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Investigating sodium sulphate as a phosphate depressant in acidic media

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ABSTRACT

Reverse flotation is a commonly used technique for separating carbonate impurities from sedimentary phosphate ores using fatty acids collectors. Although, oleic acid represents one of the famous fatty acids that have been used as a collector in phosphate flotation circuits, it is a non-selective collector. Therefore, the selection of depressing agent is the most controlling factor. In this study, sodium sulphate was used as a phosphate depressant. The role of sodium sulphate in separating phosphate from its impurities and producing an acceptable concentrate grade for phosphoric acid production (equals or more than 30% P_2O_5) was evaluated using augmented factorial design. The collector dose, depressant dose, solid %, flotation time and pH were chosen as main affecting variables. The results showed that the addition of sodium sulphate improves the phosphate grade and recovery especially at highly acidic pH. They showed also that the solid % and the pH represent additional key factors in achieving a good grade concentrate due to their role in controlling the amount of ionic species in the flotation pulp. A concentrate contains >32% P_2O_5 was obtained with a recovery ranges from 84% to 87%.

1. Introduction

Most of the world production of phosphate rocks is used in phosphoric acid production as well as fertilizers manufacturing. The depletion of easy-to-beneficiate siliceous phosphate ores attracts more attention to solve the problems associated to the flotation of calcareous phosphate ores which require further research work [1–10].

Reverse flotation, in particular, showed promising results with different types of phosphate ores all over the world [1,2,11–13]. It depends on depressing phosphate and floating carbonate minerals by anionic collectors at acidic pH [7,10,14]. The most extensively used collectors in the reverse flotation scheme are long-chain fatty acids and their salts, especially oleic acid and sodium oleate [14]. On the other hand, several types of depressants were reported in the literature such as: sodium silicate, H_2SO_4, H_3PO_4, starch, carboxyl methyl cellulose (CMC), tannic acid, aluminium sulphate, fluosilicic acid, fluoric acid, (Na, K) Tartarat, sodium carbonate/bicarbonate, sodium tripolyphosphate, diphosphonic acid, sulforganic compound, and dipotassium hydrogen phosphate [15–18].

Reverse flotation of calcareous phosphate encounters some difficulties due to its successful pH range is acidic. High solubility of metal cations such as Ca-ions and Mg-ions represents one of the controlling factors in accomplishment of good phosphate grade and recovery. The effectiveness of a phosphate depressant measured by its effect on the phosphate recovery as well as its interaction with the type and level of collector used [19].

Selection of suitable depressant depends on some characteristics that should be present in the used depressant such as the depressant specificity towards the mineral, that needs to be depressed, and their pH dependence. The functional groups of the depressant play an important role in its working mechanism. A dual function is needed in the depressant which is the adsorption to the needed to be depressed mineral in addition to improve its hydrophilicity. It was reported that the depressants with sulphate groups may work preferentially in depressing phosphate at acidic pH especially when the sulphuric acid and its sulphate salts were used as pH modifiers. On the other hand, the used depressant should have no or very low competition with the collector functional groups [19].

Therefore, the present work aims at studying the role of sodium sulphate (Na_2SO_4) as a phosphate depressant in beneficiating Al-Jalamid phosphate ore by flotation process using oleic acid as a collector. Sodium sulphate, Na_2SO_4, depressing action of the phosphate mineral as well as its effect on the flotation concentrate grade and recovery was studied using experimental design in

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terms of the controlling factors; namely: pH, collector dosage, solid content, depressant dosage and flotation time. The collector and depressant interaction was also investigated.

2. Experimental

2.1. Materials

2.1.1. Phosphate sample

A low-grade sample of phosphate ore, Al-Jalamid area, Northern region of Saudi Arabia, was used in the current study. The representative sample was primarily crushed by jaw crusher followed by screening to get the −4.25 + 0.106 mm size fraction. The −4.75 + 4.25 mm cut was taken to an attrition mill for further grinding. After several grinding and sieving steps the −0.425 + 0.106 mm cuts were collected and mixed together thoroughly to prepare the flotation feed.

2.1.2. Chemical reagents

Oleic acid of 99% purity was used as a collector (Aldrich Chemicals, Germany). Analytical grade of NaOH and H_2SO_4 were used as pH regulators. In addition, sodium sulphate (Na_2SO_4) was used as a phosphate depressant.

