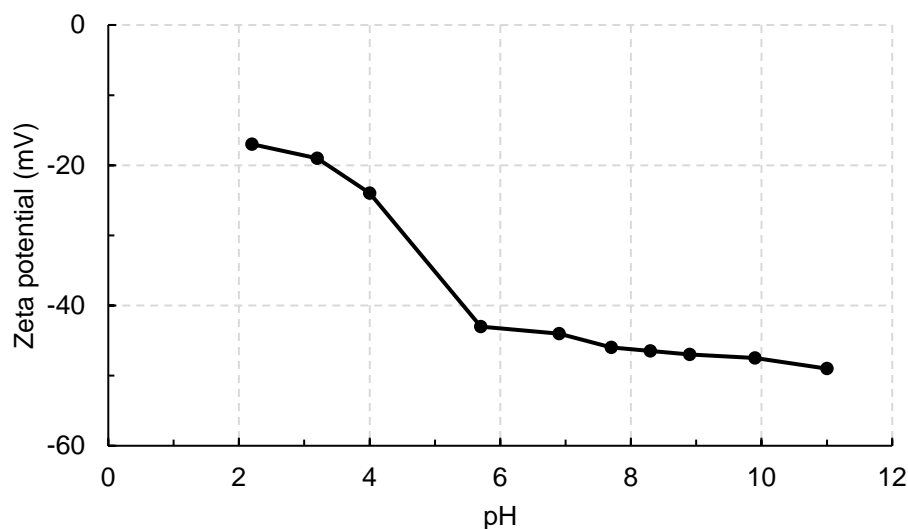


Figure 1. Zeta potential curve of the quartz as a function of pH at  $1.10^{-3}$  mol/L concentration of indifferent electrolytes ( $\text{KNO}_3$ ).

#### 3.2. Acid dissociation constant (pKa)

The dissociation constant (pKa) indicates the basicity of molecules. Table 2 shows the pKa and solubility data for all etheramines studied.

**Table 2. Experimental pKa values and solubilities of etheramines**

Collector	Etheramine type	pKa <sub>1</sub> at 25 °C	pKa <sub>2</sub> at 25 °C	Solubility (mol/L) at 25 °C
Flotigam™ EDA-3	Ethermonoamine of medium chain length (MCM)	9.0	-	7.7x10 <sup>-4</sup>
Flotigam™ 3135-3	Etherdiamine of medium chain length (MCD)	8.9	4.8	3.1x10 <sup>-4</sup>
Flotigam™ 2835-3	Etherdiamine of long chain length (LCD)	8.2	4.7	8.4x10 <sup>-6</sup>

The protonation reactions of amines in solution are influenced by the inductive effect of the carbon chain length and the hetero atom presence. In addition, the molecule basicity is also affected by aggregation, i.e. the solubility (Dantz et al. 1997; Matulis, 2001; Evjen et al. 2017). Consequently, the ethermonoamine Flotigam™ EDA-3 has higher pKa value than the etherdiamine Flotigam™ 3135-3 because it does not have a nitrogen atom as a hetero atom in its structure.

With regard to the etherdiamine Flotigam™ 2835-3, its lower pKa value is due to the effect of aggregation and solubility since it has a longer carbon chain. Therefore, the order of the basicity of these collectors is ethermonoamine of medium chain length (MCM) > etherdiamine of medium chain length (MCD) > etherdiamine of long chain length (LCD).

The species distribution diagrams of etheramines of medium chain length (MCM and MCD) as a function of pH at the bulk total concentrations of 3.10<sup>-3</sup> mol/L are shown in Figures 2 and 3. Furthermore, the variation of species concentrations of etherdiamine of long chain length (LCD) as a function of pH at the total concentration of 1.10<sup>-4</sup> mol/L is shown in Figure 4,

