REMOVAL OF IRON-BEARING MINERALS FROM GIBBSITIC BAUXITE BY DIRECT FROTH FLOTATION

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Abstract

The refractory bauxite needs to present less than 2.5% of Fe_2O_3 to be applied in the ceramics industry. The depletion of high Al_2O_3 grade deposits has stimulated the improvement of bauxite concentration methods in order to remove iron-bearing minerals. The objective of this study was to evaluate the influence of collector dosage, pH and milling time on the gibbsite flotation performance. Firstly, the sample mineralogical composition was determined by means of X-ray diffraction (XRD) and binocular loupe analysis. X-ray fluorescence (XRF) analysis was used to determine the sample chemical composition. Flotation was then accomplished by using hydroxamate as gibbsite collector, sodium silicate as silicate depressant and starch as iron-bearing minerals depressant. The bauxite Fe_2O_3 content was reduced from 7.66% to 4.81-5.03%. In addition, the flotation performance decreased by diminishing the pH from 9.5 to 8.5 or increasing the pH to 10.5. The milling time influence on the flotation indicates that the presence of slime can significantly affect the gibbsite concentration.

Keywords: Bauxite; Gibbsite; Direct flotation; Hydroxamate.

REMOÇÃO DE MINERAIS PORTADORES DE FERRO DE BAUXITA GIBSÍTICA POR FLOTAÇÃO DIRETA

Resumo

As bauxitas refratárias precisam apresentar teores de ferro menores que 2,5% para serem utilizadas na indústria de cerâmica. A exaustão dos depósitos com altos teores de Al_2O_3 tem estimulado a pesquisa por métodos de concentração de bauxita de forma a remover os minerais portadores de ferro. O objetivo deste estudo foi o de avaliar a influência da dosagem de coletor, pH e tempo de moagem no desempenho da flotação de gibsita em escala laboratorial. Inicialmente a amostra foi caracterizada mineralogicamente utilizando-se as técnicas de difração de raios-x (DRX) e análise em lupa binocular. A composição química da amostra foi determinada pelo método de Fluorescência de raios-x (FRX). Em seguida, foram realizados ensaios de flotação utilizando o hidroxamato como coletor de gibsita, silicato de sódio e amido como depressores de silicatos e minerais portadores de ferro, respectivamente. O teor de Fe_2O_3 foi reduzido de 7,66% para 4,81-5.03%. O desempenho da flotação reduziu com a diminuição do pH de 9,5 para 8,5 e com o seu aumento para 10,5. O tempo de moagem influenciou os resultados da flotação, indicando que a maior quantidade de lamas influenciou de forma significativa a concentração de gibsita.

Palavras-chave: Bauxita; Gibsita; Flotação direta; Hidroxamato.

1 INTRODUCTION

Aluminum (Al) is the third most abundant element in the earth's crust, and bauxite is the commercially most relevant material that comprises this metal. Bauxite contains aluminum hydroxide minerals (gibbsite – $Al(OH)_3$, diaspore – α-AlO(OH) or bohemite – γ–AlO(OH)) and impurities such as clay (especially kaolinite - $Al_2Si_2O_5(OH)_4$) goethite (FeO(OH)), rutile (TiO_2), hematite (Fe_2O_3) and quartz (SiO_2) [1-4].

About 90% of the industrially processed bauxite is consumed for metallic aluminum production, and 10%

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is applied in the production of refractory and abrasive materials, clay cement and chemical products [1]. In order to feed the aluminum metallurgical processes (Bayer and Hall-Héroult processes) the bauxite can contain 5-30% of Fe_2O_3. Nevertheless, refractory bauxite used in ceramics production must have less than 2.5% of Fe_2O_3 [4,5].

Brazil is the world's third largest producer of bauxite (12.7%), after Australia (29.9%) and China (18.2%). Most of the Brazilian bauxite beneficiation plants comprise the operations of comminution, classification and solid-liquid separation. Very few beneficiation processes include gravimetric concentration in order to remove silicates or magnetic separation to decrease the amount of iron-bearing minerals [4]. In this context, the depletion of high Al_2O_3 grade deposits has stimulated the improvement of bauxite concentration methods. Flotation has been pointed to as a promising technique to be applied in order to suit the bauxite to the market specifications [1-4,6,7].