2.2. Methods

2.2.1. Chemical analysis

Wet chemical analysis of samples was conducted using standard methods for phosphate analysis. Magnesium, calcium and iron oxides were determined by inductive couple plasma spectrometer (ICP). Phosphorous was determined by spectrophotometric method using 'Perkin–Elmer, model Lambda 3B' spectrophotometer.

2.2.2. Scanning Electron Microscope (SEM)

The surface characteristics of the phosphate ore samples, before and after the flotation process, were identified using scanning electron microscopy (SEM-JEOL 840).

2.2.3. Flotation experiments

The experimental runs were conducted in a Wemco Fagergren type flotation cell with a volume capacity of two litres. Feed sample was −0.425 + 0.106 mm size fraction. The feed was added to the flotation cell with water to get a required pulp density. The pulp density (solid %) was adjusted to be 50% at conditioning and 25% during flotation. Then the required amount of depressant (sodium sulphate) was added and conditioned for two minutes. Then the measured amount of collector was added and pH value was adjusted by adding sulphuric acid. The air was turned on after one minute of collector addition. The flotation time was taken according to the statistical design. Both the concentrate and the float were filtered, dried and weighed. Concentrate and float were analysed for P_2O_5 content.

2.2.4. Factorial experimental design

A series of 18 experiments, following a two-level randomized 2^{5-1} fractional factorial design (FFD) with two centre points (two repeated represented by experiments 17 and 18, Table 3), were designed based on the important factors that affect the reverse flotation process. This design was augmented with "star" design (extra 10 experiments). The studied parameters are pH, collector dosage, solid content, depressant dosage and flotation time. The other operating parameters, such as air flow rate and particles size were maintained constant. The levels of the studied parameters are specified in Table 1.

Table 1
The operating parameters and their levels.

Operating parameters	Units	Symbol	Low (−)	High (+)
pH	–	A	4.5	6.5
Oleic acid dosage	kg/t	B	1.75	5.5
Solids content	%	C	10	30
Na_2SO_4 dosage	kg/t	D	0.0	15
Flotation time	min	E	2.0	5.0

The experimental results will be fitted to a second-order model, which enables the prediction of the output responses namely; P_2O_5%, and P_2O_5 recovery within the studied region, Table 1. The statistical software package Design-Expert, Stat-Ease, Inc., Minneapolis, USA was used for regression analysis of the experimental data and to plot the contour graphs. Analysis of variance (ANOVA) was used to estimate the statistical parameters.

3. Results and discussion

3.1. Chemical analysis and XRD

Al-Jalamid ore is a calcareous phosphate ore of sedimentary origin and consists mainly of calcium fluorapatite $Ca_{10}(PO_4)_6F_2$ with other impurities like Cl, SiO_2, $CaCO_3$, Al_2O_3, Fe_2O_3 and MgO. This ore deposit is characterised by its low P_2O_5 content (20–25%), high calcite ($CaCO_3$), CaO:P_2O_5 weight ratio above 2.0 with low amount of silica among the other impurities [13]. Table 2 shows the chemical analysis of −0.425 + 0.106 mm size fraction used in the flotation experiments of this study. In addition, the X-ray diffraction analysis indicated that the main ore phases are fluorapatite, calcite and silica [20].

3.2. Statistical analysis

The factorial design results of phosphate reverse flotation are given in Table 3 in terms of P_2O_5%, and P_2O_5 recovery %. It is clear that the acidic pH gives a higher grade where the P_2O_5% reaches 35% with reasonable recovery 84% at 10%, 5.5, 3 kg/t, 7.5 kg/t and 3.5 min for solid %, pH, collector dosage, Na_2SO_4 dosage and flotation time, respectively, Table 3.

The analysis of variance (ANOVA) indicated that the R^2 is 0.8520 and 0.9537 and the standard deviation is 1.94 and 8.35 for P_2O_5% and P_2O_5 recovery %, respectively. The analysis shows that the main factor (A) is the most effective and significant factor for P_2O_5% while the main factors (A, B, C, D, E) and their interaction AB, AC, AD, AE are significant model terms for P_2O_5 recovery %, within 95% confidence interval.

Table 2
Chemical characteristics of phosphate sample.

Constituents	% Weight (Dry basis)
P_2O_5	22.6
CaO	50.72
Fe_2O_3	0.10
Al_2O_3	0.23
MgO	0.19
Na_2O	0.32
K_2O	<0.050
F	2.630
Cl	1.540
SiO_2	2.37
L.O.I.[a]	18.25

[a] L.O.I = Loss on Ignition.