The challenging passage through micro-flotation to mechanical flotation cell: Case of low-grade Moroccan phosphates

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Abstract

Due to increased population growth, arable land is becoming more and more depleted. Phosphorus is then needed more than ever. Therefore the focus is brought to the possibility of returning to the deposits considered so far without interest (Cao et al. 2019; Chen et al. 2017; Dong et al. 2017; Filippov et al. 2019). Within a major project with the group OCP, this work aims at upgrading low grade phosphates. New beneficiation routes are investigated to selectively separate apatite from gangue minerals, mainly carbonates and quartz. A micro-flotation study was conducted on pure minerals of fluorapatite, calcite and quartz based on mineralogical feature of low grade Moroccan phosphates. Results showed the possibility for a direct flotation. The most potent combinations of reagents were: carboxymethylcellulose and sodium alginate when used with sodium oleate and ATRAC as collectors (Aarab et al, 2020). A scale up of these findings to the mechanical cell fail to obtain the same tendency. An asymmetrical fractional factorial design was used with, as parameters: the type of the collector and depressant, their dosage and conditioning time, flotation time, pH and desliming. Partial matrix results presented in this work confirmed the non-possibility of direct flotation under these conditions, but a reverse flotation is possible.
Conclusions will be drawn once the DOE results are completed, considering the possibility of direct and reverse flotation of low grade phosphate ore.

Keywords: direct flotation; DOE; batch flotation; low-grade phosphate ore.

1. Introduction

Given the burgeoning populations, phosphorus is becoming more and more in demand. As a result, considerable concern has been brought to the direct flotation of low-grade phosphates. Because of their complexity and high content in gangue minerals, highly selective reagents should be developed to ensure separation efficiency. Many researchers addressed new formulations (Huang et al., 2011; Karlkvist et al., 2015; Jong et al., 2017; Cao et al., 2019; Nan et al., 2019) and tested the synergistic effect of reagents (Cao et al., 2016; Chen et al., 2017; Dong et al., 2017; Filippov et al., 2019). In the light of these research works, a micro-flotation study was conducted to investigate promising reagents for low-grade Moroccan phosphate beneficiation. Sodium oleate and ATRAC, combined with sodium alginate and carboxymethylcellulose, were the most potent combinations. They allowed 95% recovery of apatite, with only 19% of calcite (Aarab et al., 2020). Zeta potential measurements, adsorption tests, and FTIR analyses complied with Hallimond tube findings and demonstrated chemisorption. This work is a continuance of the previous study where reagent combinations are tested for low-grade Moroccan phosphate ore at the mechanical flotation cell scale.

2. Materials and methods

2.1. Mineral specimens

Two samples A and B were provided by the OCP group to form a database of low-grade Moroccan phosphate flotation. Figure 1 and Table 1 show that both of them contain fluorapatite, calcite, and quartz as the main constituent phases, but in different proportions considering chemical analyses.

Figure 1: Moroccan phosphate ore sample diffractogram

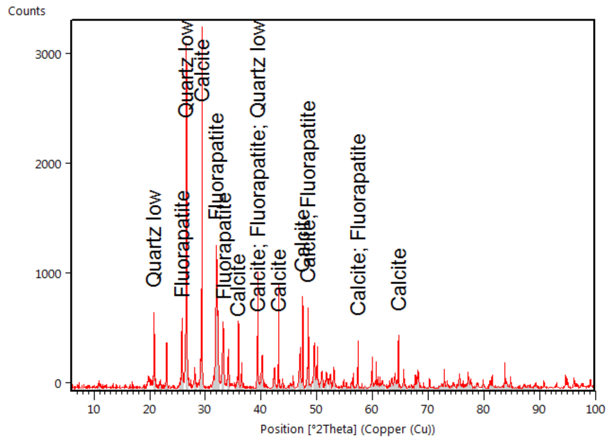

Table 1. Chemical composition of the two samples studied

Element composition Wt.%	P_2O_5	SiO_2	CO_2	MgO
Sample A	17.05	26.90	8.70	0.53
Sample B	25	6.87	10.24	0.98

2.2. Chemical reagents

The collectors tested in this study were Sodium oleate (NaOl) and ATRAC. The depressants used were carboxymethylcellulose (CMC), sodium silicate, sodium alginate (NaAl), and corn starch. Methyl isobutyl carbinol (MIBC) was used as a frother and NaOH as a pH controller. Sigma Aldrich provided all the flotation reagents used except for ATRAC, which was from AkzoNobel.

