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EFEITO DOS ÍONS Ca^{2+} E Mg^{2+} NA FLOTAÇÃO CATIÔNICA REVERSA DE MINÉRIO DE FERRO ITABIRÍTICO

D. G. DA CRUZ[1], P. S. M. GONÇALVES[2], D. F. LELIS[3], R. M. F LIMA[4]
Departamento de Engenharia de Minas, Universidade Federal de Ouro Preto[1,2,3,4]
ORCID ID: https://orcid.org/0000-0003-3647-2029
daniel.cruz@ufop.edu.br

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RESUMO

Neste trabalho foi avaliado o efeito dos íons Ca^{2+} e Mg^{2+}, adicionados como sais de cloreto, na flotação catiônica reversa em pH 10,5 de uma amostra de minério de ferro itabirítico. Em geral, foi observado que concentrações totais de íons maiores que 83 mg/L em solução aquosa aumentou a recuperação de Fe e teor de SiO_2 nos concentrado obtidos comparado com os ensaios efetuados na ausência dos mesmos. Ocorreu o oposto com o teor de Fe. Este efeito é devido à atração eletrostática entre as espécies positivas provenientes da hidrólise destes íons em solução aquosa e a superfície negativa do quartzo, que evitou a adsorção dos íons amonium, cujo principal mecanismo de adsorção é por atração eletrostática dos mesmos com a superfície mineral negativa. Contudo, o uso de ácido etilenodiiaminotetracético - EDTA após o condicionamento do minério com os íons Ca^{2+} e Mg^{2+}, em proporções adequadas de EDTA para íons totais, produziu concentrados com teores de Fe e SiO_2 similares aos concentrados desta amostra na ausência de íons, uma vez que eles foram complexados pelo EDTA e não foram mais adsorvidos na superfície negativa do quartzo.

PALAVRAS-CHAVE: minério de ferro, flotação catiônica reversa, íons Ca^{2+} e Mg^{2+}, EDTA.

EFFECT OF Ca^{2+} AND Mg^{2+} IONS ON THE REVERSE CATIONIC FLOTATION OF ITABIRITIC IRON ORE

ABSTRACT

In this work the effect of the Ca^{2+} and Mg^{2+} ions, added as salts of chlorine, on the reverse cationic flotation at pH 10.5 of an itabiritic iron ore sample was evaluated. In general, it was observed that the total ion concentrations higher than 83 mg/L in aqueous solution increased the Fe recovery and SiO_2 content in obtained concentrates compared in absence of them. The opposite occurred with Fe content. This effect is due to the electrostatic attraction between the positive species from these ions hydrolysis in aqueous solution and the negative surface of quartz, which avoided the adsorption of the ions aminium, whose main adsorption mechanism is by electrostatic attraction of them with the mineral negative surface. However the use of ethylenediaminetetraacetic acid - EDTA after the ore conditioning with Ca^{2+} and Mg^{2+} ions, into adequate proportions of EDTA to total ions, produced concentrates with similar contents of Fe and SiO_2 with the concentrates of this sample in absence of ions once they were complexed by EDTA and is no longer adsorbed on negative quartz surface.

KEYWORDS: iron ore, reverse cationic flotation, Ca^{2+} and Mg^{2+} ions, EDTA.

1 INTRODUCTION

The reverse cationic flotation is applied to concentrate itabiritic iron ores, with particle size -210 µm, using amine as a quartz collector and gelatinized corn starch as iron minerals depressant. The process, generally conducted at pH 10.5 with 45%-50% w/w solids pulp, aims to obtain concentrates with Fe content higher than 65% and SiO_2 smaller than 2% for *pellet feed* production, used in pelletizing and subsequent application in the steel industry (Moraes & Ribeiro, 2019; Pattanaik & Venugopal, 2018; Nakhaei & Irannajad, 2017; Filippov *et al.,* 2014; Lima *et al.,* 2013; Ma *et al.,* 2011; Vieira & Peres, 2007; Araujo *et al.*, Lima, 1997; Houot, 1983).