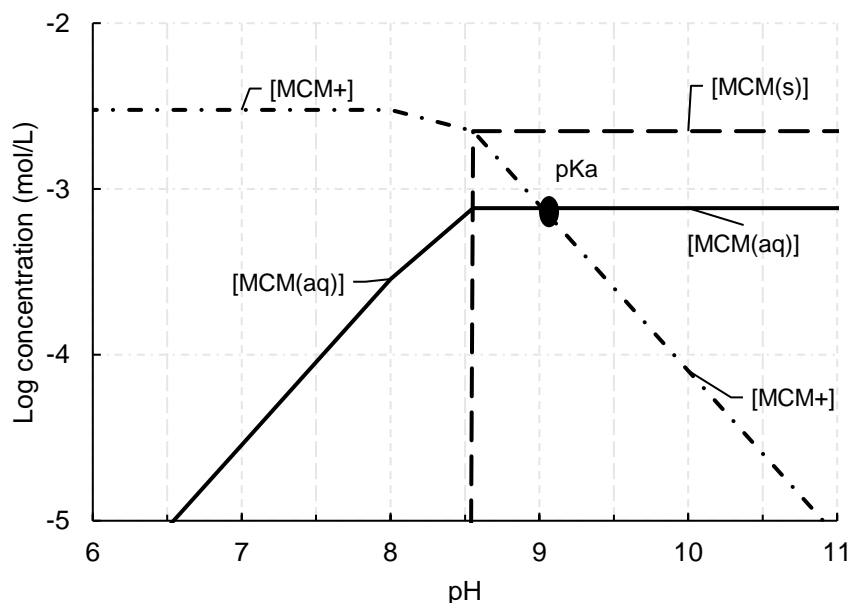


Figure 2. Log concentration diagram for 3.10<sup>-3</sup> mol/L total concentration of ethermonoamine of medium chain length (MCM).

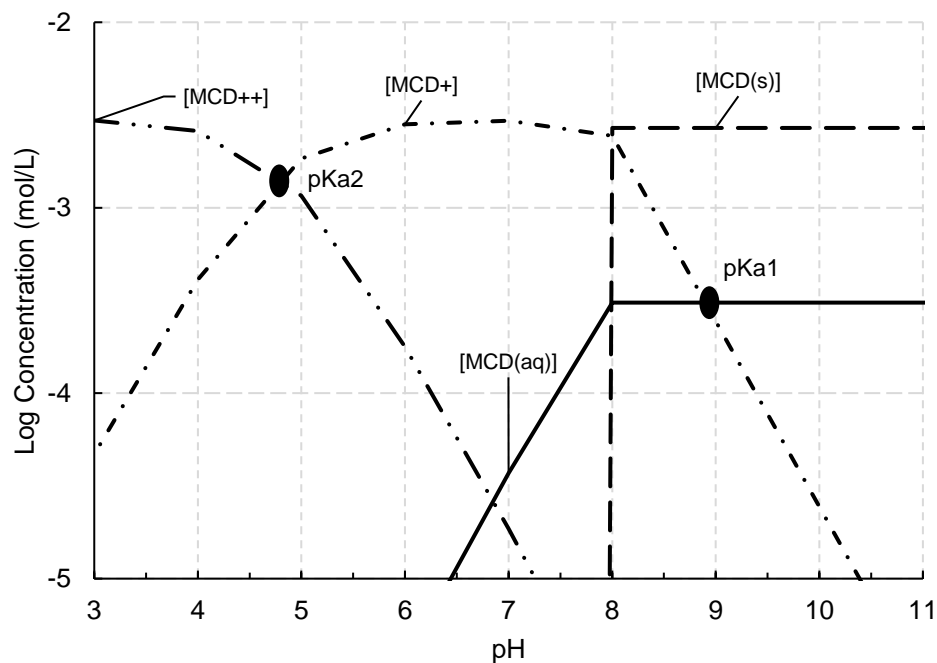


Figure 3. Log concentration diagram for  $3.10^{-3}$  mol/L total etherdiamine of medium chain length (MCD).

