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A.A.S. SEIFELNASSR,[*] A.A. AHMED[**]

ENRICHMENT OF ABU-TARTUR PHOSPHATE ORE WASTES

In this work, the amenability of upgrading the reject generated from beneficiating Abu-Tartur phosphate ore by magnetic separation was investigated by two methods: leaching with acetic acid solutions and froth flotation. The reject is characterized by its ferriferous dolomite impurities. To optimize the leaching process, the effects of grain size, acetic acid concentration, acid/solid ratio and the reaction time on loss on ignition percent were investigated. The results of this series of experiments gave unsatisfactory results. Separation of phosphate minerals from the associated impurities has been successfully achieved by flotation. The studied parameters such as particle size distributions, conditioning time, mode of collector addition and depressant type were investigated to reach optimum conditions. The experimental results show that, it is possible to obtain phosphate concentrate assaying 30% P_2O_5 and 0.75% MgO with P_2O_5 recovery of about 75% from a feed assaying 23% P_2O_5 and 3.77% MgO.

INTRODUCTION

For many years the mining industry has considerable interest in reprocessing the wastes of mineral processing plants. The most satisfactory way of dealing with wastes is to make positive use of them, such as reprocessing in order to recover additional values.

Abu-Tartur phosphate (Egypt) beneficiation process discards large amount of phosphate tailings as magnetic wastes and slimes. These wastes represent about 20% of the run-of-mine ore. Out of this, the magnetic waste assays about 23% P_2O_5 and comprises about 10% of the mined P_2O_5 value (Abu-Tartur, 1990). Therefore, recovery of phosphate values from those tailings would provide additional substantial amount of phosphate values annually. These tailings consisted mainly of ankerite, dolomite and pyrite in addition to phosphate minerals. The dolomitic impurities cannot be reduced by conventional techniques to produce concentrates containing less than 1%MgO.

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[*] Mining Engineering Department, Faculty of Mining and Petroleum Engineering, Suze Canal University, Suez, Egypt.

[**] Mining and Metallurgy Engineering Department, Faculty of Mining Engineering, Assiut University, Egypt.

Several efforts to eliminate the dolomtic impurities from phosphate ores have been reported. These efforts are concerned with the application of calcination, flotation, acid washing, and magnetic separation. Although calcination is one of the better known processes (Zafar et al., 1995; Kaljuvee et al., 1995) it suffers from several serious drawbacks, among which are the high energy needed as well as the low reactivity of the end product (Bibawy, Yousef, 1983; Elgillani, Abouzied, 1993). In order to avoid the physico-chemical changes in the nature of the phosphate minerals as well as the problems encountered in using this product, flotation of carbonates from calcareous phosphate ores may be more beneficial. Also, acid washing process can be applied successfully in this respect.

Extensive research work on flotation behavior of carbonates from phosphate minerals has been conducted under various experimental conditions (Elgillani, Abouzeid, 1993; Moudgil, Chanchani, 1985; Houot et al., 1985; Anazia, Hanna, 1988; Acarkan et al., 1992; Rao et al., 1985; Houot). Some other attempts concerning the flotation of carbonates from phosphate ores, using anionic, cationic and amphoteric collectors in both acid and basic environments have been also reported (Moudgil, Chanchani, 1985; Houot et al.. 1985). Theoretical contribution (Elgillani, Abouzeid, 1993) based on thermodynamic analysis of phosphate–carbonate–water system showed that it is possible to float carbonates from phosphates in acidic media (pH = 5.8) using an oleate collector in the presence of phosphate and calcium precipitating and complexing ions. The theoretical concept was confirmed experimentally using pure minerals. Also different Egyptian calcitic-dolomitic phosphate ores were successfully upgraded by flotation in acidic media (Mostafa et al., 1980; Orphy et al., 1969; Orphy et al., 1968; Abouzeid et al., 1992; Abdel, 1994; Taha et al., 1977; Abdel-Hamid, 1989; Ramadan et al., 1995; Boulous et al., 1994; El-Shafei, Abu-Gharib, 1977; Mostafa et al., 1980).

