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Review Article

Reduction of Fe_2O_3 content of foyaite by flotation and magnetic separation for ceramics production

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ABSTRACT

Feldspars are used as a fluxing agent in ceramics and glass production. However, the presence of iron-bearing minerals reduces whiteness of the ceramics. The objective of this work was to decrease the Fe_2O_3 content of a foyaite to <1% by flotation and magnetic separation, individually or combined, to make the sample suitable for white ceramics manufacture. The sample was prepared regarding its particle size distribution, characterised, and submitted to concentration experiments. Flotation was arried out to investigate promising anionic collectors and the most suitable conditions of dosage and pH. Wet magnetic separation was used in several stages, increasing the magnetic field. Lastly, magnetic separation was applied to the concentrate of the most efficient flotation test. Technological characterisation showed that the foyaite sample displays 3.2% Fe_2O_3 from pyroxene/amphibole. The most efficient flotation condition was the reverse flotation of iron-bearing minerals with alkyl sulphate as collector under 400 $g\,t^{-1}$ at pH 4. The desired specification was reached after three cleaner stages, yielding a concentrate with 0.92% Fe_2O_3 and 74.6 wt.% recovery. By magnetic separation, the Fe_2O_3 content was reduced to 0.8% with 70.6 wt.% recovery, after five stages of concentration with the magnetic field varying from 0.78 to 1.12 T. The most efficient combined circuit comprised three stages of flotation followed by one stage of magnetic separation at 0.78 T. The concentrate presented 0.55% Fe_2O_3 and 71.2 wt.% recovery.

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1. Introduction

Feldspars are sodium, potassium, and calcium aluminosilicates that constitute 60% of the Earth's crust [1]. Commercial feldspars are albite ($NaAlSi_3O_8$), orthoclase ($KAlSi_3O_8$), microcline ($KAlSi_3O_8$), and anorthite ($CaAl_2Si_2O_8$) [2]. They are applied as a fluxing agent, lowering the melting temperature of the mixture, in ceramics and glass production. However, the presence of iron and titanium-bearing minerals is responsible for a dark colour in fired products [1,2].

In Italy, Turkey, and China, flotation is used to concentrate feldspars [3–6]. Traditionally, reverse flotation of mica and other silicates takes place first using amines as collectors at pH 2.5–3.5, then iron and titanium-bearing minerals are floated with anionic collectors at pH 3–4 [2–4]. The anionic collectors that have been used are fatty acids, alkyl sulphonates, sulphates, succinamates, sarcosines, and hydroxamates [7–12].

Karagüzel [11] obtained an albite concentrate assaying 0.33% $Fe_2O_3+TiO_2$ and 11.07% Na_2O+K_2O from a slime feed consisting of 1.06% $Fe_2O_3+TiO_2$ and 10.36% Na_2O+K_2O by using dissolved air flotation and fatty acid as collector. Abdel-Khalek et al. [7], by using a mixture of dodecyl benzene sulphonic acid, rice bran oil, and kerosene, were able to reduce the Fe_2O_3 content of an Egyptian feldspar from 0.53 to 0.1%.

It is also possible to directly float feldspars from silicates like quartz using cationic collectors if the feldspars are activated by hydrofluoric acid (HF). However, environmental limitations to the use of HF have motivated research about non-fluoride processes to separate feldspars and quartz [6,13,14].

The combination of flotation and magnetic separation has been used to remove iron/titanium-bearing particles from feldspars [15,16]. Burat et al. [15] found that flotation followed by dry magnetic separation was suitable to reduce the Fe_2O_3 content of a nepheline syenite ore from 0.19 to 0.09%. Silva et al. [16] used magnetic separation after flotation to reduce the Fe_2O_3 grade of a Brazilian foyaite from 3.14 to 0.61–0.58%. These works, however, did not evaluate the influence of concentration parameters that could improve the concentrate

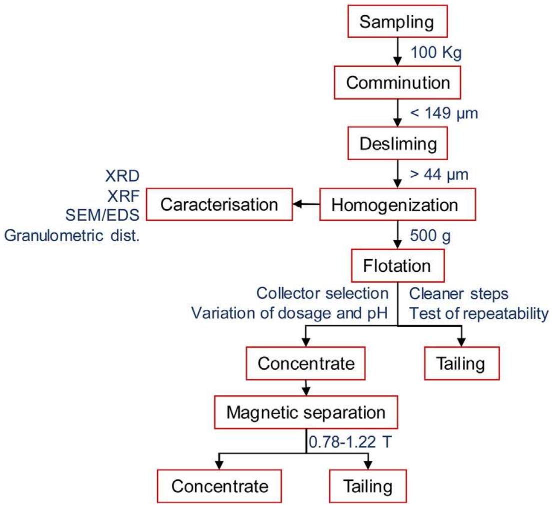

Fig. 1 – Flowchart of experiments.

quality, such as dosage of reagents or pH on flotation and magnetic field on magnetic separation.

