TECHNOLOGICAL CHARACTERIZATION AND FLOTATION STUDIES ON THE IRECÊ (BAHIA, BRAZIL) PRIMARY PHOSPHATE ORE

Monte, M.B.M[1], Alcover Neto, A.[1], Neumann R.[1], Albuquerque, G.S.[1]

[1]CETEM – Center for Mineral Technology – Avenida Ipê, 900, 21941-590 Ilha da Cidade Universitária – Rio de Janeiro – RJ – Brazil – mmonte@cetem.gov.br

ABSTRACT

The results of the technological characterization and flotation studies performed on the primary phosphate ore from Irecê, Bahia State, Brazil are presented here. The ore presents 19.3% P_2O_5 and a CaO/P_2O_5 ratio of 2.19, due to the high amount of non-apatitic calcium. The mineralogical composition of the ore is fluorapatite (45%), dolomite (45%), calcite (6%), quartz (2%) and iron oxihydroxides (2%). The natural liberation spectrum indicates that apatite is never fully liberated from its gangue, although below 104 μm the gangue starts to liberate from the apatite. Flotation tests were performed using orthophosphoric acid as modifier and oleic acid as the collector showed a good selectivity, considering the small particle size of the sample (56.45% below 37 μm). The best result of carbonate gangue flotation experiments achieved a concentrate with 35.4% of P_2O_5 at a phosphate recovery of 66.7%, discharging 45% (wt) of the initial sample with about 9% P_2O_5. Other assays for the concentrate are CaO 50.6%, Fe_2O_3 0.2%, MgO 1.7%, LOI 4.8%, CaO/P_2O_5 ratio of 1.43, MgO/P_2O_5 of 0.05, and R_2O_3/P_2O_5 of 0.03. This concentrate matches specification for the phosphoric acid plants.

INTRODUCTION

The sedimentary deposit of phosphatic rock from Irecê- Bahia State represents one of the scarce possibilities for phosphate production in Northeastern Brazil. The weathered ore is currently beeing exploited, and, after dry processing, produces about 120,000 t of concentrate per year, but the primary ore contains high amounts of dolomitic limestone as impurity. Attritioning, screening and desliming operations can reduce a number of impurities, but further reduction in its levels is achieved by flotation.

In igneous deposits the phosphate mineral (apatite) is well crystallized and exhibits a greater floatability than in sedimentary deposits. All phosphate ores currently processed by flotation in Brazil are of igneous origin, being associated to carbonatitic rocks. This peculiarity promoted the need to develop specific technological routes to treat Brazilian phosphate ore (Araujo and Peres, 1996). However, the application of these methods to process sedimentary phosphate ores has not been successful (Cassola and Cassola, 1988). This has been attributed to the: i) high specific surface area of sedimentary ore; ii) inability to liberate carbonaceous material from phosphates in the flotation size range; and iii) substitution of CO_3^{2-} and F for PO_4^{3-} in the apatite.

Alternative techniques to beneficiate dolomitic phosphate ores are based on the differences in surface chemical properties of the two minerals and involve flotation of apatite or dolomite/apatite from their mixture (Moudgil & Somasundaran 1995). The main difficult is to obtain an apatite concentrate containing MgO/P2O5 ratio less than 0,015. High amounts of MgO (>0.8%) increases the viscosity of the phosphoric acid, thus raising processing costs.

Cassola & Cassola (1988) observed that the addition of orthophosphoric acid depressed dolomite particles. The use of a two-stage conditioning step, at different pH values, yielded promising flotation results for the primary phosphate ore from Irecê.

Researchers from the Rajastahn State Mines & Minerals Ltd., India, also developed a technique to selectively float dolomitic gangue from this ore. Orthophosphoric acid was employed to depress phosphate and an aqueous oleic acid emulsion was used as the collector for carbonate materials under slightly acidic pH conditions. The application of this process yielded a concentrate containing less than 1.5% MgO at phosphate recoveries in the range of 60 to 74%.

In this work, the results of technological characterization of Irecê phosphate ore are presented (specially, the liberation spectrum of apatite in relation to the carbonatic gangue by means of image analysis) to provide framework for concentration studies.