The selectivity in the separation between quartz and hematite using starch and amine is possible due to the differences in affinity of the starch with the minerals (stronger affinity with hematite compared with quartz). The quartz isoelectric point occurs at pH = 1.8 (Lopes & Lima, 2009). As in pH > 1.8, the number of negative sites on its surface is greater than the number of positive sites, this fact favors the electrostatic attraction between the aminium ions and the negative sites, making the mineral hydrophobic. The adsorption mechanisms of the starch on the hematite surface are: hydrogen bonds, hydrophobic interactions between the mineral surface and the hydrophobic portion of the polysaccharide, and the formation of complexes with Fe (chemisorption) on the mineral surface (Khosla *et al.,* 1984, Pinto *et al.*, 1992; Peres & Correa, 1996; Dziechciarek *et al.,* 2002, Pavilovic & Brandão, 2003; Filipov *et al.*, 2014; Shrimali & Miller, 2015; Aguiar *et al.*, 2017; Veloso *et al.*, 2018). Thus, the adsorbed starch on the hematite surface prevents the adsorption of the amine species on the mineral surface, which remains hydrophilic. In addition, hematite's aggregates with particles < 5 µm are formed by the starch polymer macromolecules (selective flocculation) in flotation of siliceous iron ore, avoiding the mechanical drag of iron oxide fine particles to floated product (Oliveira & Rubio, 2011; Liu & Peng, 2016; Shrimali et al., 2018).

The depressant effect of polyvalent cations (Ca^{2+}, Al^{3+}, Fe^{3+}, Mn^{2+}, Mg^{2+}) and their hydrolysis products on the quartz flotation is related to these electrostatic attraction by the negative sites present on the mineral surface, preventing the adsorption of the amine (competition occurs between the cationic species from hydrolysis of polyvalent positive ions and aminium by the quartz surface), whose main mechanism occurs by electrostatic attraction between the aminium cations and the negative sites, besides the formation of hydrogen bonds between the NH_2 groups with SiOH on the quartz surface, and precipitation of the reagent at the solid/liquid interface (Scott & Smith, 1993; Vidyadhar *et al.,* 2008; Pinheiro *et al.,* 2010, Pinheiro *et al.,* 2012, Liu *et al.,* 2013, Boujounoui *et al.,* 2015; Cruz & Lima, 2015; Lelis *et al.,* 2016, Nakhaei & Irannajad, 2017; Mhonde *et al.,* 2017; Pattanaik & Venugopal, 2018; Tripathy, 2018). Therefore, the concentrations of Ca^{2+} and Mg^{2+}, in the industrial recycled waters of process, higher than 15 mg/L (Houot, 1983), coming from the addition of reagents such as lime for pH control of pulps for pipeline transport or from the dissolution of carbonates present in the gangue, should be controlled to levels that do not affect the performance of the cationic flotation of iron ore containing quartz and carbonates (Mamede *et al.,* 2016; Carvalho & Peres, 2004).

In this work, the systematic studies of the effects of the Ca^{2+} and Mg^{2+} ions, present in aqueous medium, on the reverse cationic flotation of an itabiritic iron ore are presented. The possibility of using EDTA as a complexing agent of these cations was evaluated, aiming at the reestablishment of the selective separation between quartz and iron minerals.

2 MATERIALS AND METHODS

2.1 Ore and reagents

An itabiritic iron ore sample collected in the industrial flotation circuit of a mine located in Quadrilátero Ferrífero-Brazil was used. As can be seen on Table 1, approximately 35% of the sample particles were -37 µm, with Fe_T content (51.3 wt %) much higher than in the particle size range +37 µm (38-44 wt %). The inverse occurs with the SiO_2 contents. The overall contents of the other impurities (P= 0.04 wt %, Al_2O_3=0.3 wt %) are already within the specifications for pelletizing application (P=0.07 wt %, Al_2O_3=1.49 wt %) (Silva *et al.,* 2016; Lima, 1997).

Table 1: Granulochemical analysis of iron ore sample (Cruz, 2015).