The practice of diasporic bauxite concentration by flotation is common in China [8-15]. The bauxite beneficiation plant in Zhongzhou, Henan Province, for instance, concentrates diaspore by reverse froth flotation. The feed material contains 65% of diaspore as the primary bauxite mineral, with kaolinite, illite $((K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)])$ and pyrophyllite $(Al_2Si_4O_{10}(OH)_2)$ as the gangue silicate minerals. The plant operates with a mass recovery of 78.8% and an Al_2O_3 recovery in the concentrate of 84.9% [15].

On the other hand, gibbsitic bauxite has a significantly higher Al_2O_3 content than the diasporic bauxite [6]. Therefore, few works in the literature are dedicated to concentrate gibbsite from bauxite [2,3,5-7,10,16-18].

Regardless of the bauxite type, direct and reverse flotation routes have been applied in order to concentrate the aluminum hydroxide (gibbsite or diaspore) mineral. In the reverse flotation route the silicates are floated with cationic collectors at a pH under 6 and gibbsite/diaspore is depressed. Common reagents used in the cationic reverse flotation have been fatty amines as collectors, sodium silicate and starch as dispersant/depressants, and sulfuric acid and sodium carbonate as pH regulators. In the case of diasporic bauxite, hexametaphosphate is also applied as a diaspore depressant [2,3,6,7,10,18].

On the other hand, in direct flotation gibbsite/diaspore is floated with anionic collectors at pH greater than 9, while silicates and iron-bearing minerals are depressed with sodium silicate and starch, respectively [5-7]. Marino et al. [6] tested the direct and reverse flotation routes in order to concentrate the bauxite from Paragominas-PA, Brazil, which contains 47.7% of Al_2O_3 and 4.0% of reactive silica, besides kaolinite and iron-bearing minerals. The reverse flotation was accomplished at pH under 6 by using a mixture of fatty acids and amine as kaolinite collector, sodium hexametaphosphate as gibbsite depressant and corn starch as iron-bearing minerals depressant. The gibbsite concentrate comprised 55.9-70.9% of Al_2O_3 and 7.41-9.83% of Al_2O_3/SiO_2. The direct gibbsite flotation was carried out at pH 10.5 with a mixture of alkyl hydroxamate and fatty acid as gibbsite collector and sodium carbonate plus sodium silicate as iron-bearing minerals and kaolinite depressant. The resulting concentrate showed 53.4-58.3% of Al_2O_3 and 8.44-10.58% of Al_2O_3/SiO_2.

Bittencourt [5] were able to concentrate Brazilian refractory gibbsitic bauxite (50% gibbsite, 15% kaolinite and 35% quartz) by direct flotation using a quaternary amine salt as kaolinite and gibbsite collector at pH 6. The gibbsitic concentrate presented Al_2O_3 content of 97.4% and 90% metallurgical recovery. Barbosa et al. [18] and Barbosa et al. [19] carried out direct flotation experiments in order to remove iron-bearing minerals from the Barro Alto (Goiás state, Brazil) gibbsitic bauxite. S9849 hydroxamate (Cytec) was used as gibbsite collector, starch as iron-bearing minerals depressant and sodium silicate as silicates depressant. The gibbsite Fe_2O_3 content decreased from 7.57% to 3.44% and the Al_2O_3 grade increased from 84.55% to 93.80% with an Al_2O_3 recovery of 45.76%. In addition, the concentrate was submitted to magnetic separation and the non-magnetic product showed 1.91% of Fe_2O_3.

The purpose of this study was to investigate the most appropriate conditions (collector dosage, pH and milling time) for the gibbsite concentration by direct flotation with the Barro Alto bauxite. The main objective was to reduce the Fe_2O_3 content in order to make the bauxite suitable for ceramics production.

2 METODOLOGY

2.1 Sample Preparation and Characterization

The bauxite sample from Barro Alto, located in the north of Goiás state, Brazil, was crushed (in a Contenco jaw crusher) until 100% of the particles were smaller than 1.70 mm. The crushed product was then homogenized and divided in 1 kg samples. Each of these samples was then milled during 6, 8 or 10 minutes just before the flotation experiments in order to avoid the gibbsite surface oxidation. The samples were milled in rod mill under 50 % of solids. Different milling times were used in order to investigate the particle size influence on the gibbsite flotation performance. For the milling time of 10 minutes 99% of the particles were lesser than 0,119 mm.