EXPERIMENTAL

The sample of the primary phosphate ore, obtained from CBPM - Mineral Research Company of Bahia State, was homogenized, split and crushed to 100% below 1.7 mm. Subsamples were send for chemical assays and size distribution analysis, by sieving following Tyler series from 295 to 37 μm. All fractions were analysed by X ray diffraction, using a Siemens AXS D5005 diffractometer, Cu kα radiations and parallel geometry. All fractions above 37 μm were mounted in epoxy resin, ground and polished with diamond grinding media, and analysed in a LEO S440 scanning electron microscope, fitted with a Pentafet SiLi detector and Oxford ISIS L300 energy dispersion microanalysis system. Image analysis was performed on at least 40 high-contrast, high quality SEM backscattered electron images for each narrow-sized fraction, using MMIA™ (Mineral Metallurgical Image Analysis – King & Schneider 1993) and Stereosoft™ (King & Schneider 1998) for stereological correction.

The flotation tests were conducted in a self-aerating D12 Denver flotation cell with 500 g sample grounded in a bar mill to a particle size P_{90}= 74 μm. Initially, the material was conditioned with 0.4 kg/t · sodium hydroxide and with an emulsion which consisted of 1.18 kg/t oleic acid, 98 g/t methyl isobutyl carbinol (MIBC), and 49 g/t sodium hydroxide, during 5 minutes, at pH 10. In a second step of conditioning, the pH of the pulp was adjusted to 5, using orthophosphoric acid (5.85 kg/t), as modifier, for a conditioning time of 1 min. The flotation tests were performed as a rougher and two cleaner steps, 20% or 55% solids in the pulp. The flotation time of rougher and cleaner steps was 8 and 6 min, respectively. An extra modifier dosage (3.4 or 3.9 kg/t) was added for each cleaner step. The flotation products were filtered, weighted, and chemically analysed. The flotation tests were carried out in triplicate.

RESULTS

Table 1 presents the ore's chemical composition. The CaO/P_2O_5 ratio of 2.19 is due to high non-apatitic calcium minerals, mainly dolomite and calcite.

As determined by XRD and SEM/EDS, the main minerals in this ore are fluorapatite and dolomite (45% each), with minor calcite (6%) and quartz (2%), muscovite and caolinite (2%) in the fine fractions, barite and undetermined iron oxihydroxides. Mineral quantification was done by stoichiometry.

Table 1 – Chemical composition (wt% unless indicated) of the primary phosphate ore from Irecê.

SiO_2	2.2	K_2O	0.17
TiO_2	0.03	Na_2O	0.12
Al_2O_3	0.16	P_2O_5	19.3
FeO	0.28	PF	23.64
Fe_2O_3	0.44	Total	98.54
MgO	9.9	S (ppm)	1.681
MnO	< 0.01	F (ppm)	24.000
CaO	42.3	Cl (ppm)	56

The natural liberation spectrum of the apatite against its gangue minerals, conditional on size and calculated from digital image analysis, is presented in Figure 1. Apatite is never completely liberated from its gangue, as most richer particles, even for the small particle size class, concentrate in the 80-90% class. The gangue, on the other hand, starts to liberate at 74 μm, but gangue liberation is not good, down to 37 μm.
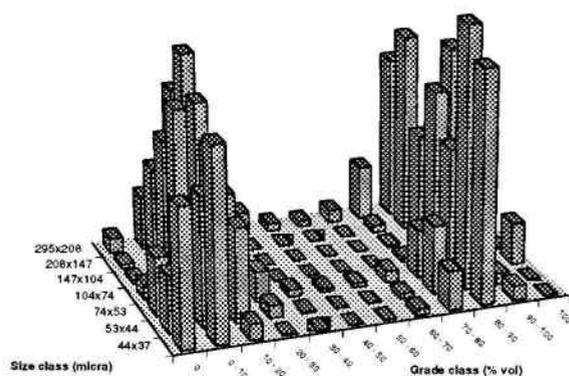

Figure 1 – Natural liberation spectrum of apatite. Conditional on size.