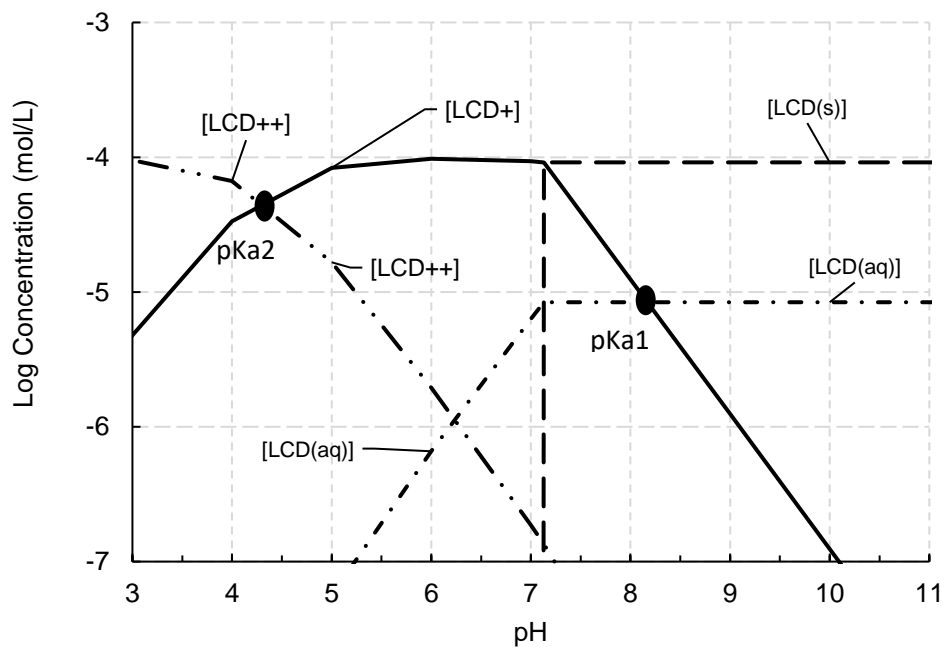


Figure 4. Log concentration diagram for  $1.10^{-4}$  mol/L total etherdiamine of long chain length (LCD)

### 3.3. Microflotation without depressant

The quartz floatabilities as a function of different etheramine chains at various pH values and concentrations are shown in Figures 5 and 6. These results of the microflotation tests with the

different ether amines in the absence of depressant indicated that the maximum floatability of quartz occurred at pH very close to the pKa value of each collector, as predicted in the literature (Gaudin, 1957; Ari, 2001; Mhonde, 2016). Moreover, it was possible to observe that the etheramines concentration increase has improved the quartz floatability, however, ethermonoamine showed a low performance at a 1 ppm collector, probably due to the low concentration of the aqueous species.

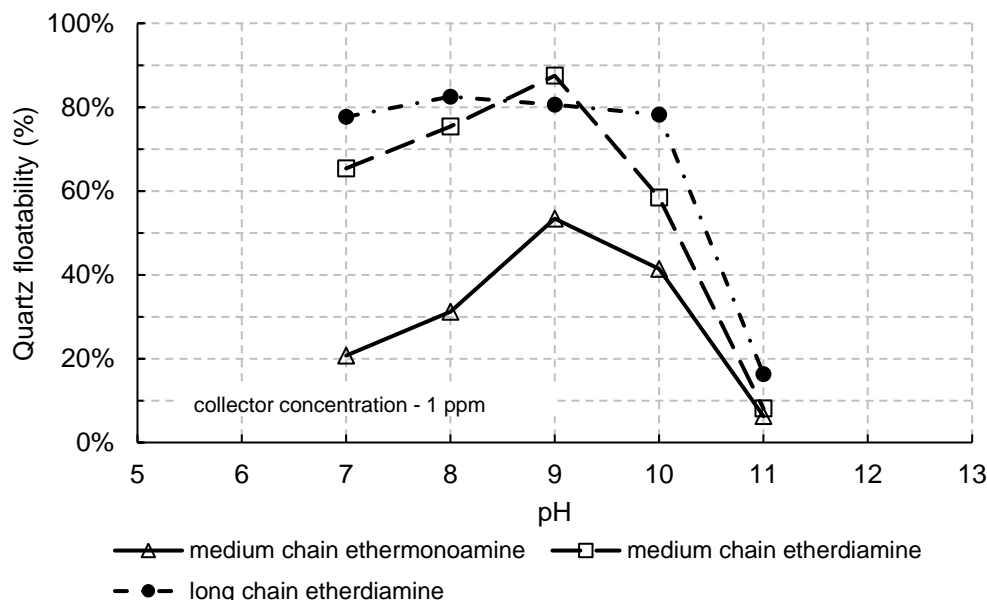


Figure 5. Flotation of quartz as a function of pH at a collector concentration of 1 ppm.