Size fraction (µm)	Weight (%)	Content (wt %)				LOI (%)	Distribution (%)	
		Fe_T	SiO_2	Al_2O_3	P		Fe	SiO_2
+147	16.6	45.8	29.0	0.4	0.06	4.0	17.1	15.1
-147+105	14.3	38.3	41.3	0.2	0.04	2.5	12.2	18.5
-105+53	18.7	38.0	42.1	0.2	0.03	2.3	15.9	24.8
-53+37	15.6	42.3	36.0	0.2	0.03	2.1	14.8	17.7
-37	34.8	51.3	21.9	0.4	0.04	2.7	40.0	24.0
Feed	100	44.6	31.8	0.3	0.04	2.7	100	100

By size analysis, using the Cilas 1064 laser size analyzer, it was verified that approximately 4.5% of the sample particles were -4 µm. The minerals identified in the X-ray diffractogram using the total powder method collected by the Panalytical Expert^3 X-ray diffractometer (λ_{Cu} = 1.54 Å), operating at 45 kV and current of 40 mA, were: quartz, hematite and goethite. Therefore, the LOI (loss of ignition) and Al_2O_3, contents are probably related to goethite aluminous, which is very common in Algeria's ore reserves (Santos & Brandão, 2005; Magalhães *et al.,* 2007).

The reagents used in the flotation experiments were: commercial amine Flotigam EDA, with a neutralization grade equal to 50% (Clariant S.A.), commercial corn starch (Unilever S.A.), $CaCl_2$, $MgCl_2.6H_2O$ and ethylenediaminetetraacetic acid, disodium salt - EDTA ($C_{10}H_{14}N_2O_8Na_2.2H_2O$) of the brand Synth, HCl and NaOH (Vetec LTDA), for pH control. The ions concentration at feed water used in flotation tests were of 2.4 mg/L Ca^{2+} and 0.8 mg/L Mg^{2+}.

2.2 Flotation tests

For the bench flotation tests, carried out at pH 10.5 (adjusted with NaOH) and 45% w/w of solids on the pulp, a 2 L CDC mechanical cell, model CFB-1000-EEPNBA, with automatic froth removal, adjusted rotation at 1200 min^{-1} was used. During the flotation time (3 minutes), the pulp volume was kept constant by the addition of tap water with pH previously adjusted to 10.5.

In the first phase, preliminary experiments were carried out, using statistical planning of experiments (Minitab 17 software) with replica for starch (200 and 400 g/ton) and amine (50 and

150 g/ton) dosages, aiming to obtain concentrates with Fe and SiO_2 contents compatible with the grades of rougher stage concentrates (63-66 wt% Fe, 4-6.5 wt% SiO_2 and 75-85% Fe recovery), usually obtained with *Quadrilátero Ferrífero* itabiritic iron ores (Lopes and Lima, 2011, José et al., 2018). The conditioning times used for the starch and amine were respectively 5 and 3 minutes, previously determined. Afterwards, the optimum dosages of starch (400 g/ton) and amine (50 g/ton) were fixed to evaluate the effects of cations concentration, conditioned at natural pH for 6 minutes (Lelis, 2014), on the iron ore flotation. At this stage, the concentrations of the salts ($CaCl_2$ and $MgCl_2$) varied from 0 to 1000 mg/L: Ca^{2+} = 0 to 360 mg/L; Mg^{2+} = 0 to 255 mg/L and Ca^{2+} + Mg^{2+} = 0 a 320 mg/L, in the stoichiometric proportion of these ions in the chemical formula of dolomite (62:38). Finally, in the complexation tests (with EDTA) of the cations present in the pulp, the total concentration of the salts was fixed at 250 mg/L: Ca^{2+} = 90.1 mg/L; Mg^{2+} = 63.7 mg/L; Ca^{2+} + Mg^{2+} (56 mg/L + 24 mg/L) = 80 mg/L, then the EDTA was added and the conditioning was carried out at natural pH for another 6 minutes (Lelis, 2014). In all tests, the pH of the flotation pulp was adjusted at 10.5 only after the addition of the starch (adjusted with NaOH).