The size distribution was determined after each milling time by screening in 0.354, 0.251, 0.178, 0.125, 0.089, 0.066 and 0.044 mm sieves.

The mineralogical composition was determined by means of X-ray diffraction (XRD) and binocular loupe analysis. X-ray fluorescence analysis (XRF) was used to determine the content of silica (SiO_2), alumina (Al_2O_3) and iron oxide (Fe_2O_3). The XRD and XRF analysis were carried out in the Curimbaba Company characterization Laboratory. In addition, the sample Liberation Level was evaluated by using the grain-counting technique.

2.2 Flotation Experiments

Direct flotation experiments (flotation cell: CFB - 1000N, Engendrar) were carried out with a rougher and two cleaner steps in order to investigate the effect of collector dosage (264, 395 and 528 g/t), flotation pH (8.5, 9.5 and 10.0) and milling time (6, 8 and 10 min) on the gibbsite flotation performance. Conditioning was performed under 60% of solids and flotation under 40%. All experiments were conducted with distilled water.

Hydroxamate (AERO 6493, Cytec) was used as gibbsite collector (2 min of conditioning), sodium silicate (Akzo Nobel) as silicates depressant (2 min conditioning) and corn starch as iron-bearing minerals depressant (5 min of conditioning in the rougher step and 2 min in the cleaner steps). The depressant dosages were kept constant along the flotation experiments. It was used 400 g/t in the rougher step and 200 g/t in the cleaner steps for all experiments. The collector dosage was performed only in the rougher step.

3 RESULTS AND DISCUSSION

3.1 Characterization

Table 1 shows the chemical analysis (XRF) for different particle sizes. While the Al_2O_3 content is higher in the rough fraction, SiO_2 and Fe_2O_3 contents increased with decreasing particle size. It indicates that the impurities are concentrated in the finer sizes.

The head sample grades are 84% of Al_2O_3, 6.6% of SiO_2 and 7.6% of Fe_2O_3. While the Al_2O_3 grade is considered adequate for the ceramics industry, the Fe_2O_3 is higher than the market specification (2.5%). Accordingly, the bauxite ore must be submitted to concentration operations in order to remove iron-bearing minerals before feeding the ceramics production.

Table 1. XRF Analysis in different size ranges

Size (mm)	Retained mass (%)	Content %		
		Al_2O_3	SiO_2	Fe_2O_3
+1.180	20.80	89.49	4.66	4.85
−1.180 +0.850	10.90	86.80	5.88	6.08
−0.850 +0.600	11.10	84.80	6.74	7.09
−0.600 +0.425	9.90	84.20	6.85	7.61
−0.425 +0.300	8.10	83.60	7.07	7.96
−0.300 +0.212	6.30	83.50	6.93	8.16
−0.212 +0.150	7.00	83.40	6.83	8.31
−0.150 +0.106	6.20	82.30	7.28	8.87
−0.106 +0.075	6.70	80.40	8.15	9.84
−0.075 +0.053	5.30	79.10	8.49	10.80
−0.053 +0.038	5.50	78.80	8.57	11.00
−0.038	2.30	78.70	8.50	11.20
Total calculated	100	84.44	6.63	7.59
Total analyzed	-	84.30	6.66	7.66

According to the XRD analysis the bauxite is composed mostly of gibbsite. Aluminum is also present in clay minerals, especially kaolinite, which represents around 29% of the sample. The iron-bearing minerals are goethite, limonite $(Fe(OH)_3.nH_2O)$ and lepidocrocite $(\gamma\text{-}FeO(OH))$, as well as traces of limonitic aggregates (<0.96%). Hematite (Fe_2O_3), magnetite (Fe_3O_4) and ilmenite $(FeTiO_3)$ are present in very low amounts (<0.02%) too.

As observed in the photomicrographs of Figure 1, aluminum-bearing minerals are present as: (A) small tabular clay particles with iron hydroxide impregnation/association; (B) Prismatic gibbsite crystals, sometimes containing inclusions or associations of iron oxides and hydroxides such as clays; (C) Slim granular clay aggregates. It can be observed in Figure 1d that lepidocrocite grains are interspersed with goethite grains and limonitic aggregates, causing pigmentation in this mineral.