2.3. Experimental procedure

2.3.1. Batch flotation tests

A 1.5 L flotation cell was used for the flotation tests. For each experiment, 500 g of phosphate ore was wet ground with a ball mill to 80 wt.% passing 135µm.
The flotation test protocol was as follow:

- The phosphate pulp is adjusted to the desired density, transferred into the flotation cell and stirred for 1 min,
- pH was adjusted using NaOH with 3 min of conditioning time,
- The depressant is added, and the solution was conditioned for the desired time,
- The collector is added, and the slurry was stirred for the desired time,
- Before froth collection, 30 g/t of MIBC was added with 1 min of conditioning time.

All conditioning processes were conducted in the absence of airflow. After completion of the flotation test, the concentrate and tailings products were filtered, dried, weighed, and analyzed by spectrophotometric method (Bridger and Boylan, 1953) and Bernard calcimeter. Because of the challenging similarity of apatite and carbonates, the elements assayed were P_2O_5 and CO_2, which allowed to calculate their recoveries.

2.3.2. Experimental design

The control parameters considered in the experiment included the type of collector and depressant, their dosage and conditioning time, pH, desliming, and flotation time. A mixed-level factorial design ($2^2 3^3 4^1 5^3$) was conducted to screen the favorable conditions.

3. Results and discussion

3.1. Batch flotation validation tests

The qualified combinations of micro-flotation obtained by Aarab et al. (2020) were first tested in the mechanical flotation cell. All the experiments in this paragraph were conducted at the constant operating conditions (collector dosage = 1000 g/t, depressant dosage = 667 g/t, pH = 9.5, conditioning time of each reagent = 5 min, flotation time = 5 min, sample A being deslimed at 40 µm). Table 2 shows that the transition from micro-flotation to the mechanical cell is not as obvious. Unlike the results of the Hallimond tube where direct flotation was achieved, cell flotation results were different. These contradictory findings led us to suspect other parameters that may influence flotation response as attrition, solid content Cs and desliming (Alsafasfeh et al., 2017; Kawatra et al., 2013).

Table 2. Results of flotation experiments replicated based on micro-flotation findings (collector dosage: 1000 g/t, depressant dosage: 667 g/t, pH 9.5, conditioning time of each reagent: 5 min, flotation time: 5 min)

N° Exp	Collector	Depressant	Products	Yield (%wt)	$\%P_2O_5$	$R(\%P_2O_5)$	$\%CO_2$	$R(\%CO_2)$
1	NaOl	CMC	Concentrate	15.43	16.54	14.88	11.35	17.51
			Tailings	84.57	17.27	85.12	9.75	82.49
			Feed	100	17.16	100	10	100
2	NaOl	NaAl	Concentrate	8.34	15.43	7	11.07	9.58
			Tailings	91.66	18.67	93	9.5	90.42
			Feed	100	18.4	100	9.63	100
3	ATRAC	CMC	Concentrate	10.73	16.18	9.84	10.94	11.82
			Tailings	89.27	17.83	90.16	9.81	88.18
			Feed	100	17.65	100	9.93	100
4	ATRAC	NaAl	Concentrate	7.68	15.52	6.58	11.03	7.94
			Tailings	92.32	18.34	93.42	10.63	92.06
			Feed	100	18.12	100	10.66	100

3.2. Effect of attrition

Attrition scrubbing is generally used as a pre-treatment stage for the removal of clays and weakly bonded gangue minerals to clean the valuable mineral surface. Thus, 3.5% of gangue was removed by attrition from sample B. At pH 11, NaOl was the collector used at a dosage of 1500 g/t with 5 min of conditioning time. The depressant was used at a dosage of 50 g/t and conditioned for 2 min. 300 g/t sodium silicate was used as a dispersant with 2 min of conditioning time. Flotation time was 5 min. Compared to results in Table 2, results shown in Table 3 demonstrated that attrition improved significantly separation although tending toward reverse flotation, and thus highlighting how mineral surface coating with clays and gangue minerals can alter beneficiation efficiency.