3 RESULTS AND DISCUSSION

Figure 1 shows the cube plots, which express the effect of the starch and amine dosages on the average values of Fe metallurgical recovery, Fe and SiO_2 contents in the concentrates and the Gaudin selectivity index (S.I.), calculated as a function of Fe and SiO_2 contents. As can be observed, in all the conditions, Fe recoveries higher than 87% and concentrates with Fe content higher than 64.5 wt % and SiO_2 contents less than 3 wt % were achieved, which are compatible with iron ore flotation concentrates from the Quadrilátero Ferrífero (Lima *et al.,* 2011; José *et al.,* 2018). Thus, for the subsequent tests, the starch and amine dosages were fixed at 400 g/ton and 50 g/ton, respectively.

Figure 2 shows the effects of Ca^{2+} and Mg^{2+} cations on the studied variables: Fe metallurgical recovery, Fe and SiO_2 contents in the obtained concentrates and Gaudin's selectivity index (S.I.).

Figure 2 clearly shows the deleterious effect of the cations on all variable responses analyzed, especially from 250 mg/L of the salts concentration [90.1 mg/L Ca^{2+}; 63.7 mg/L Mg^{2+}; 80 mg/L (Ca^{2+} + Mg^{2+})]: in this condition, a decrease of 10% in Fe content and a 15% increase in the SiO_2 content in the concentrate is observed. An accentuated decrease in the selectivity index of Gaudin (S.I.) from 12 to values below 5 is also noted. These results are consistent with values reported in the literature (Araújo & Coelho, 1992; Pinheiro *et al.,* 2012; Lelis, 2014). The concentration of cations fixed for the following stage [salts at 250 mg/L: Ca^{2+} = 90.1 mg/L; Mg^{2+} = 63.7 mg/L; Ca^{2+} + Mg^{2+} = 80 mg/L] is consistent with the values obtained by Pinheiro *et al.* (2012) (93 mg/L Ca^{2+} and 69 mg/L Mg^{2+}), which achieved a lower quartz recovery (32%) when compared to the condition using distilled water [Ca^{2+} + Mg^{2+} < 10 mg/L], in which the floatability was about 90%, for 400 g/ton dodecylamine, at pH 10.0.

At pH 10.5, the predominant species in the pulp are: Ca^{2+} for $CaCl_2$ and Mg^{2+}, $MgOH^+$ and $Mg(OH)_{2(s)}$, in this order, for $MgCl_2$ (Butler, 1964 *apud* Fuerstenau *et al.,* 1985). The isoelectric point of $Mg(OH)_{2(s)}$ occurs at pH 11 (Pokrovsky & Schott, 2003; Schott, 1981). Thus, the species from the dissolution of the added salts followed by cations hydrolysis in the pulp (pH ~ 10.5) have a positive charge and are attracted by negatively charged quartz, preventing the adsorption of aminium ions

(Scott & Smith, 1993; Pinheiro *et al.,* 2010; Cruz & Lima, 2015; Lelis *et al.,* 2016; Pattanaik & Venugopal, 2018).