The bivariate liberation spectrum of the apatite, considering the size distribution of the ore as used for the concentration tests (after 20 minutes grinding in a bar mill), is represented in Figure 2. Most particles are of liberated or almost liberated (more than 90%) gangue, or of apatite containing up to 10% gangue. Based on this data, prediction of the grade of an hypothetical concentrate (considering perfect separation), overall apatite recovery and mass discharge in the tailings is possible, and represented in Figure 3.
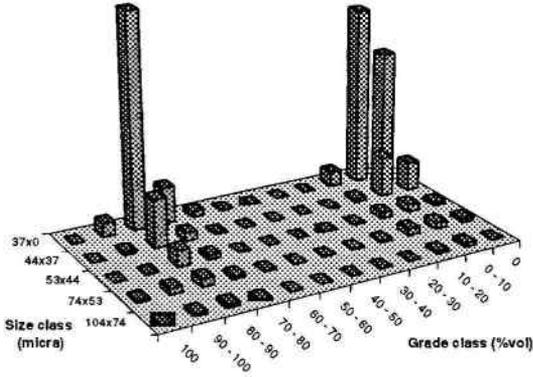
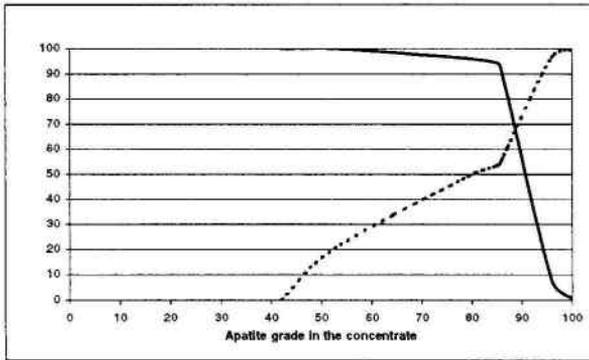**Figure 2** – Bivariate liberation spectrum of apatite.

Figure 3 – Prediction of overall apatite recovery (full line) and mass discarded to the tailings (dashed line) as a function of the concentrate's grade, considering perfect separation.

The best predicted result is a concentrate containing about 85% apatite (corresponding to 36% P_2O_5), 92% overall recovery of the apatite, and discarding 55% of the mass to the tailings.

Table 2 shows the results and conditions of the bench scale flotation tests using two different concentrations of modifier (ortophosphoric acid) in the cleaner steps. As can be seen, with the increase of the modifier, P_2O_5 concentration rises and MgO concentration falls slightly, and there is some increase in the phosphate recovery.

Using a lower solid density in the pulp (Table 3) may have a favorable effect on selectivity, generating a concentrate with MgO content around 2.5% and a recovery of almost 76% of the phosphate, and promoting a small increase in the grade of the concentrate, at a modifier concentration of 3.4 kg/t. This effect was more pronounced when the modifier was added at a dosage of 3.9 kg/t in the cleaner step, and best grades were obtained on this way (35.4% P_2O_5 and 1.70% MgO).

The best results achieved in the bench scale flotation tests (condition 2 and 4) showed a phosphate concentrate with 35.4% of P_2O_5, recovery of 66.7%, and discarding more than 62% of the mass, or with a lower P_2O_5 content, 32.9%, but recovering 78.2% of the phosphate in 47% of the mass. Both concentrates are within the limits accepted for phosphoric acid production, and further fine-tuning of the process should be conducted on pilot scale.

Table 2 - Flotation test results for primary phosphate ore from Irecê, using **55%** solids in the pulp.

Products	Condition 1: Rougher step: emulsion +5.85 kg/t H₃PO₄ Cleaner step 3.4 kg/t H₃PO₄				
	Weight (%)	Content (%)		Distr. (%)	
		P_2O_5	MgO	P_2O_5	MgO
Conc. (rgh.+cl.)	44.38	32.52	3.27	73.2	14.9
Tail	55.62	9.50	14.90	26.8	85.1
Feed	100	19.72	9.74	100	100
Products	Condition 2: Rougher step: emulsion +5.85 kg/t H₃PO₄ cleaner step 3.9 kg/t H₃PO₄				
	Weight (%)	Content (%)		Distr. (%)	
		P_2O_5	MgO	P_2O_5	MgO
Conc. (rgh+cl)	46.93	32.90	2.90	78.2	15.1
Tail	53.07	8.10	15.10	21.8	84.9
Feed	100	19.73	9.40	100	100

Table 3 - Flotation tests results for primary phosphate ore from Irecê using **20%** solids in the pulp.