Table 3. The effect of attrition on flotation response (collector dosage: 1500 g/t, conditioning time: 5min, depressant dosage: 50 g/t, conditioning time: 2 min, flotation time: 5 min, pH: 11. 300 g/t sodium silicate was used as a dispersant with 2 min of conditioning time)

Collector	Depressant	Products	Yield (%wt)	$\%P_2O_5$	$R(\%P_2O_5)$	$\%CO_2$	$R(\%CO_2)$
NaOl	CMC	Concentrate	27.8	15.8	18.22	20.55	46.63
		Tailings	72.2	27.29	81.78	9.05	53.37
		Feed	100	24.09	100	12.25	100
	NaAl	Concentrate	39.62	18.13	30.10	13.71	56.96
		Tailings	60.38	27.63	69.90	6.80	43.04
		Feed	100	23.86	100	9.54	100
	Starch	Concentrate	21.33	12.06	10.77	22.48	37.31
		Tailings	78.67	27.08	89.23	10.24	62.69
		Feed	100	23.87	100	12.85	100

3.3. Effect of solid content Cs

Tests were conducted at different solid content (4, 8, and 12%) on the sample B being deslimed, attrition scrubbed, and then deslimed to evaluate its effect on flotation response. At the same operating conditions as attrition tests, the results presented in Table 4 show that the decrease in Cs (from 12 to 4%) does not significantly improve the $\%P_2O_5$ content; on the other hand, the yield decreases, which is probably due to the unstable froth observed during the tests.

Table 4. The effect of solid content on flotation response(collector dosage: 1500 g/t, conditioning time: 5min, depressant dosage: 50 g/t, conditioning time: 2 min, flotation time: 5 min, pH: 11. 300 g/t sodium silicate was used as a dispersant with 2 min of conditioning time)

Cs (%)	Depressant	Products	Yield (%wt)	$\%P_2O_5$	$R(\%P_2O_5)$	$\%CO_2$	$R(\%CO_2)$
4	CMC	Concentrate	3.86	–[1]	–	–	–
		Tailings	96.14	17.86	–	11.47	–
		Feed	100				
8		Concentrate	17.52	19.68	15.44	26.94	38.58
		Tailings	82.48	22.90	84.56	9.11	61.42
		Feed	100	22.33	100	12.23	100
12		Concentrate	27.80	15.80	18.23	20.55	46.64
		Tailings	72.20	27.29	81.77	9.05	53.36
		Feed	100	24.1	100	12.24	100
4	NaAl	Concentrate	2.31	–	–	–	–
		Tailings	97.69	24.48	–	12.97	–
		Feed	100		100		
8		Concentrate	29.73	24	33.03	23.95	55.12
		Tailings	70.27	24.29	66.97	8.25	44.88
		Feed	100	24.2	100	12.91	100
12		Concentrate	32.47	20.65	30.46	23.22	56.34
		Tailings	67.53	21.69	69.54	8.65	43.66
		Feed	100	21.35	100	13.38	100

3.4. Effect of desliming

Table 5 showed that desliming strongly decreases the yield: slimes, although negatively affect selectivity, help stabilize the froth. During the flotation tests with deslimed sample B at 40µm, the scum recovered becomes less charged around the first 5 min.

3.5. Mixed-level fractional factorial design

Since the examined parameters were not responsible for the reversal of the flotation response, the design of experiments with a broader scan of the factor levels has been developed.
The experimental design was carried out in part. The results presented in Table 6 relate to NaAl/NaOl combination on sample B. We can conclude that:

- Pending the results of the whole experimental design, the trend of these partial results shows that a reverse flotation is possible.
- The combination of NaAl/NaOl favors reverse flotation with a slight selectivity for carbonates. Experiment No 46, the tailings of which reached 30.46% P_2O_5, approached the requirement for a salable product with 68.15% of the recovery.