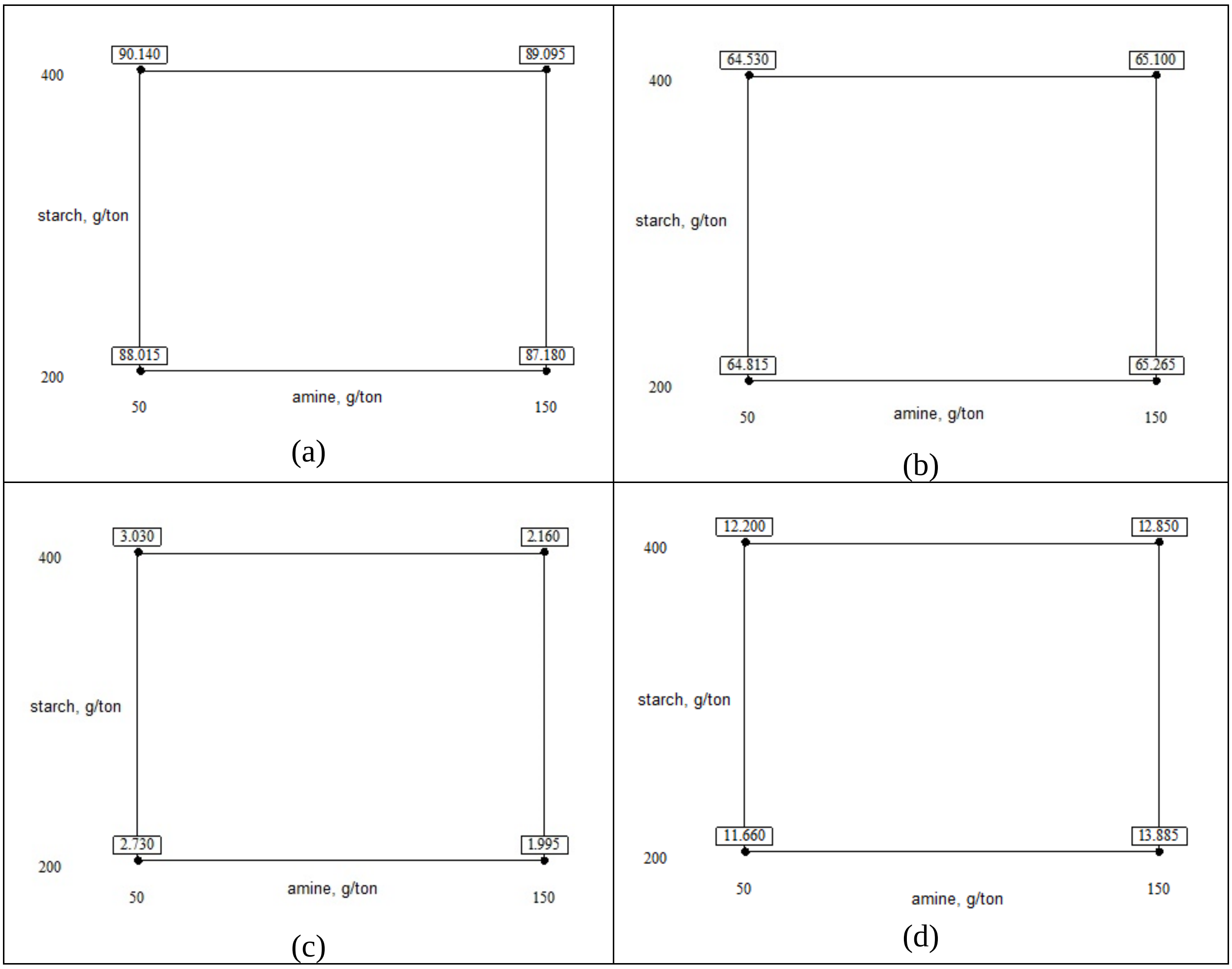

Figure 1: Cube plots for response variables: (a) Fe metallurgical recovery, (b) Fe content, (c) SiO_2 content and (d) S.I., in function of amine and starch dosages, for a confidence level of 95%.

Figure 3 shows the effect of the EDTA addition on the iron ore flotation, pulp containing 250 mg/L salts [Ca^{2+} = 90.1 mg/L; Mg^{2+} = 63.7 mg/L; Ca^{2+} + Mg^{2+} = 80 mg/L]. It is observed that there was a gradual increase of Fe contents and selectivity index with the increase of the EDTA concentration, the inverse occurred with the SiO_2 contents. It is noticed that for the EDTA concentration equal to 600 mg/L, the Fe recovery and the quality of the concentrate obtained (Fe = 63 wt %, SiO_2 = 5.5 wt %), as well as the selectivity index (S.I. ≅ 10), were approximately equal to the values obtained in the flotation of the ore in the absence of cations (Fe = 64.5 wt %, SiO_2 = 3.0 wt %, S.I. ≈ 12). In other words, the reestablishment of the selectivity in the separation between quartz and hematite was achieved for the ratio EDTA : Ca^{2+} = 6.6, EDTA : Mg^{2+} = 9.4 and EDTA : (Ca^{2+}+Mg^{2+}) = 7.5. Lelis (2014) in quartz and hematite microflotation tests using Ca^{2+} (72 mg/L) and Mg^{2+} (51 mg/L), for 600 mg/L of EDTA, obtained the proportions EDTA : Ca^{2+} = 8.3 and EDTA : Mg^{2+} = 11.7, for the reestablishment of mineral floatability. According to the data presented here, the EDTA : cations ratio is coherent with that used by Lelis (2014), which obtained slightly higher values for this ratio.