Some low grade calcareous phosphate ores around the world were treated by acid washing with dilute acetic acid solutions (Abu-Eishah et al., 1991; Abu-Eishah et al., 1991; Ahmed, 1996). The technique of acid washing for the reduction of carbonates content of phosphate ores was proposed by some authors (Sadeddin, Abu-Eishah, 1990). This technique is based on using dilute acetic acid solutions as a leaching agent for the calcium carbonates without affecting the phosphate mineral itself. Abu-Eishah et al. (1991) reported the reasons of why acetic acid is preferable than other acids. From these reasons, acetic acid is a weak organic acid whose salts are highly soluble in water and are easily separated from the benefeciated solid phosphate product by filtration. Also the dilute acid will be less destructive if some of the produced acetate solution is added to the reaction solution or recycled to the process. They also explained the routes for the recovery of acetic acid from acetate solutions.

In this work, both acid washing and flotation approaches were applied to upgrade the phosphate wastes (the magnetic stream) produced from the high gradient magnetic separator (HGMS) at Abu-Tartur mine to obtain acceptable phosphate product.

EXPERIMENTAL

Materials

Test sample. A representative sample was collected from the magnetic fraction of the high gradient magnetic separator (pilot plant) at Abu-Tartur mine. The received sample has a particle size of 82% – 200 μm. The chemical analysis of the received sample is shown in Table I. The X-ray diffraction (XRD) analysis of the sample showed that mineralogical constituents of the waste sample are mainly appatite, ankerite, quartz and pyrite.

Leaching reagents. Glacial acetic acid was used for acid washing tests.

Flotation reagents. Oleic acid and MIBC were used as collector and frother respectively, sulphuric acid, phosphoric acid and mixture of them (1:1) were chosen as pH regulators and depressants for phosphate minerals.

Table 1. Chemical analysis of the head sample

Component	%	Component	%
P_2O_5	23.2	Na_2O	0.88
CaO	42.69	K_2O	0.09
MgO	3.77	L.O.I	16.24
Al_2O_3	0.64	I.R.	2.71
Fe_2O_3	1.35		

Procedure

The dissolution tests were carried out in a glass reactor of 1000 cm^3 capacity. The pulp was stirred with a variable speed mechanical stirrer fitted with a stainless steel impeller. At the beginning of each run a known weight of the phosphate sample was added to known weight and concentration of diluted acetic acid solutions in a continuous stirred reactor vessel. The reaction has been conducted for a specified time at room temperature. After the completion of each leaching run the content of the reaction vessel was then filtered and washed with water to remove any soluble matter. The solid leached residues were dried and analyzed for its loss on ignition (L.O.I) percent as a quick assessment for the amount of carbonates in the sample.

The flotation experiments were carried out as follows: 100 grams of the undeslimed sample were added to 1000 cm^3 of tap water in a Denver sub-aeration flotation machine of 1 litter capacity. pH of the slurry was adjusted at pH value of (5.8 during conditioning and flotation by adding the phosphate depressant and pH controlling reagents. It is worth to mention here that the pulp pH remains nearly constant during flotation tests. This may be due to the fact that ankerite is an inert type of carbonate. The collector dosage (oleic acid) was added in stages after conditioning the pulp for two minutes and the pulp was further conditioned for anther two minutes in absence of air flow. One minute before flotation two drops MIBC were added as a frother. Then air flow was opened and the flotation continued until barren froth was observed (2–3 minutes). Both the carbonate float and unfloated phosphate concentrate were filtered, washed, dried, weighed and analysed. It is worth to mention here that the loss on ignition (LOI) was used as a fast and convenient assaying parameter to assess the carbonate content in both carbonate float and phosphate concentrate.

RESULTS AND DISCUSSION

Acid washing

The experiments were carried out batchwise to investigate the influence of different parameters such as particle size, concentration of acid solutions, liquid/solid ratio and leaching time on the dissolution of carbonates from the dolomitic phosphate ore.

