Influence of the pH regulator on the dolomite hydrophobization process

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Abstract

A study was carried out to investigate the combined effect of released ions in the pulp by pH regulator and mineral surface on dolomite flotation. The tests were carried out using a dolomite sample in a bench flotation cell with automatic froth remover. Zeta potential measurements, solution conductivity and FTIR analysis were done to support the interpretation of the results. The main reagents used for pH adjustment in the alkaline range (calcium hydroxide, sodium hydroxide, and sodium carbonate) were compared using amine or sodium oleate as collector. Dolomite is a sparingly soluble-type calcium mineral (as calcite, apatite, fluorite and scheelite), releasing different amounts of calcium and magnesium ions in the pulp. Amine adsorbs mainly by a chemical complexation mechanism between the molecular specie RNH_2 with calcium and magnesium ions present on the dolomite surface. In this system calcium hydroxide must be used because in addition to adjusting the pH it is a calcium ion supplier, thus increasing the collector adsorption. In turn, in the dolomite flotation with oleate, the pH must be regulated with soda ash that precipitates Ca^{2+} and Mg^{2+} as carbonate allowing for higher adsorption of the collector.

Keywords: pH regulators; dolomite flotation; sparingly soluble minerals; calcium-bearing mineral flotation; dissolved ions.

1. Introduction

The pH of the pulp affects the surface electric charge, the ionization of the reagents, and the dispersion levels of the particles; hence it is a very important parameter in the flotation. In most flotation processes, pH adjustment is necessary to get the best separation conditions. The pH regulators are the most consumed reagent in industrial flotation plants (Fuerstenau and Urbina, 1987; Prasad, 1992). Nevertheless, there are few published studies concerning the influence of the type of the pH regulator on mineral flotation and sometimes the cost of reagent is the only consideration for its choice.

The dolomite and other calcium-bearing minerals (as calcite, apatite, fluorite and scheelite) flotation can be performed with amine or sodium oleate as the collector (Kupka and Rudolph, 2018; Filippova *et al.*, 2018; Filippov *et al.*, 2019). The cationic collectors are adsorbed by electrostatic interactions, hence, above the isoelectric point of the mineral surface and are generally used in alkaline media. In oleate solutions the concentration of active specie $RCOO^-$ increases as the pH increases (Pugh and Stenius, 1985). So, the flotation usually is carried out at alkaline medium too. The main pH regulators used in alkaline flotation are calcium hydroxide, sodium hydroxide and sodium carbonate (Table 1).

Table 1 - Hydrolysis reactions of the main regulators used in flotation.

Regulator	Chemical Formula	Hydrolysis Reaction
Calcium hydroxide	$Ca(OH)_2$	$Ca(OH)_{2(s)} \rightleftharpoons Ca^{2+}_{(aq)} + 2(OH)^-_{(aq)}$
Sodium hydroxide	$NaOH$	$NaOH_{(aq)} \rightleftharpoons Na^+_{(aq)} + OH^-_{(aq)}$
Sodium carbonate	Na_2CO_3	$Na_2CO_{3(s)} \rightleftharpoons 2\,Na^+_{(aq)} + CO_3^{2-}{}_{(aq)}$
		$CO_3^{2-}{}_{(aq)} + H_2O_{(l)} \rightleftharpoons HCO_3^-{}_{(aq)} + OH^-_{(aq)}$