In Brazil, only high-grade feldspars are applied in ceramics and glass production since concentration is not a common practice [17]. However, the exhaustion of deposits containing low iron/titanium content demands the development of concentration routes. The potassic rock deposits in Poços de Caldas, for example, display Fe_2O_3 content ranging from 2 to 6% [18]. The Poços de Caldas alkaline massif (800 km^2) comprises foyaite (plutonic) and tinguaite (subvolcanic) with the presence of phonolite, leucitite, alkaline lavas, tuff, agglomerates, and volcanic gaps. Foyaite is a textural variety of nepheline syenite that occurs in the Poços de Caldas massif. The predominant minerals are anorthoclase, nepheline, aegirine, and sanidine, and as accessories there are mainly found magnetite, fluorite, zirconite, and titanite [18,19].

The objective of this work was to use flotation and magnetic separation, individually and combined, to reduce the Fe_2O_3 content of a foyaite sample from Poços de Caldas to <1% [20]. The tested techniques applied separately or combined were able to meet the desired specification.

2. Materials and methods

The Mineração Curimbaba company supplied 100 kg (<149 µm) of foyaite that was collected to ensure it represents the currently mined area. The sample was prepared, characterised, and submitted to concentration experiments (Fig. 1).

2.1. *Sample preparation*

The material was homogenised and split in 2 kg aliquots for the removal of slimes (<44 µm) by siphoning. Representative aliquots were mixed with water (25% solids), stirred (23.3 s^{-1}), and the pH was adjusted to 10.5 with sodium hydroxide (NaOH) 10 wt.% solution. Then, the pulp was left to sedimentation and siphoned. The sedimentation times of 1, 2, and 5 min were tested to determine the period enough to guarantee that 90% of the slimes had been removed. This time (2 min) was applied to siphon the whole sample.

After that, the material was homogenised and split in 400 g aliquots to feed the concentration experiments. A head sample was taken for characterisation.

2.2. *Characterisation*

The sample chemical composition was assessed by quantitative XRF analysis (Zetium spectrometer, PANalytical) in fused beads. Loss on ignition (LOI) was assayed at 1020 °C for 2 h and represents the weight loss at a selected temperature. The volatile materials lost usually consist of combined water from minerals (hydrates and hydroxy-compounds) and carbon dioxide from carbonates. Mineralogical composition was evaluated by powder X-ray diffraction (XRD — X'Pert PRO, PANalytical with X'Celerator detector) on ground samples with backload hand mount to minimise preferential orientation. The identification of the crystalline phases was performed by comparing the XRD pattern to the databases of the International Centre for Diffraction Data; semiquantitative mineralogical composition was assessed by Referent Intensity Ratio (RIR) method combined with the chemical composition. Scanning Electron Microscopy coupled with Energy Dispersive X-Ray Spectroscopy (SEM/EDS) were carried out in selected fractions for evaluating the particle composition. The granulometric distribution was determined by wet sieving at 600, 500, 300, 212, 150, 106, 75, and 45 µm.

2.3. *Flotation*

Flotation tests comprised reverse flotation (flotation cell CFB-1000 EEPN from CDC) of iron-bearing minerals with anionic collectors since those minerals are present in lower content compared to feldspars [2]. The flotation experiments started by mixing 400 g of foyaite sample with 500 ml of distilled water (50 wt.%). The pH was adjusted with sulphuric acid (H_2SO_4 10 wt.%), and conditioning with collector took place for 4 min and 2 min in the rougher and cleaner stages, respectively. After that, conditioning with pine oil (frother) was conducted for 2 min at 147 $g\,t^{-1}$ and 73.5 $g\,t^{-1}$ in the rougher and cleaner stages, respectively.