According to the liberation analysis the gibbsite particles in the size range of -0.105+0.074 mm are 85.62% liberated with a Beribé Coefficient of 91.15%. The Beribé coefficient is an average percent of the interest mineral area (aluminum oxides and hydroxides). There is less literature information about the ideal gibbsite liberation for flotation. Marino (2012) flotation work, for instance, was accomplished with a more than 50% liberated gibbsitic bauxite. Therefore, the obtained value of 85,62% of liberation was considered satisfactory.

Finally, the granulometric distribution variation with the milling time is shown in Table 2. It can be observed that the slime amount significantly increases by increasing the milling time. The content of particles smaller than 44 μm increased from 6.1% to 11.8% when the milling time was raised from 6 to 10 min.
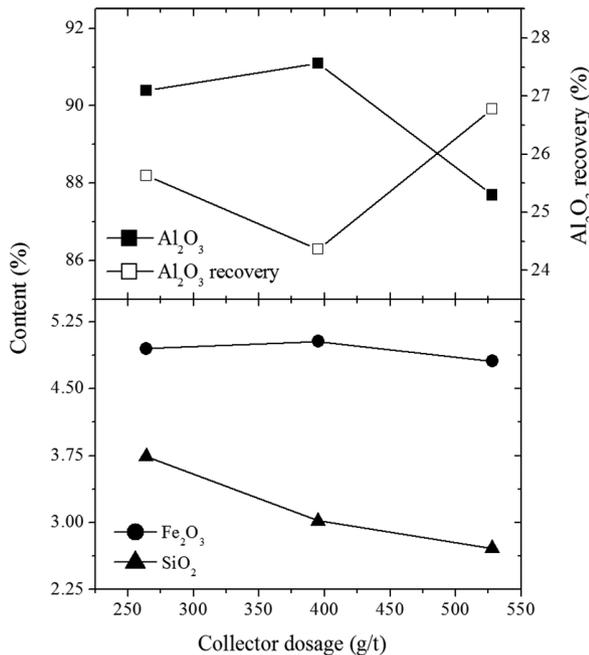
3.2 Flotation

The direct gibbsite flotation results will be analyzed in terms of the Al_2O_3, Fe_2O_3 and SiO_2 contents (%) in the concentrates, besides the Al_2O_3 recovery (%). The influence of the collector dosage on the flotation performance is shown in Table 3 and Figure 2. Regardless of the collector dosage, the direct gibbsite flotation resulted in a Fe_2O_3 reduction from 7.66% to 4.81-5.03%. In addition, the Fe_2O_3 decrease