While the Na^+ ion in moderate concentration usually has poor influence on flotation, the Ca^{2+} and CO_3^{2-} participate actively in the reaction, modifying the conditions for adsorption of the collector onto the mineral surface. In the cationic flotation with amine, Ca^{2+} released from lime competes with the collector for the negative sites on the mineral surface. In addition, it decreases the availability of negative sites on the mineral surface, if it is a potential-determination ion. In flotation systems with carboxylic collectors, $Ca(RCOO)_2$ precipitates are formed in solution leading to unproductive consumption of collector (Pugh and Stenius, 1985; Morgan *et al.*, 1986; Dávila-Pulido and Uribe-Salas, 2014). Ca^{2+} can also act as an activating agent for gangue minerals (Kou *et al.*, 2016) adsorbing on the quartz surface and promoting the oleate adsorption by an ion exchange mechanism, in which the surface hydroxyl (OH^-) is replaced by the collecting ion ($RCOO^-$). So, in these systems, the presence of Ca^{2+} in solution is detrimental since it contributes to the depression of the mineral to be floated and/or for greater reagent consumption (Nanthakumar *et al.*, 2009) and it can also promote flotation of the gangue mineral. In others systems, regardless the type of collector, $Ca(OH)^+$ ions specifically adsorbed on mineral surfaces behave as active sites for adsorption of organic depressants such as polysaccharides (Solari *et al.*, 1986; Liu and Zhang, 2000; Bicak *et al.*, 2007; Baltar *et al.*, 2013), lignosulfonates (Ansari and Pawlik, 2007; Ma and Pawlik, 2005; Ma and Pawlik, 2007; Fu *et al.*, 2018) and quebracho (Iskra *et al.*, 1973; Fuerstenau, 1982). Ansari and Pawlik (2007) and Ma and Pawlik (2007) pointed out that lignosulfonates only adsorb on chalcopyrite and talc surfaces when the pH is regulated with calcium hydroxide. In the flotation of complex sulfide ores, the ion Ca^{2+} acts as a depressant for gangue minerals (such as pyrite). The $Ca(OH)^+$ species adsorbs by electrostatic interaction on the gangue mineral surface with excess of negative charge preventing dixanthogen formation (Dávila-Pulido and Uribe-Salas, 2014). The ionic Ca^{2+} predominates largely in acidic and moderately alkaline media, while $Ca(OH)^+$ is the most abundant specie at pH range 10,0-12,5. Above pH 13 prevails the $Ca(OH)_2$ precipitate (Mu *et al.*, 2016; Kou *et al.*, 2016). In turn, the carbonate ions (CO_3^{2-}) from soda ash can form insoluble precipitates with the dissolved cations in the pulp, preventing these species from participating in the flotation process.

Amines are weak bases derived from ammonia, so they are present in the pulp as molecular, ionic or dimers species, depending on the pH (Somasundaran and Ananthapadmanabhan, 1979; Gao *et al.*, 2015). The amine ionization occurs by protonation and the cationic specie RNH_3^+ predominates at the acid to low alkaline pH range. The adsorption occurs predominantly by a nonspecific mechanism based on electrostatic interactions (Laskowski, 1993). The adsorption is extremely fast; Baltar and Oliveira (1998) observed that the zeta potential of the quartz surface changes from -60 mV to about zero in 8 seconds by addition of dodecylamine. The floatability depends on the adsorption density of the collector to provide a minimum contact angle for efficient flotation (Trahar, 1981; Muganda *et al.*, 2011). Non-specific physical adsorption is poorly selective since the cationic amine can adsorb on any negative surface. In addition, the soluble cationic species compete with the amine for the negative sites on the mineral surface (Scott and Smith, 1993). In industrial flotation plants, water usually contains a significant amount of dissolved ions from the mineral surfaces (Rao *et al.*, 1988; Biçak *et al.*, 2012; Ikumapayi *et al.*, 2012; Manono *et al.*, 2013) which reduce the collector adsorption density and, as a consequence, decrease the flotation kinetics and/or increase reagent consumption. Since the amine group has a positive charge, it might attach to a negatively-charged mineral surface. So, the pH adjustment is a key step in most flotation processes.

Oleate is a salt of the oleic acid, which is a carboxylic acid with 17 carbons in the chain. The aqueous solution of a carboxylic collector may contain species ($RCOO^-$), (RCOOH), as well as those resulting from chain-chain associative interactions (RCOOH.RCOO) and $(RCOO)_2^{2-}$. The ionic species $RCOO^-$ and $(RCOO)_2^{2-}$ widely predominate in a strongly alkaline medium. These species adsorb by a chemisorption mechanism producing a hydrophobic chemical compound on the mineral surface. This mechanism predominates until formation of a monolayer. Carboxylic collectors have high affinity for the alkaline earth metal ions (such as calcium) to form virtually insoluble compounds. Until the monolayer is completed these cations in solution can precipitate oleate ions inducing an unproductive consumption of the collector and so, lowering surface hydrophobicity (Pugh and Stenius 1985, Morgan *et al.*, 1986; Hu *et al.*, 1986).