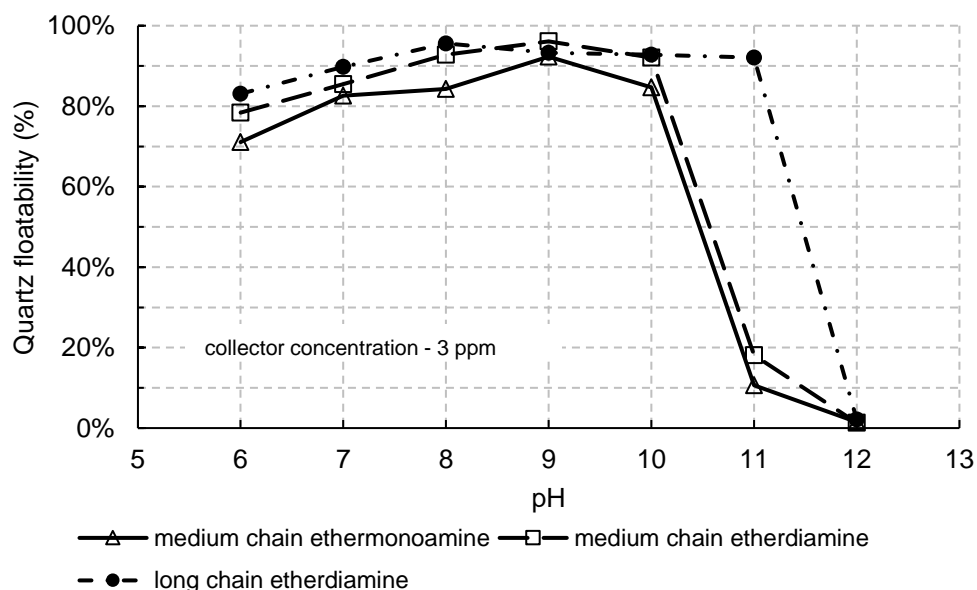


Figure 6. Flotation of quartz as a function of pH at a collector concentration of 3 ppm.

In addition, it was observed that flotation declines sharply at pH higher than 11 likely because etheramine precipitates developed a negative electrical charge and the low availability of cationic etheramine species, which are responsible for non-specific adsorption on negatively-charged surface of quartz. On the other hand, the flotation response over the pH range from 8 to 10 reached the biggest values due to the high surface activity of etheramine aqueous solution, such as ionic and molecular

species. Moreover, the quartz flotation with etherdiamines is enhanced by the presence of their precipitated species, which were co-adsorbed on quartz surface.

The flotation response of quartz particles at two different pH values of 8 and 9 as a function of collector concentration are depicted in Figure 7. The highest quartz recoveries are obtained from etherdiamines, since the flotation was carried out at pH near their pKa, evidencing the strong adsorption of ionic and molecular species, mainly close to the pH of precipitation ( $pH_{sol}$ ).