Table 2. Size distribution after different milling times

Size (mm)	Accumulated passing (%) / 6 min	Accumulated passing (%) / 8 min	Accumulated passing (%) /10 min
0.354	100.0	100.0	100.0
0.251	94.1	96.1	99.1
0.178	76.2	80.1	83.4
0.125	59.6	66.7	72.3
0.089	45.6	54.5	63.5
0.066	21.9	31.3	43.2
0.044	6.1	8.4	11.8
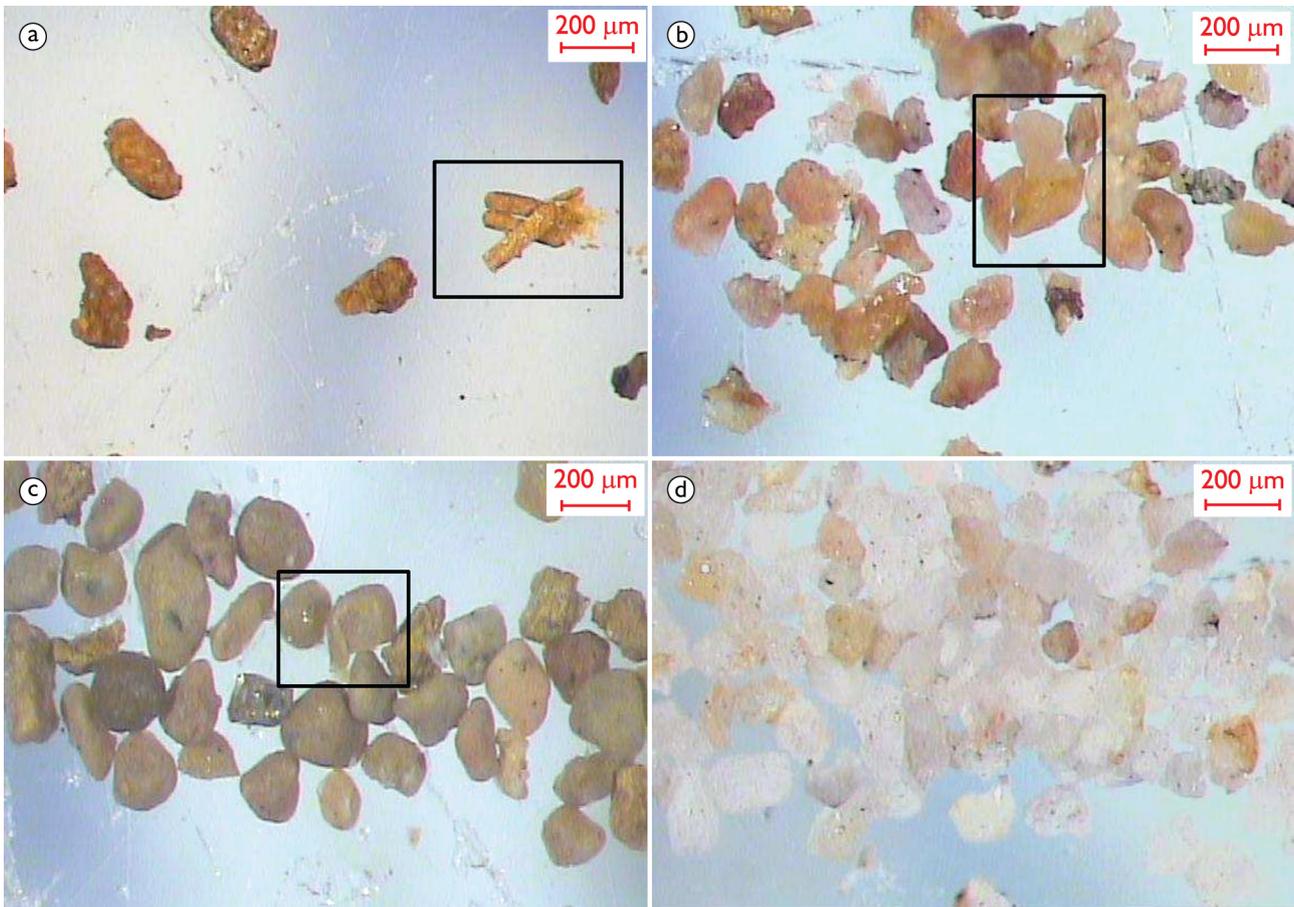
Figure 1. Bauxite particle photomicrographs: A and B -particle size + 0.106 mm, 50x magnification; C -particle size + 0.154 mm, 50x magnification.

Figure 2. Influence of hydroxamate dosage on the gibbsite flotation performance.

Table 3. Influence of the hydroxamate dosage on the gibbsite flotation performance (pH = 9.5, starch and sodium silicate dosages in the rougher step = 400 g/t, starch and sodium silicate dosages in the cleaner steps = 200 g/t, milling time = 8 min)

Collector dosage (g/t)	Concentrate mass (g)	Al_2O_3 (%)	Fe_2O_3 (%)	SiO_2 (%)	Al_2O_3 recovery (%)
264	95.53	90.40	4.95	3.74	25.63
395	89.61	91.10	5.03	3.02	24.35
528	97.94	87.70	4.81	2.71	26.78

was followed by the SiO_2 content decrease due to the fact that iron is present mainly as silicates.

Comparing the different hydroxamate dosages, the SiO_2 content presented a reduction from 4.95% to 2.71% when the collector dosage increased from 264 g/t to 528 g/t. Nevertheless, the hydroxamate dosage seems not to have influenced the Fe_2O_3 grade. Regarding the Al_2O_3 content, the highest value was obtained with the collector dosage of 395 g/t (Table 3 and Figure 2). Therefore, this dosage was selected to be applied in the experiments conducted under different pH values and milling times.