The first set of flotation experiments was conducted with different anionic collectors comprising the fatty acids RADIACID (Oleon), LUPROMIN (BASF), FLOTIGAM 5806 (Clariant), KORTACID 0810 (Oleon), SYLFAT FA-1 SPECIAL (Arizona Chemical), the alkyl sulphate MDB 908 (AkzoNobel), and the sulphonate MDB 1425 (AkzoNobel). The experiments were performed with the rougher dosage of 400 $g\,t^{-1}$ at pH 4. Two cleaner stages were carried out using half of the collector dosage applied at the rougher stage. The flotation tests displaying higher Fe_2O_3 reduction with higher concentrate mass recovery were considered to yield the most suitable concentration performance.

Collectors that yielded the most promising results in the first set of flotation experiments were applied in the second set that was carried out under different rougher dosages (200, 400, and 600 $g\,t^{-1}$). Then, the most suitable dosage was used in experiments at pH 3–5.

Table 1 – Foyaite chemical composition determined by XRF.

Species	SiO_2	TiO_2	Fe_2O_3	K_2O	Al_2O_3	CaO	MnO	MgO	Na_2O	LOI
Content (%)	53.9	0.37	3.47	8.45	21.5	0.92	0.19	0.27	8.54	1.58
STD	1.20	0.02	0.26	0.04	0.35	0.01	0.01	0.05	0.17	0.06

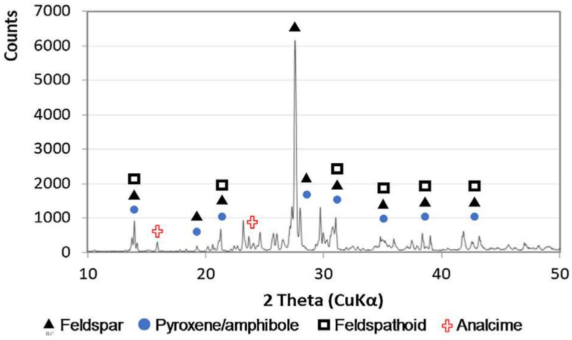

Fig. 2 – X-ray diffractogram of the foyaite sample.

To evaluate the repeatability of the flotation tests, the experiment using alkyl sulphate as collector at 400 g t^{-1} and pH 4 was repeated four times. Finally, the influence of the number of cleaner stages was evaluated by applying the latter condition in two experiments carried out with four cleaner stages.

2.4. *Magnetic separation*

The foyaite samples were submitted to magnetic separation in a Wet High Intensity Magnetic Separator (WHIMS, Inbras-Eriez), using the magnetic fields of 0.78, 0.93, 1.07, 1.14, 12.5, and 1.22 T. Approximately 50 g of material was added to the magnetic separator in each pass, until all the required material was processed. The magnetic product was separated for chemical analysis, and the non-magnetic product passed through again at the higher field. Magnetic separation tests were applied to the head sample (without previous flotation) and to the flotation concentrates obtained after two and four cleaner stages.

3. Results and discussion

3.1. *Characterisation*

The foyaite chemical composition is shown in Table 1, and the mineralogical composition is shown in Fig. 2.

The sample comprises different aluminium silicates as suggested by the high grades of SiO_2 (53.9%) and Al_2O_3 (21.5%), mostly sodium and potassium feldspars [(Na, K), Al, Si, O)] (microcline/orthoclase and albite/anorthite), and feldspathoids (nepheline, sodalite [Na, K, Al, Si, O], confirmed by XRD and associated to the content of K_2O (8.5%) and Na_2O (8.3%). The content of feldspar and feldspathoid is around 85%, estimated by semiquantitative analysis from XRD combined with the chemical composition; as minor minerals, pyroxene-amphibole (7–10%, mainly aegirine) and analcime (5–7%) were identified.

Considering the desired application of the studied material, the chemical analysis indicates that concentration is indeed required because the foyaite does not meet the company specification for white ceramics ($Fe_2O_3 < 1\%$) since the Fe_2O_3 content is 3.47% (Table 1). The loss on ignition was 1.58% and can be accounted for mainly by analcime and by amphibole or minerals that could not be identified by XRD due to very low concentrations.

In Fig. 3, the general aspect of the foyaite sample highlighting the iron occurrence (EDS analysis) in pyroxene/amphibole minerals (in red colour) is shown. Details of some particles were taken to evaluate the content of Fe in minerals; semi-quantitative analysis (Table 2) demonstrates that Fe occurs in different grades with a high variability (from 0.8 to 39%) in bearing minerals. Rare earth elements were also identified.