This study aimed to verify the influence of the ionic species released by the pH regulators in the calcium-bearing mineral flotation with amine or oleate as collector.

2. Experiment

2.1. Materials

2.1.1 Mineral sample

A dolomite sample supplied by ARMIL Mining, from Rio Grande do Norte State (Brazil), was used as a model of the salt-type minerals.

2.1.2 Reagents

A commercial diamine manufactured by the *Air Products Brazil* under the trade name of *Tomamine* M73 was used as collector. Oleate was produced by saponification of oleic acid (supplied by Nuclear Chemicals) in a 5/1 weight ratio. Analytical grade calcium hydroxide, sodium hydroxide, and sodium carbonate provided by *Química Moderna*, were used for pH adjustment.

2.2 Equipment

The high grade of the sample was confirmed by a X-ray Diffractometer Bruker, model D2 Phaser, using a *Bruker-Lynxeye* detector operating with 300 W, Cu-Kα_1 (λ= 1,5406 Å) irradiation, and 2θ = 4-80°. *Bruker EVA* with COD database (REV 89244 2013.10.11) was used for indexing the samples.

Flotation experiments were performed in a CDC mechanical cell, model CFB-1000-EEPNBA, equipped with an automatic device for froth collection.

A ZETASIZER Malvern model Nano-ZS90 and an IR Tracer 100 Shimadzu were used to support the interpretation of the flotation results.

2.3. Methods

2.3.1 Flotation

The experiments were carried out in a six-liter vat, with the 37 x 150 µm fraction of the mineral sample, following the standard flotation procedure. Dolomite was dispersed in the deionized water to form a suspension containing 10% solids by weight. The pH regulator was the first reagent added. After pH adjustment, the collector was added, and the pulp stirred for 1 minute (in the amine tests) or 5 minutes (in the oleate tests). Pulp alkalinity was not adjusted during the flotation and no frother was used. After adding the reagents, the air inlet was released to start flotation. The impeller speed was maintained at 1500 rpm in all tests. At the end of each flotation test, the products were filtered, dried and weighed for chemical analysis and mass/metallurgical recovery determination.

2.3.2 Zeta potential measurements

The Zeta potential measurements were carried out using mineral particles sized below 38 µm, added to a 10^{-3} M KBr indifferent electrolyte solution. After conditioning of the suspension containing 0.1% solids (by weight), the pH was adjusted, and the sample was placed in an appropriate cell for zeta potential determination by electrophoretic measurements.

2.3.3 FTIR spectroscopy measurements

The infrared spectra were registered for natural dolomite and, after conditioning with diamine in pH 12 adjusted with $Ca(OH)_{2(aq)}$. The FTIR spectra in the 4000-400 cm^{-1} were recorded with KBr pellets method.

3. Results and discussion

3.1 Dolomite Flotation with amine

Fig. 1 presents the dolomite recovery with diamine (100 g/t) as a function of the pH and regulator type. The results indicate that (1) dolomite floats only at very high alkalinity, and (2) the best results were achieved with calcium hydroxide.

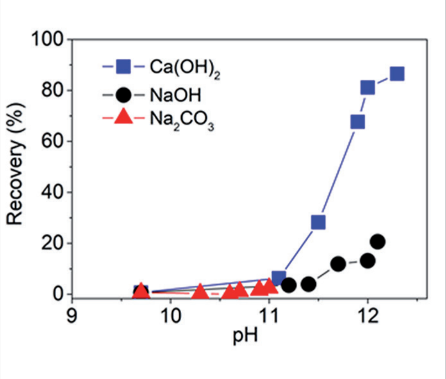

Figure 1 – Dolomite flotation with different regulators as a function of the pH.