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The pH decrease from 9.5 to 8.5, as illustrated in Table 4 and Figure 3, resulted in the Fe_2O_3 content enhancement from 5.03% to 5.38%, followed by a reduction in the Al_2O_3 content from 91.10% to 89.60% and consequent Al_2O_3 distribution increase from 24.36% to 25.63%. The flotation performance reduction can be explained by a reduction in the hydroxamate collector activity at pH 8.5. As the hydroxamic acid pKa (solubility product) is approximately 9.0, there is a predominance of protonated species [20] below this pH. Considering that the collector activity of the protonated species (R-CONHOH) is lower than that of the hydroxamic ions (R-CONHO⁻), the hydroxamic acid concentration increase, instead of the hydroxamic ion, could have reduced the gibbsite collection and consequent flotation.

In addition, an increase in the flotation pH from 9.5 to 10.5 resulted in a significant Al_2O_3 content reduction (91.10% to 81.00%). Under this condition there was no iron-bearing minerals removal from the bauxite since the SiO_2 and Fe_2O_3 contents (9.75% and 7.72%, respectively) are higher than those of the feed (6.66% and 7.66%, respectively). The reduced flotation performance observed at pH 10.5 could be related to the contribution of electrostatic forces in the interaction between collector molecules and mineral surface.

The collector adsorption on the solid/liquid mineral interface is affected by parameters such as electrostatic forces and chemical interaction [21,22]. Regarding the hydroxamate adsorption on aluminum-bearing minerals, while chemical interaction seems to be the main driving force for the collector interaction with the diaspore surface [12,13], no literature work has reported its interaction on gibbsite particles. On the other hand, the electrostatic contribution to the collector adsorption depends on the mineral zeta potential (Ψ) and the collector charge. As the gibbsite isoelectric point ($\Psi = 0$) is

10, its surface presents an excess of negative charges ($\Psi < 0$) at pH 10.5 [23,24]. Considering that the polar hydroxamic group also has a negative charge (CONHO⁻), there can be an electrostatic repulsion between the gibbsite surface and collector molecules. This effect may have contributed to the decrease in the gibbsite flotation performance at pH 10.5. In order to confirm this possibility microflotation experiments with pure gibbsite must be carried out.

The third parameter evaluated was the milling time (6, 8, and 10 min), which is directly related to the flotation feed particle size. Table 5 and Figure 4 illustrate that the milling time reduction from 8 to 6 min resulted in lower flotation performance since the Al_2O_3 content decreased from 91.10% to 88.9% and the Fe_2O_3 content increased from 5.03% to 6.75%. This outcome can be explained by a possible decrease in the liberation level as the sample amount in the particle size range corresponding to 85.62% of liberation reduces from 85.5% to 67.5%, as observed in the size distribution after 6 min of milling, as shown in Table 2.

Similarly, the milling time increase from 8 to 10 min impaired the gibbsite flotation performance since the
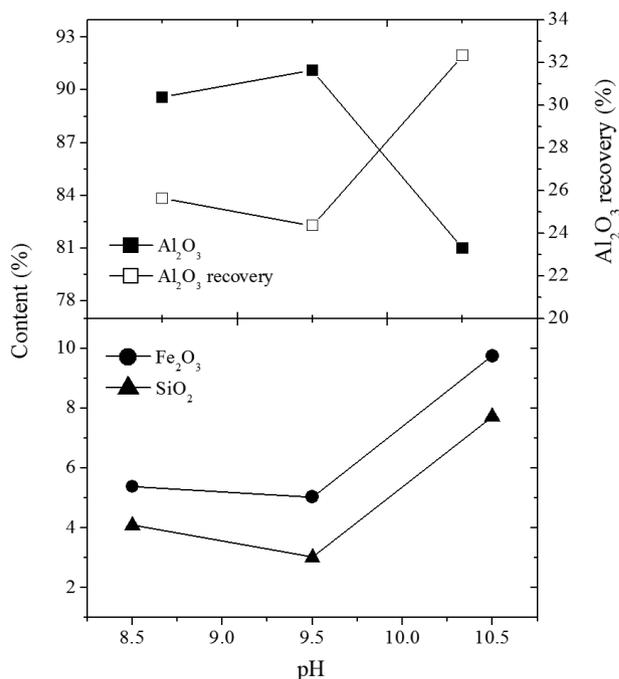
Table 4. Influence of pH on the gibbsite flotation performance (hydroxamate dosage = 395 g/t, starch and sodium silicate dosages in the rougher step = 400 g/t, starch and sodium silicate dosages in the cleaner steps = 200 g/t, milling time = 8 min)