According to the granulometric distribution (Fig. 4), the foyaite sample presents 90% of the material below 145 μm and 100% greater than 30 μm, which is suitable to allow concentration by flotation or magnetic separation.

3.2. *Flotation*

In Fig. 5, the flotation performance of experiments conducted with different commercial reagents is illustrated. Since the flotation result is represented by concentrate mass recovery (wt.%) as a function of Fe_2O_3 content (Fig. 5), the most effective collectors are indicated by the red circle in the graph.

Alkyl sulphate (MDB 908) and sulphonate (MDB 1425) yielded the most satisfactory flotation performance because the Fe_2O_3 content of the concentrate is close to the specification (1%) and the recovery is greater than 65 wt.%. Therefore, these reagents were selected to be applied in additional experiments for evaluating the influence of collector dosage and pH.

The RADIACID fatty acid was also selected because, despite the low recovery, it is the only collector capable of reducing the Fe_2O_3 content to <1%. The other fatty acids can be considered inadequate, as the Fe_2O_3 content of the concentrates is much higher than the specification (Fig. 5). Although the flotation of iron-bearing minerals with fatty acids at low pH was reported in the literature [21], the lower flotation performance of those reagents (Fig. 5) could be related to the surfactant speciation. At pH < pKa, the collector is predominantly in the acidic form (RCOOH) and should not adsorb on iron-bearing minerals if we consider this interaction to be physical.

In Fig. 6, the flotation outcome of experiments accomplished at different dosages and values of pH is presented. The dosage of 400 g t^{-1} is the most efficient for the objective of reducing the Fe_2O_3 content using alkyl sulphate (1.18%) and fatty acid (0.82%). At a lower dosage (200 g t^{-1}), the amount of collector is likely insufficient to remove all iron-bearing

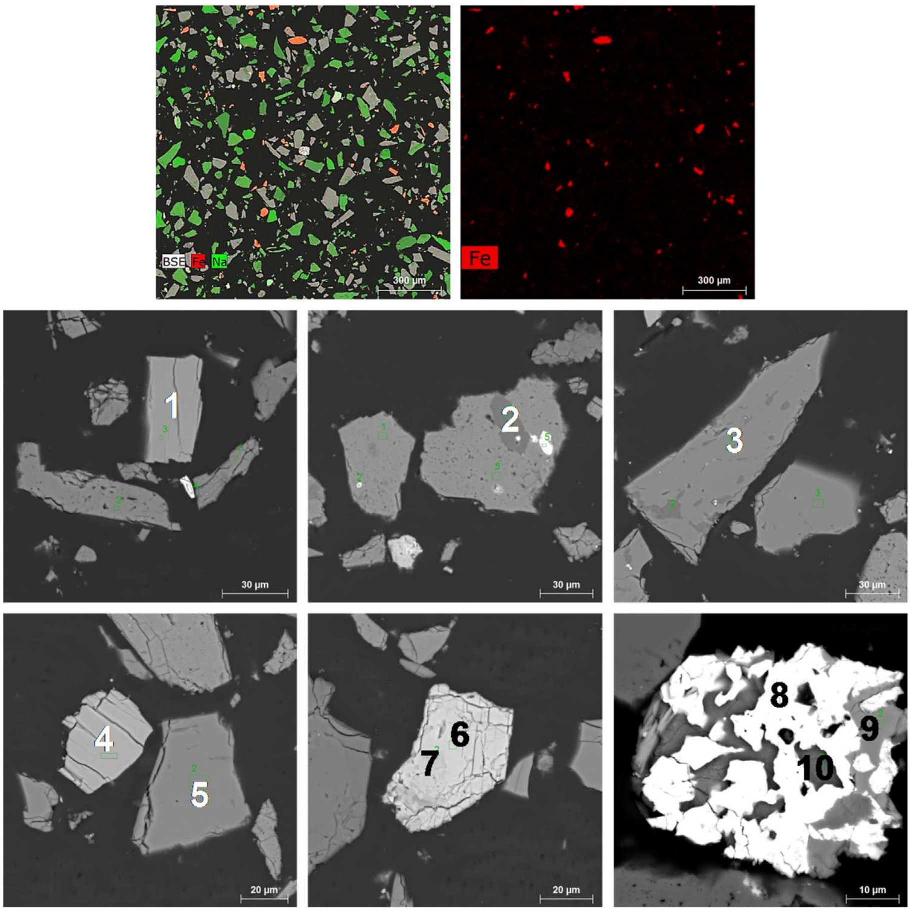

Fig. 3 – SEM images indicating spots of EDS analysis.