The results (Fig. 1) show that dolomite only floats at pH range above 11. Three main factors contribute for this: (1) as can be shown by the species distribution diagram (Fig. 2), the concentration of the cationic specie RNH_3^+ decreases around pH 9; (2) in the pH range 9.5-10.5, there are few negative sites in the dolomite surface for adsorption of the cationic species (Fig. 3); and (3) dolomite is a sparingly soluble mineral, releasing Ca^{2+} and Mg^{2+} ions into the pulp (Chen and Tao, 2004). These dissolved cationic species compete for the few surface active sites, resulting in a low adsorption density of the collector and so, in an insufficient surface hydrophobicity for flotation.

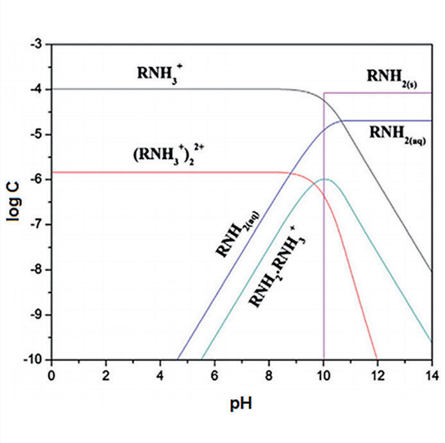

Figure 2 – Species distribution diagram for DDA (1 x 10^{-4}M) as a function of pH (Liu et al., 2015).

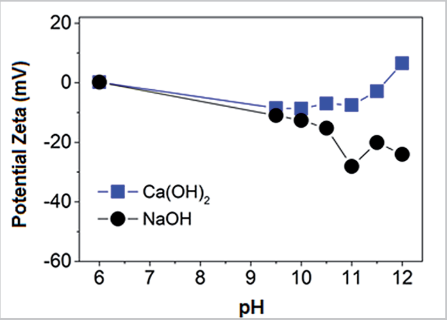

Figure 3 – Zeta potential of dolomite as a function of the pH for different regulators.

The higher recovery is observed when pH is adjusted with calcium hydroxide (Fig. 1) in the pH range where DDA molecular specie predominates (Fig. 2), indicating a non-electrostatic mechanism for collector adsorption on the dolomite surface. Therefore, the adsorption occurs by a chemical interaction mechanism. The amine molecule (RNH_2) has an unshared electrons pair in the nitrogen atom that has a strong ability to complex metals by means of coordinated covalent bonds (Cartmell and Fowles, 1956). This adsorption mechanism was also suggested by Freeman *et al.* (2009) and Gao *et al.* (2015). The best performance of the calcium hydroxide, as compared with the other regulators can be explained by the formation of RNH_2-Ca complex on the surface of dolomite. The increase of the zeta potential shown in Figure 3 suggests an adsorption of the Ca^{2+} ions on the surface of dolomite. The dissolution of calcium hydroxide releases Ca^{2+} that is a determining-potential ion for dolomite, hence, there is a calcium-enrichment at the dolomite surface enhancing the availability of active sites for collector adsorption. Dolomite does not float if the pH is adjusted with sodium carbonate. This can be attributed to surface carbonation (Valdiviezo and Oliveira, 1991; Sayilgan and Arol, 2004), due to the formation of calcium carbonate that prevents the adsorption of the collector. Therefore, there is a strong relationship between the presence of free calcium on the dolomite surface and DDA molecular specie adsorption.

The predominance of the molecular species of the diamine in solution at high alkalinity was confirmed by conductivity determinations. Fig. 4 shows the difference in conductivity of the two different diamine concentrations (12 and 24 mg/L) compared to distilled water. In the pH range where the cationic species prevail, the conductivity increases with the diamine concentration. At pH 10, no increase in conductivity with amine concentration was observed, indicating predominance of the molecular specie.