| pH	Concentrate mass (g)	Al_2O_3 (%)	Fe_2O_3 (%)	SiO_2 (%)	Al_2O_3 recovery(%)
8.5	95.70	89.60	5.38	4.09	25.63
9.5	89.61	91.10	5.03	3.02	24.36
10.5	133.18	81.00	9.75	7.72	32.32

Figure 3. Influence of pH on the gibbsite flotation.

Table 5. Influence of the milling time on the gibbsite flotation performance (pH = 9.5, hydroxamate dosage = 395 g/t, starch and sodium silicate dosages in the rougher step = 400 g/t, starch and sodium silicate dosages in the cleaner steps = 200 g/t)

Milling time(min)	Concentrate mass (g)	Al_2O_3 (%)	Fe_2O_3 (%)	SiO_2 (%)	Al_2O_3 (%) distribution
6	138.76	88.90	6.75	3.33	36.71
8	89.61	91.10	5.03	3.02	24.36
10	90.30	90.40	5.64	3.09	24.40

Al_2O_3 content decreased and the Fe_2O_3 grade increased. By increasing the milling time, the fine particles (< 0.066 mm) amount increased from 6.1% to 11.8%, as shown in Table 2. This result indicates that the gibbsite direct flotation could be sensitive to the presence of slime. Many literature works have already reported the harmful effect of fine particles in the reverse diaspore flotation [8-11,25].
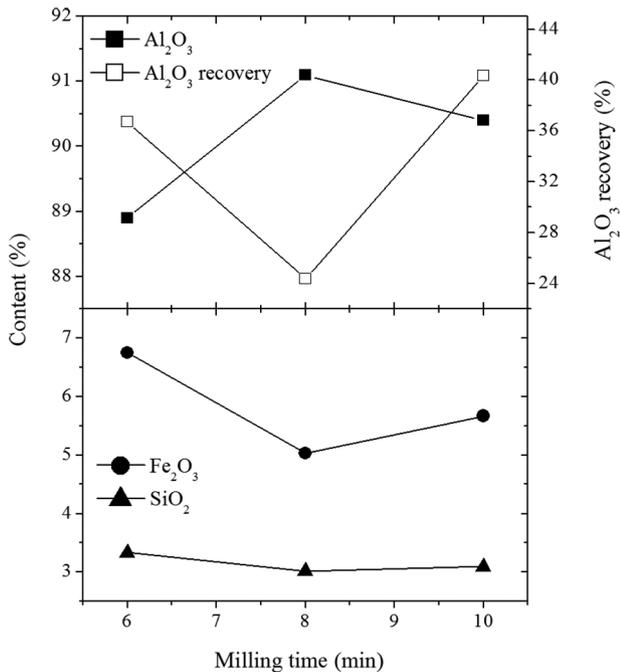
Figure 4. Influence of the milling time on the gibbsite flotation performance.

4 CONCLUSION

It was possible to reduce the bauxite Fe_2O_3 content from 7.66% to 4.81-5.03% with reverse gibbsite cationic flotation. However the market specification (2.5%) has not yet been reached. In addition, all Al2O3 recoveries are lesser than what is considered satisfactory for an industrial plant. This way it is necessary to improve the concentration strategy by introducing cleaner steps, varying the hydrodynamic conditions and adding other concentration techniques such as magnetic separation.

The most adequate flotation pH for direct gibbsite concentration is 9.5. The flotation performance decreases by decreasing the pH to 8.5 or increasing the pH to 10.5. The first effect could be related to the decrease in the hydroxamic ions availability while the second may be due to the contribution of electrostatic repulsion forces between gibbsite surface and collector molecules. Finally, the investigation of the milling time effect on gibbsite direct flotation indicates that the concentration can be harmed by the presence of slimes.

Acknowledgements

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