Table 2 – Semiquantitative analysis by SEM-EDS.

Phase	O	Na	Si	K	Ca	Fe	Al	Cl		
1		13.1	39.6		7.7	39.5				
2	41.7		31.7	13.2		1.2	12.2			
3	39.8	13.6	19.2	6.6		1.1	19.7			
4	37.3	10.3	25.8		4.9	20.8		0.9		
5	36.0	20.9	16.2			0.8	18.4	7.6		
	O	Na	Si	K	Ca	Fe	Mn	Mg	Ti	Zr
6	28.3	9.8	15.9		23.0		2.0		7.2	13.9
7	35.4	3.9	16.6	1.9	1.3	4.3	23.6	0.8	7.6	2.0
	O	F	Ca	Sr	La	Ce	Pr	Nd		
8	28.5		3.7	25.0	14.0	21.4	2.8	4.6		
9		47.2	52.8							
10	50.6		49.4							

particles, while at 600 g t^{-1} the flotation froth could be entraining feldspar particles, which causes an increase in the Fe_2O_3 grade. In contrast, the Fe_2O_3 grade decreases with the increase of the sulphonate dosage from 200 to 600 g t^{-1}, indicating that greater sulphonate dosages could yield cleaner concentrates. However, the recovery would be reduced below 60% (Fig. 6).

While the fatty acid leads to the highest removal of iron-bearing minerals, the mass recovery is very low (<30%). The recoveries corresponding to alkyl sulphate and sulphonate are very similar, and alkyl sulphate leads to the highest removal of Fe_2O_3. Therefore, the latter reagent can be considered the most effective collector (Fig. 6).

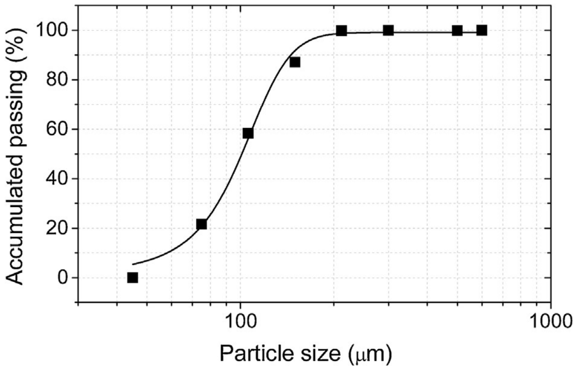

Fig. 4 – Granulometric distribution of the foyaite sample.

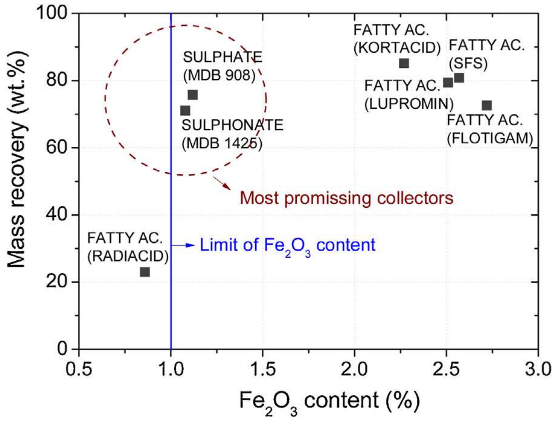

Fig. 5 – Mass recovery (wt.%) versus Fe_2O_3 content for flotation tests under: rougher collector dosage = 400 g t^{-1}, cleaner collector dosage = 200 g t^{-1}, and pH = 4.

Regarding the effect of pH, at pH 3 none of the reagents was successful in removing iron-bearing minerals from the foyaite, since the Fe_2O_3 content of all concentrates is higher than 2.5%. When the pH increases from 3 to 4, the performance of all tested reagents enhances, as the reduction of Fe_2O_3 is more prominent. The decrease in Fe_2O_3 content is, as is likely to happen, followed by a decrease in mass recovery. However, this value is much smaller for the fatty acid (<25 wt.%) than for alkyl sulphate or sulphonate (70 and 80 wt.%, respectively) (Fig. 6).