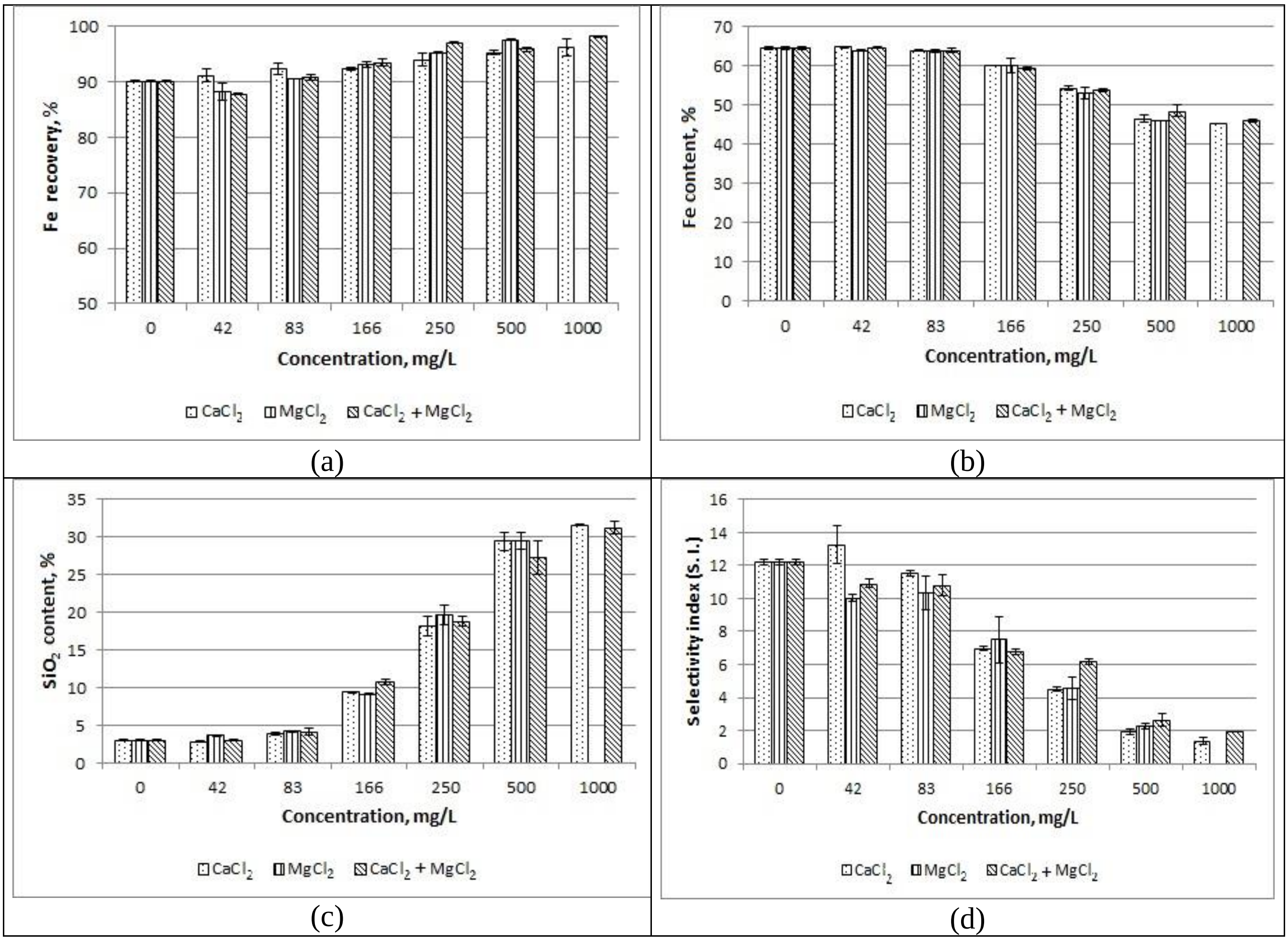

Figure 2 – Fe metallurgical recovery (a), Fe content (b) SiO_2 content (c) in the concentrates obtained and selectivity index (S.I.) (d) in function of the concentration of $CaCl_2$, $MgCl_2$ and mixture $CaCl_2$ + $MgCl_2$ in the proportion 1.6 : 1. Other conditions: 45% w/w of solids on the pulp, 400 g/ton starch, 50 g/ton amine, pH 10.5.

4 CONCLUSIONS

Based on the results presented previously, it is concluded that total concentrations of Ca^{2+} = 30 mg/L, Mg^{2+} = 21 mg/L and (Ca^{2+} + Mg^{2+}) = 26.7 mg/L (18.7 mg/L Ca^{2+} + 8.0 mg/L Mg^{2+}) did not produce significant effects on the metallurgical recovery of Fe, on Fe and SiO_2 contents flotation concentrates of the itabiritic iron ore sample studied. For larger concentrations, increases in both Fe recovery and in the SiO_2 contents were observed. An inverse effect was observed in the Fe content and selectivity index. The EDTA use after the conditioning with the salts of the cations studied [Ca^{2+} = 90.1 mg/L; Mg^{2+} = 63.7 mg/L; Ca^{2+} + Mg^{2+} = 80 mg/L] allowed for the reestablishment of the selectivity in the separation between quartz and hematite for the following proportions: EDTA : Ca^{2+} = 6.6, EDTA : Mg^{2+} = 9.4 and EDTA : (Ca^{2+} + Mg^{2+}) = 7.5. Under these conditions, concentrates with up to 63 wt % of Fe and 4.5 wt % of SiO_2 were obtained, which are approximate to the contents (64.5 wt % Fe and 3.0 wt % SiO_2) of the tests conducted in the absence of the cations.