The presence of amine on the dolomite surface was detected by infrared spectroscopy analysis (Fig. 5). The region of the spectra located in the range of 1800 cm^{-1} to 1400 cm^{-1} displays the amine adsorption. The bands at 1636 cm^{-1} and 1653 cm^{-1} (Fig. 5b) are attributed to NH_2 (Nakanishi and Solomon, 1977; Lima *et al.*, 2005). Other bands characteristic for amine are not observed due to the superposition of vibration bands of water and dolomite.

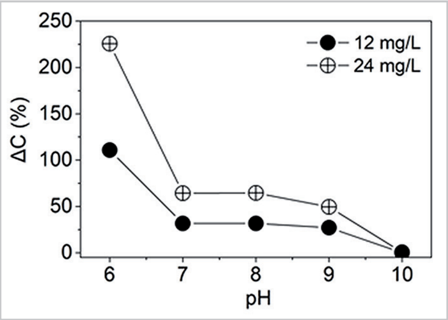

Figure 4 – Amine solution conductivity increases (ΔC) in relation to conductivity of the distilled water as a function of pH.

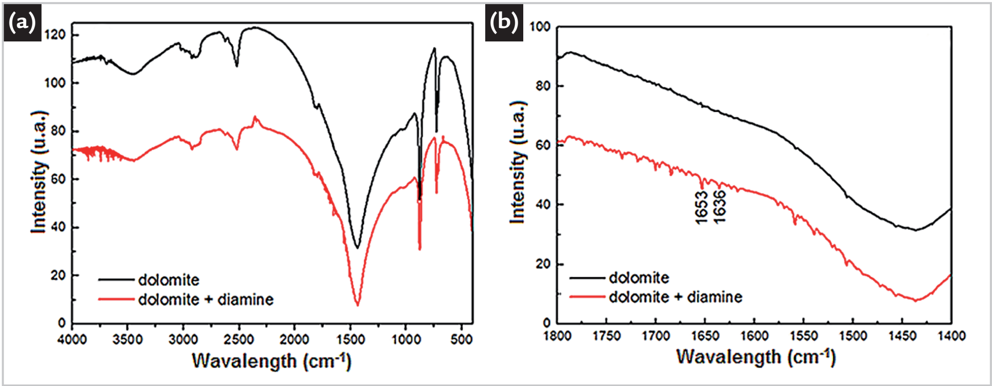

Figure 5 – FTIR spectra of natural dolomite and dolomite after
contact with the diamine solution: (a) highlighting the most interesting region of the spectra (b).

3.2 Dolomite flotation with oleate

Dolomite is a sparingly soluble mineral that releases Ca^{2+} and Mg^{2+} in the pulp. These species consume part of the collector with the formation of precipitates, decreasing the adsorption density on the mineral surface, and hence lowering hydrophobicity and recovery (Pugh and Stenius, 1985). The mineral solubility decreases by increasing pH (Chen and Tao, 2004; Horta *et al.*, 2016).

Fig. 6 shows the results of the dolomite flotation with oleate (100 g/t) as a function of pH and regulator. As can be seen, recovery increases from a given pH that is different for each reagent. As alkalinity increases, the amount of Ca^{2+} and Mg^{2+} dissolved in the pulp decreases and, consequently, increases the availability of the collector to the surface. There seems to be a limiting concentration for the cationic species, below which the collector reaches the mineral surface in adequate amount, increasing recovery. The alkalinity required for the recovery increases following the order: calcium hydroxide > sodium hydroxide > sodium carbonate.

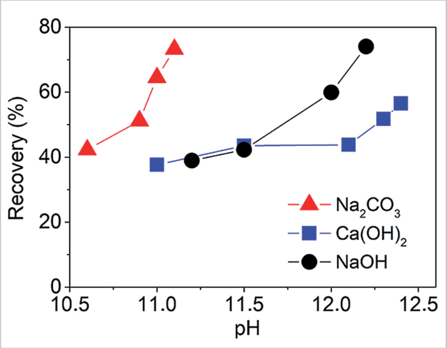

Figure 6 – Dolomite flotation with oleate as a function of the pH.