The increase of pH from 4 to 5 causes a decrease in the fatty acid performance as the Fe_2O_3 grade increases to 2.92%. In addition, the Fe_2O_3 grade slightly reduces for alkyl sulphate and sulphonate. However, there is a significant mass recovery decrease, which makes pH 5 not optimal for the foyaite concentration (Fig. 6).

The influence of pH on flotation performance agrees with results reported in the literature [8,22]. Bayraktar et al. [8], for instance, found that pH 4 was the most efficient condition (pH ranging from 3 to 7) to increase the whiteness of a Na-feldspar using sulphonate (750 g t^{-1}) as collector. El-Rehiem and El-Rahman [22] reported a reduction of TiO_2+Fe_2O_3 content from pH 4 to 6 under the same conditions of collector and dosage. However, Gulsoy et al. [23] observed that the TiO_2+Fe_2O_3 content decreased by increasing pH from 4 to 5.5 and kept constant up to pH 7.5 in the flotation of Na-feldspar with sodium oleate (2000 g t^{-1}), which contrasts with the findings of our study (Fig. 6).

The flotation condition that resulted in the most satisfactory performance is using alkyl sulphate as collector at 400 g t^{-1} and pH 4. The concentrate shows 1.18% Fe_2O_3 and 76.5 wt.% mass recovery. This experiment was repeated yielding a concentrate with 1.19 ± 0.04% Fe_2O_3 and 78 ± 3.81% recovery, indicating that the repeatability of the flotation experiments was satisfactory.

3.3. *Flotation and magnetic separation*

In Fig. 7, the flotation and magnetic separation, individually applied, are compared (A), and the performance of a combined concentration route in which magnetic separation was fed with the concentrates of the second and fourth

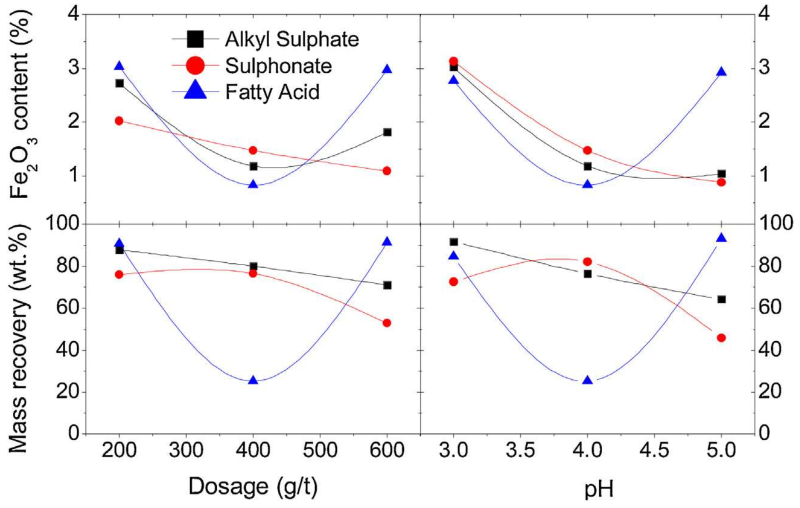

Fig. 6 – Fe_2O_3 content and mass recovery (wt.%) under different collector dosages (at pH 4) and values of pH (at 400 g t^{-1}).

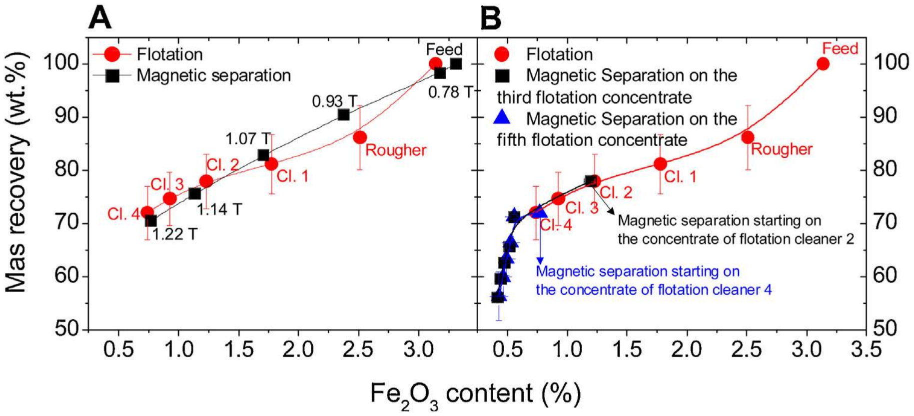

Fig. 7 – Mass recovery (wt.%) versus Fe_2O_3 content for (A) flotation with sulphonate, under 400 g t^{-1} at pH 4 after four cleaner stages compared to magnetic separation under different magnetic fields and (B) magnetic separation under different magnetic fields applied to the flotation concentrates.