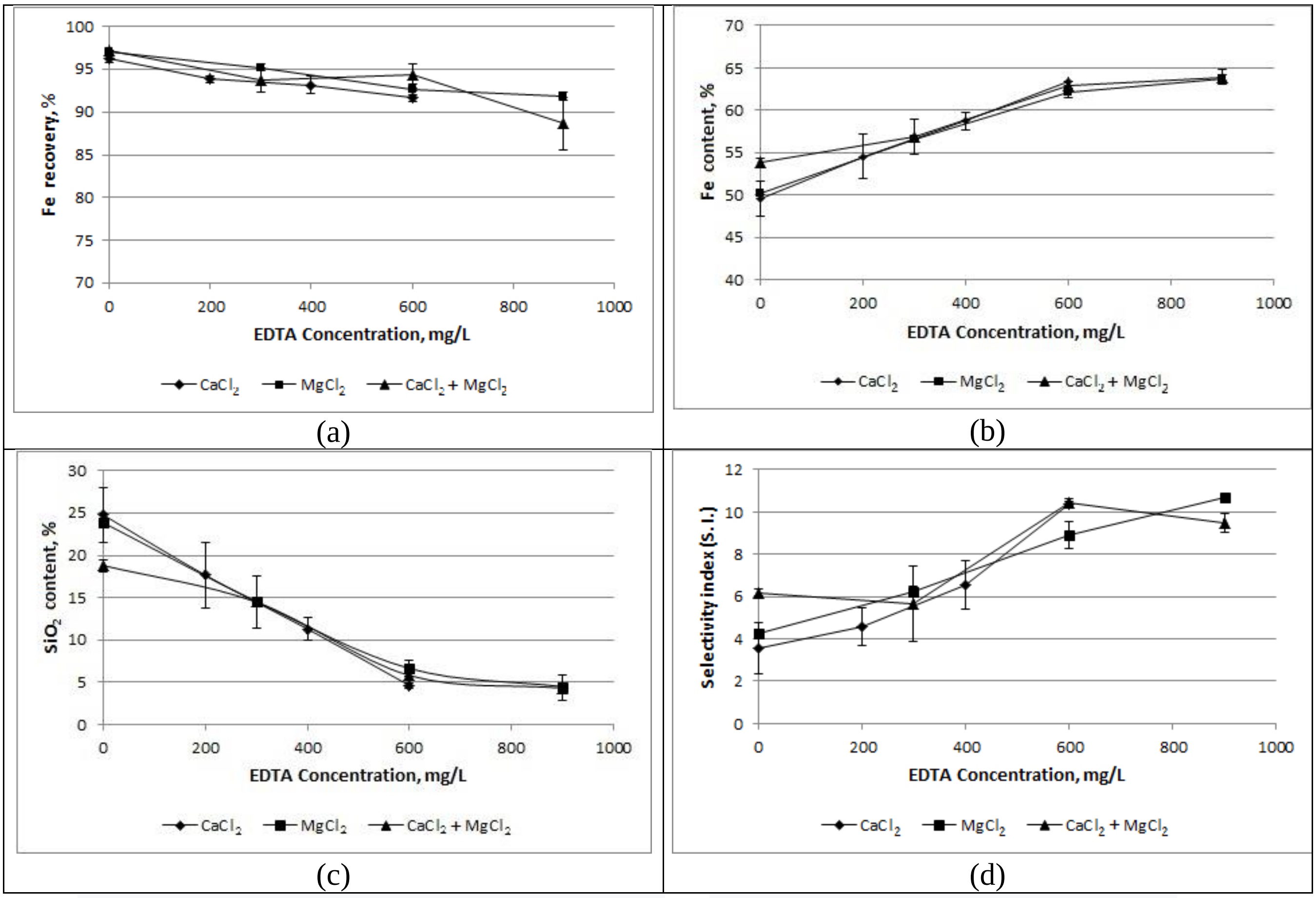

Figure 3 – Fe metallurgical recovery (a), Fe content (b) SiO_2 content (c) and S. I. (d) as function of EDTA concentration. Other conditions: 45% w/w of solids on the pulp, 250 mg/L of salts $CaCl_2$, $MgCl_2$ and mixture $CaCl_2$ + $MgCl_2$; 400 g/ton starch; 50 g/ton amine; pH 10.5.

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SOBRE OS AUTORES

D. G. DA CRUZ
Engenheiro Metalurgista e de Materiais pela UFOP. Mestrado e doutorado em Engenharia Mineral - Tratamento de Minérios pelo Programa de Pós-Graduação em Engenharia Mineral da UFOP.
E-mail: daniel.cruz@ufop.edu.br
ORCID ID: https://orcid.org/0000-0003-3647-2029

P. S. M. GONÇALVES
Engenheira de Minas pelo Departamento de Engenharia de Minas - Escola de Minas - Universidade Federal de Ouro Preto (DEMIN/EM/UFOP).
E-mail: paolasuzan@yahoo.com.br
ORCID ID: https://orcid.org/0000-0003-4487-0983

D. F. LELIS
Engenheira de Materiais pelo Centro Universitário do Leste de Minas. Mestrado em Engenharia Mineral - Tratamento de Minérios pelo Programa de Pós Graduação em Engenharia Mineral da UFOP.
E-mail: deisiane.lelis@gmail.com
ORCID ID: https://orcid.org/0000-0002-3197-0478

R. M. F LIMA
Engenheira de Minas. Mestrado e doutordo em Tecnologia Mineral pela UFMG. Professora titular do Departamento de Engenharia de Minas da Escola de Minas da Universidade Federal de Ouro Preto (DEMIN/EM/UFOP). Áreas de atuação: Caracterização tecnológica de minérios e rejeitos,flotação, métodos físicos de concentração de minérios, reprocessamento de rejeitos e separação sólido-líquido.
E-mail: rosa@ufop.edu.br
ORCID ID: http://orcid.org/0000-0002-0326-1797

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