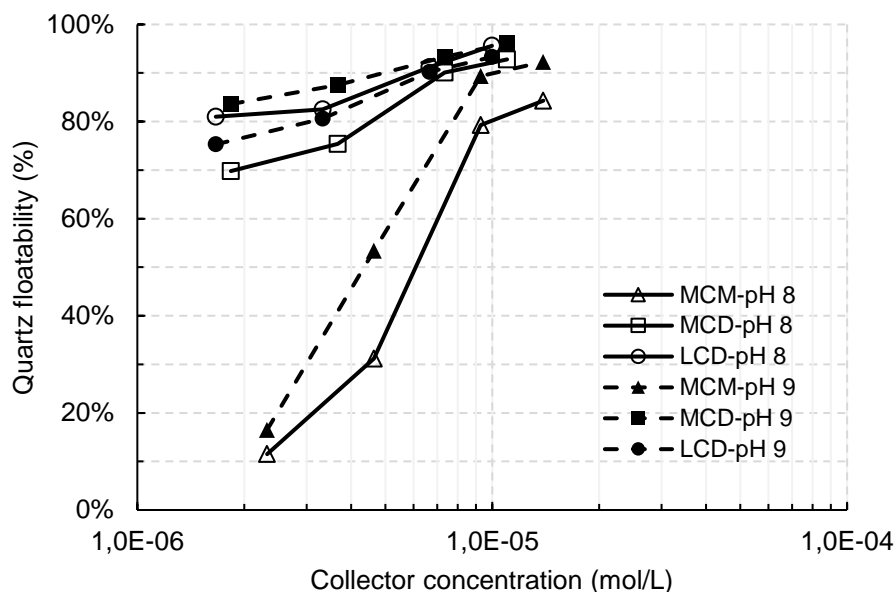


Figure 7. Quartz flotation results as a function of collector concentration at pH 8 and 9. Ethermonoamine of medium chain length (MCM), etherdiamine of medium chain length (MCD) and etherdiamine of long chain length (LCD).

With respect to the quartz flotation response with ethermonoamine as collector, it was possible to observe the strong influence of the concentration on the floatability due to the low presence of precipitated species ( $MCM_{(s)}$ ) below  $pH_{sol}$  8.6. Therefore, the quartz floatabilities at pH 8 and 9 increase sharply as result of a rise in the collector concentration, which indicates that the quartz flotation required higher collector dosages towards the ethermonoamine solubility. Moreover, it can be highlighted that the pH increase significantly improved the flotation performance because of the concentration increase of molecular ethermonoamine species ( $MCM_{(aq)}$  and  $MCM_{(s)}$ ).

### 3.4. Microflotation in the presence of depressant

The flotations responses of the quartz as a function of pH with the addition of 5 ppm starch are presented in Figure 8. It was pointed out that the depressant was able to displace the maximum floatability of quartz to pH values greater than the pKa of each etheramine. Furthermore, the sharp drop in the quartz floatability can probably be attributed to the starch-etheramine interactions and the flocculation of quartz particles by the starch presence.



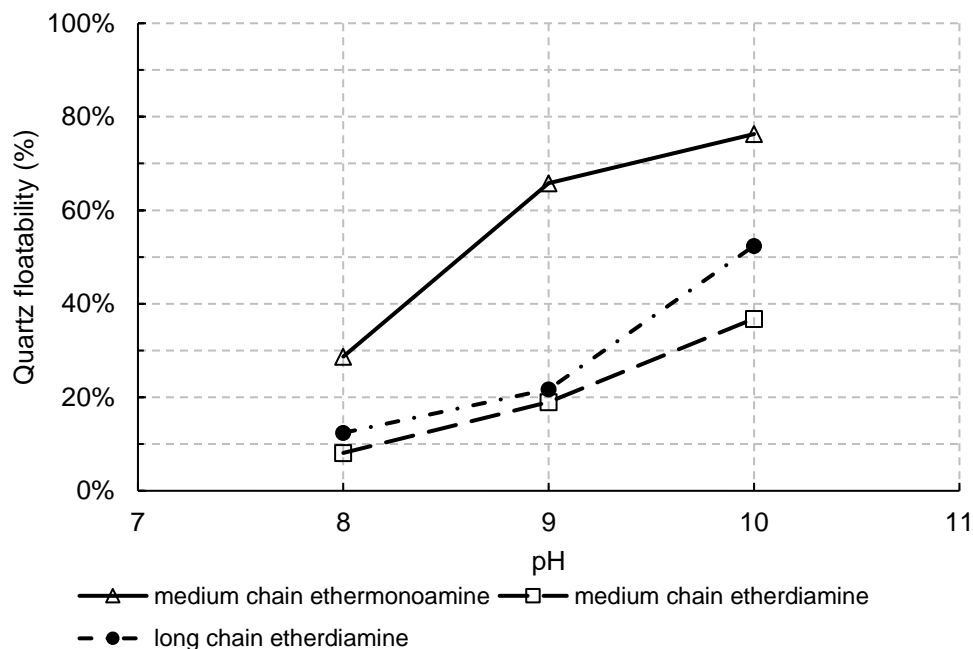


Figure 8. Quartz floatability as a function of pH at starch concentration of 5 ppm and  $2.10^{-5}$  mol/L etheramine concentration.