The higher recovery was observed with soda ash as pH regulator. The dissolution of sodium carbonate generates CO_3^{2-} ions in the pulp (reaction 1), which can interact: (a) with the water molecule raising the pH (reaction 2); and (b) with the soluble cationic species (Ca^{2+} and Mg^{2+}) to form precipitates (reaction 3), increasing the availability of the $RCOO^-$ ions for mineral surface adsorption (DiFeo *et al.*, 2004; Dávida-Pulido and Uribe-Salas, 2014), as illustrated in Fig.7.

$$Na_2CO_{3(s)} \rightleftharpoons 2Na^+_{(aq)} + CO_3^{2-}{}_{(aq)} \quad (1)$$

$$CO_3^{2-}{}_{(aq)} + H_2O_{(l)} \rightleftharpoons HCO_3^-{}_{(aq)} + OH^-_{(aq)} \quad (2)$$

$$CO_3^{2-}{}_{(aq)} + Ca_2^+{}_{(aq)} \rightleftharpoons CaCO_{3(s)} \quad (3)$$

The formation of $CaCO_3$ precipitates explains why the alkalinity requirement for increasing recovery is lower when the pH is adjusted with Na_2CO_3. Furthermore, the solubility of dolomite decreases by adding CO_3^{2-} ion in alkaline solutions (Chen and Tao, 2004). At the other extreme, the worst results appeared when using calcium hydroxide. This was attributed to calcium hydroxide dissolution that promotes an additional supply for Ca^{2+} ions, resulting in greater unproductive consumption of the collector (Fig. 7). The increase in dolomite recovery occurs at extremely alkaline pH, when the surface solubility virtually ceases (Chen and Tao, 2004).

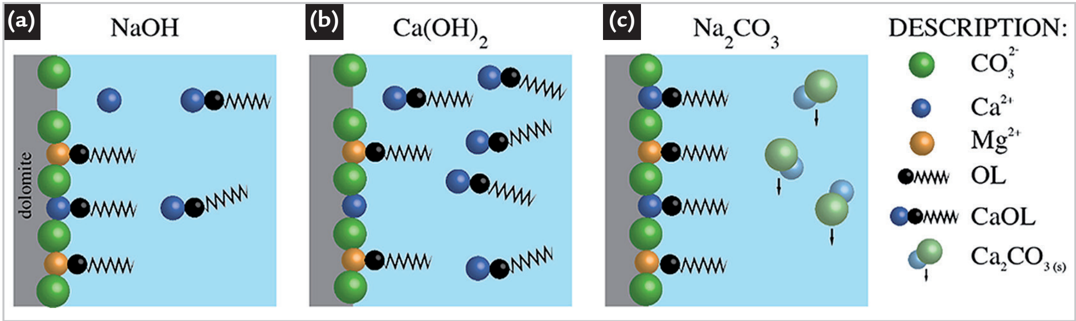

Figure 7 - Oleate adsorption on dolomite surface on the presence of pH regulators: sodium hydroxide (a), calcium hydroxide (b) and sodium carbonate (c).

4. Conclusions

The results demonstrate the importance of an appropriate choice of the pH regulator, which depends on the ore and type of collector used in the process.

In the low alkalinity dolomite flotation with amine, when the ionic species of the collector prevails, the cationic species (Ca^{2+} and Mg^{2+}) released from the soluble mineral compete with the amine for the few available negative sites on the mineral surface. This results in poor flotation recovery due to low collector adsorption density. The dolomite flotation with amine is only possible in strong alkaline medium, where the molecular specie of the collector prevails. The adsorption occurs by a complexation mechanism between the calcium (or magnesium) of the dolomite surface and the nitrogen of the molecular amine. In these systems, lime must be used because it acts as a calcium supplier to the mineral surface.

In calcium-bearing mineral flotation, using oleate as the collector, it is recommended the use of soda ash which, in addition to changing the pH, provides the system with CO_3^{2-} that precipitates the dissolved Ca^{2+} and Mg^{2+}, reducing or avoiding the unproductive consumption of the collector.

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