4. CONCLUSION

The most potent combinations in micro-flotation were sodium oleate and ATRAC combined with sodium alginate and carboxymethylcellulose. A scale-up of these findings to the mechanical flotation cell was not as automatic as expected. The investigation of other suspected parameters that may affect flotation response as attrition, solid content, and desliming showed that they might enhance separation

[1] The amount of float does not allow $\%P_2O_5$ and $\%CO_2$ analyses.

efficiency. Still, it was not responsible for the reversal of the flotation response. Partial matrix results of the asymmetrical factorial design give a possibility of reverse flotation by NaOl/ NaAl combination. These contradictory findings may be due to the high degree of substitution in the crystal lattice of the phosphate mineral or to the finer liberation size as phosphate particles can contain fine carbonates inclusions. Therefore, conclusions considering the possibility of direct or reverse flotation will be drawn once the full factorial design results with, as factors: the type of collector and depressant, their dosage and conditioning time, flotation time, pH and desliming are completed.

Table 5. Effect of desliming on flotation response

Collector	Desliming	Collector cond. time (min)	Depressant cond. time (min)	Flotation time (min)	Depressant	Collector dosage (g/t)	Depressant dosage (g/t)	pH	Products	Yield (%wt)	$\%P_2O_5$	$R(\%P_2O_5)$	$\%CO_2$	$R(\%CO_2)$
NaOl	Without	12	8	15	CMC	1500	100	10	Concentrate	63.86	21.60	56.84	13.08	73.98
									Tailings	36.14	28.99	43.16	8.14	26.02
									Feed	100	24.27	100	11.29	100
	With	8	2	15	NaAl	1500	600	9	Concentrate	8.66	16.87	5.50	16.20	10.71
									Tailings	91.34	27.46	94.50	12.80	89.29
									Feed	100	26.53	100	13.09	100

Table 6. Partial flotation results of DOE

N° Exp	Collector	Desliming	Collectorcond. time	Depressantcond. time	Flotation time	Depressant	Collector dosage (g/t)	Depressant dosage (g/t)	pH	Products	Yield (%wt)	$\%P_2O_5$	$R(\%P_2O_5)$	$\%CO_2$	$R(\%CO_2)$
1	NaOl	Without	5	2	5	NaAl	500	0	8	Concentrate	40.46	20.73	34.62	17.07	41.88
										Tailings	59.54	26.60	65.38	16.10	58.12
										Feed	100	24.22	100	16.5	100
5		With	8	5	8		3000	600	12	Concentrate	43.94	18.15	33.77	18.47	52.05
										Tailings	56.06	27.89	66.23	13.34	47.95
										Feed	100	23.61	100	15.60	100
16		With	8	2	15		1500	600	9	Concentrate	8.66	16.87	5.50	16.20	10.71
										Tailings	91.34	27.46	94.50	12.80	89.29
										Feed	100	26.54	100	13.09	100
20		With	12	2	15		1000	25	11	Concentrate	21.16	13.73	11.52	17.07	29.38
										Tailings	78.84	28.31	88.84	11.01	70.62
										Feed	100	25.22	100	12.30	100
31		Without	12	2	5		3000	25	8	Concentrate	44.63	20.45	37.51	17.10	52.58
										Tailings	55.37	27.46	62.49	12.43	47.42
										Feed	100	24.33	100	14.51	100
35		Without	5	2	5		1000	0	10	Concentrate	40.97	20.44	33.83	15.48	45.46
										Tailings	59.03	27.74	66.17	12.89	54.54
										Feed	100	24.75	100	13.95	100

43	With	5	2	15	2000	100	9	Concentrate	8.93	17.01	5.56	17.07	11.90
								Tailings	91.07	28.31	94.44	12.39	88.10
								Feed	100	27.3	100	12.80	100
46	With	5	8	8	1000	0	12	Concentrate	35.06	20.02	26.20	13.21	41.72
								Tailings	64.94	30.46	73.80	9.96	58.28
								Feed	100	26.79	100	11.1	100

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