Products	Condition 3: Rougher step: emulsion +5.85 Kg/t H_3PO_4 Cleaner step **3.4 kg/t H_3PO_4**				
	Weight (%)	Content (%)		Distr. (%)	
		P_2O_5	MgO	P_2O_5	MgO
Conc. (rgh.+cl.)	44.11	33.50	2.50	75.9	11.48
Tail	55.89	8.40	15.20	24.1	88.6
Feed	100	19.49	9.59	100	100
Products	Condition 4: Rougher step: emulsion +5.85 Kg/t H_3PO_4 Cleaner step **3.9 kg/t H_3PO_4**				
	Weight (%)	Content (%)		Distr. (%)	
		P_2O_5	MgO	P_2O_5	MgO
Conc. (rgh+cl)	37.52	35.4	1.70	66.7	6.9
Tail	62.48	10.60	14.00	33.3	93.1
Feed	100	19.91	9.40	100	100

CONCLUSIONS

The mineralogy of the primary phosphate ore from Irecê (Bahia) comprises fluorapatite and dolomite (45% each), minor calcite (6%) and quartz (2%), with 2% muscovite and caolinite in the fine fractions, barite and undetermined iron oxihydroxides.

Liberation of the fluorapatite is never complete, and most rich particles still contain 10 to 20% impurities, at 37 µm. The gangue's liberation is better, starts at 74 µm, and at 37 µm 50% of the particles are of 90% or more liberated gangue, 20% of them fully liberated.

Based on the liberation data, it is possible to predict that the best concentrate, considering perfect separation, would contain 36% P_2O_5 (85% apatite), and allow recovery of 92% of the phosphate in 45% of the starting mass.

The best bench scale flotation test, from the concentrate grade point of view, using carbonate gangue flotation method, the ore ground to 90% wt passing 74 µm, orthophosphoric acid as modifier,

pH=10 and an emulsion of oleic acid as collector, allowed a P_2O_5 content of 35.4%. Phosphate recovery was 66.7%, and other assays for the concentrate are CaO 50.6%, Fe_2O_3 0.2%, MgO 1.7%, 4.8% LOI, CaO/P_2O_5 ratio of 1.43, MgO/P_2O_5 of 0.05, and R_2O_3/P_2O_5 of 0.03.

These results are partially in accordance with the prediction based on image analysis. The concentrate grade of 35.4% P_2O_5 is close to the predicted 36%, but the overall recovery of 66.7% is far from the predicted 92%; the mass discard to tailings, on the other hand, was higher than predicted, containing about 10% P_2O_5.

The prediction based on image analysis considers perfect separation, just accounting the richer and poorer particles of Figure 2. As most of the particles are not fully liberated, a process based on the mineral's surface like flotation may be very sensitive to small changes of both, particle surface and reagents (even more if the reduced particle size is considered). This can be observed by comparing the results using condition 2 of Table 2 and condition 4 of Table 3, where the improvement of the concentrate grade in just 1,5% P_2O_5 represents a loss of 11.5% of the phosphate recovery.

ACKNOWLEDGEMENT

REFERENCES

Araújo, A.C. & Peres, A.C. Froth Flotation in Brazil. In: Proceedings of the 3rd CETEM-Aachen. Workshop on Mineral Processing and Environmental Issues, (1996) 21-43. Editions Wissenschaftsverlag, Aachen.

Cassola, H.L. & Cassola, M.S. Pesquisa de Reagentes e Definição de Rota de Processos por Flotação – Fosfato de Irecê - Ba. In: Proceedings of the XIII Encontro Nacional de Tratamento de Minérios e Hidrometalurgia., São Paulo, 1988, Brazil, Vol. 1, pp.117-130.

King, R.P. & Schneider, C.L. Stereological Correction of Linear Grade Distributions for Mineral Liberation. *Powder Technology*, 1998.

King, R.P. & Schneider, C.L. An Effective SEM-Based Image Analysis System for Quantitative

Mineralogy. *KONA Journal*, 1993, v11, pp.165-177.

Moudgil, B.M. & Somasundaran, P. Advances in Phosphate Flotation. In: *Somasumdaran, P. (ed.), Advances In Mineral Processing, Chapter 25. Colorado, Littleton.* 1995.

Rajasthan State Mines & Minerals Ltd. Beneficiation of rock phosphate from Irece, Brazil & suitability of the concentrate for manufacture of fertilisers [*sic*]. *Research report prepared for CBPM – Companhia Bahiana de Pesquisa Mineral*. Rajasthan, 39 p. Not dated.