In fact, several researchers indicate that starch can anchor onto the surface and hence flocculate the quartz particles, which decreases the collector adsorption on the mineral surface (Montes-Sotomayor et al., 1997; Pavlovic and Brandão, 2003, Filippov et al., 2010). Nevertheless, the pH rise ( $> \text{OH}^-$  concentration) reduces this flocculation of the particles because of the repulsion increase between negatively charged starch and negatively charged quartz surface. Thus, there is a greater adsorption of etheramine molecules, which enhances the quartz floatability.

Despite the depressant effect of starch on the quartz flotation response, the ethermonoamine (MCM) attained a recovery of 76.3% at pH 10, which can be explained by the higher availability of molecular ethermonoamine species. On the other hand, the flotation response with etherdiamines were more affected by the starch presence, probably because the amine additional group may increase the interaction of this collector with starch molecules.

These flotation results with etheramines are confirmed by industrial practice with some exceptions. The typical conditions of reverse flotation of magnetite and hematite are presented in table 3 (Araujo et al., 2005; Lima et al., 2013; Carmona, 2013; Cruz and Lima, 2015; Fernandes, 2017; Xiaolong et al., 2019).

**Table 3. Typical conditions of magnetite and hematite reverse flotation**

Valuable mineral	Collector	Depressant	Flotation pH	Example of application
Magnetite	Etherdiamine of long chain length	Not required	8 – 9.5	Magnetite reverser flotation circuits in Chile, USA and Ukraine
Hematite	Ethermonoamine of medium chain length	Starch	10 – 10.5	Hematite reverse flotation circuits in Brazil

#### 4. Conclusions

The species distribution diagram of medium chain ethermonoamine shows that the cationic amine species ( $\text{MCM}^+$ ) is predominant below pH 8.6 and above which the precipitation takes place in aqueous solutions. While the ionic etherdiamine species predominate below pH 7.1 and 8.0 respectively for long chain etherdiamine ( $\text{LCD}^{++}$  and  $\text{LCD}^+$ ) and medium chain etherdiamine ( $\text{MCD}^{++}$ ,  $\text{MCD}^+$ ), thereafter, molecular etherdiamine species prevail. Thus, according to the species distribution diagrams, the order of the pKa values of the etheramines is medium chain ethermonoamine ( $\text{MCM}$ ) > medium chain etherdiamine ( $\text{MCD}$ ) > long chain etherdiamine ( $\text{LCD}$ ), which can be attributed to the hetero atom presence and the inductive effect of the increase of carbon chain length.

Regarding the results of flotation tests without depressant, it is possible to observe that the highest quartz floatabilities were obtained at  $\text{pH}_{\text{flotation}}$  close to the pKa values of each etheramines, in which there is a suitable balance between ionic and molecular species. Moreover, there was a sharp drop in the quartz floatability at  $\text{pH}_{\text{flotation}}$  higher than 11 for all etheramines at concentration of 1 and 3 ppm, except for higher concentration (3 ppm) of long chain etherdiamine, in which the sharp decline of the flotation occurred at pH higher than 12. Therefore, this flotation drop at higher pH occurs not only due the low concentration of ionic etheramine species, but also because the etheramine precipitate can acquire a negative electrical charge, which is not able to coadsorb on quartz surface.

Since the etheramine solubility limit was not exceeded, the increase in collector concentration improved the quartz floatability probably due to the suitable availability of ionic and molecular species, which are responsible for the collector adsorption on mineral surface. Furthermore, the flotation results without depressant confirmed that the highest quartz recoveries are achieved when  $\text{pH}_{\text{flotation}}$  approaches the pKa values of etheramines.

In addition, the presence of starch caused a significant decrease in the quartz floatability due to its adsorption on this mineral surface through hydrogen bonding. Nevertheless, medium chain ethermonoamine attained 76.3% quartz recovery, which is much higher in comparison with the etherdiamine performances at starch concentration of 5 ppm, which can indicate a good selectivity for the industrial reverse flotation of iron ore.

#### Acknowledgements

The authors acknowledge the financial support of Clariant S.A.

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