flotation cleaners is presented (B). In flotation, the most significant Fe_2O_3 reduction takes place in the rougher concentrate (from 2.52 to 1.78%). The amount of iron-bearing minerals continues reducing until the fourth cleaner stage in which the Fe_2O_3 content is 0.74%. However, the third cleaner is enough to achieve the desired specification (<1%), since the Fe_2O_3 content is 0.92%, with a suitable mass recovery, 74.6 wt.% (Fig. 7A).

Regarding the magnetic separation, there is a linear variation of recovery with Fe_2O_3 content until the Fe_2O_3 grade of 0.8% with 70.6% recovery in the final concentrate (Fig. 7A). Similarly, El-Rehiem and El-Rahman [22] reported that the removal of colouring material increased by increasing the magnetic field intensity using dry magnetic separation; the Fe_2O_3 content decreased from 1.85 to 0.2% while the TiO_2 decreased from 0.35 to 0.03%.

It is evident that both techniques, flotation and magnetic separation, show similar results regarding reducing the Fe_2O_3 content of the foyaite sample (Fig. 7A). Both, when independently applied, can reach the desired specification for white ceramics production (<1% Fe_2O_3) with mass recovery of around 70 wt.%. In addition, the Fe_2O_3 grade keeps reducing along the cleaning stages for both concentration techniques, indicating that a higher number of cleaner stages could yield concentrates that fit the specification for glass production (<0.1% Fe_2O_3).

Regarding the combined route (Fig. 7B), the first step of magnetic separation under the lowest magnetic field (0.78 T) yields the greatest reduction of Fe_2O_3 content, compared to the subsequent steps, when this technique was applied to both the second (from 0.77 to 0.56%) and fourth (from 1.19 to 0.55%) cleaner concentrates.

It must be highlighted that the concentrate of the first step of magnetic separation displays the same characteristics (0.55–0.56% of Fe_2O_3 and 71.2–71.3 wt.% of recovery) regardless of the feed (second or fourth cleaner concentrate). Therefore, it is surely economically profitable to not perform flotation stages after the second cleaner in case of using a combined concentration route including flotation and magnetic separation.

The subsequent four steps of magnetic separation (magnetic fields of 0.93, 1.07, 1.14, and 1.22 T) yield a final concentrate with $\tilde{0}.4$% Fe_2O_3 and 56 wt.% mass recovery, regardless of the feed. Since the Fe_2O_3 reduction is low (0.55–0.4%) followed by a significant reduction in mass recovery (71–56%), the second and subsequent steps of magnetic separation do not improve the concentration result (Fig. 7B). Therefore, the most satisfactory combined route is flotation comprising rougher and two cleaner stages followed by one step of magnetic separation at low field (0.78 T).

4. Conclusions

Flotation and magnetic separation, individually applied or combined, were able to make the foyaite suitable for white ceramics production ($Fe_2O_3 < 1$%).

Fatty acid, sulphonate, and alkyl sulphate were suitable reagents for being used as collectors to the direct flotation of iron-bearing minerals. The most suitable flotation condition was using alkyl sulphate as collector under 400 g t^{-1} at pH 4. When it consists of one rougher followed by three cleaner stages, the final concentrate presented Fe_2O_3 content of 0.92% with a reasonable recovery (74.6 wt.%).

Magnetic separation individually applied reduced the Fe_2O_3 content to 0.8% with 70.6 wt.% recovery, after five stages of concentration with magnetic field varying from 0.78 to 1.12 T.

The combined route composed of flotation followed by magnetic separation yielded a concentrate with 0.55% Fe_2O_3 and 71.2 wt.% recovery, after the first stage of magnetic separation (0.78 T), regardless of the magnetic separation being applied to the second or fourth cleaner concentrates. So, the most satisfactory combined concentration route consisted of three stages of flotation (rougher and two cleaner stages) followed by one stage of magnetic separation.

Therefore, the studied foyaite, that has been applied in the construction industry, might be used for white ceramics production, after one of the proposed concentration routes is implemented to the mineral processing of this rock.

Conflicts of interest

The authors declare no conflicts of interest.

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