The effect of particle size on leaching process was investigated using three representative samples of the feed ground to 82% – 200 μm, 100% – 200 μm and 100%
– 160μm. The results showed that fine grinding to – 160μm decrease the loss on ignition only by about 2%. The samples of 82% – 200μm was found to be sufficient for all subsequent experiments. The increase of the acetic acid upto 14%, had negligible effect on the rate of leaching. This may be attributed to the fact that the ore is dolomitic whose reactivity with acetic acid solutions is poor. Moreover, the effects of both liquid/solid ratio and the reaction time on the leaching process were studied and found to have insignificant effects on the dissolution rate of carbonates from the phosphate waste sample.

In conclusion, the treatment of the given phosphate waste sample by acetic acid leaching is not technologically feasible. Therefore, an alternative approach, flotation, is suggested and investigated in order to upgrade the magnetic wastes. The results of these investigations are described in the following section.

Flotation results

It is worth mentioning that the test sample contains a very low percentage of clays (Table 1). Therefore, the feed samples were subjected to the flotation tests without desliming.

For most of the tests reported herein, various parameters were fixed unless otherwise stated. The mixture of phosphoric acid and sulphuric acid (1:1), for example, was used as pH regulator and depressing agent for phosphate minerals. The particle size of the sample was 92% passing 200 microns sieve, impeller speed was 1200 rpm and conditioning time was 2 minutes.

Effect of particle size

Size distribution of the material being floated is important in determining flotation performance. The preliminary flotation tests conducted with the as-received sample (82% – 200) µm) gave very poor flotation results. Therefore, the waste phosphate sample was further ground to different size distributions (Table 2) in order to achieve better liberation of phosphates from the associated gangue materials. The results of these experiments are presented in Figures 1 and 2. Figure 1 shows that the carbonate recovery was enhanced markedly by reducing the particle size of the feed material. Figure 2 depicts the relationship between the loss on ignition and the feed particle size in terms of percent of –200 µm size fraction which is taken as a measure of carbonate content of the floated material. It is clear that the selectivity of carbonate flotation is highly improved when the grinding was just enough to liberate the phosphates from other minerals which was reached at 92% – 200 µm. Further grinding led to abrupt decrease in the selectivity of carbonate flotation and, hence, decreased the grade and recovery of the phosphate concentrate as a result of bulk flotation beyond the optimum liberation size.

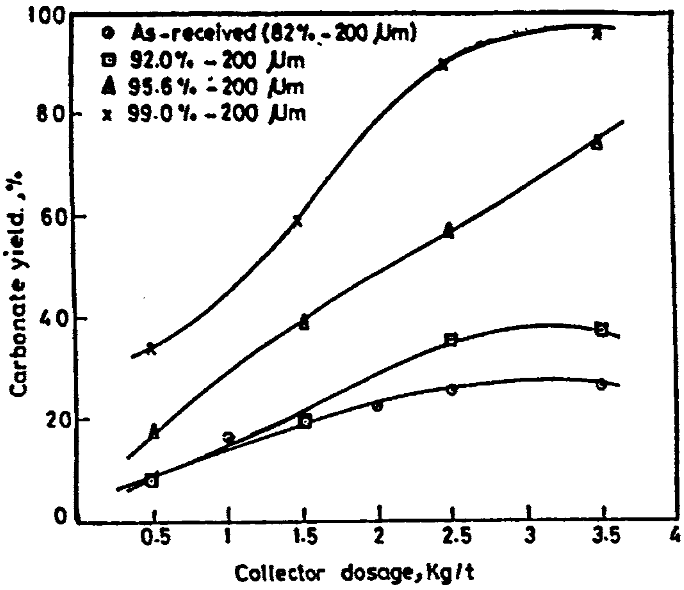

Fig. 1. Effect of particle size distribution on carbonate recovery

Table 2. Particle size distribution of different grinding times

Size fraction, µm	Weight, %			
	As-received	5 min	10 min	20 min
+200	17.97	7.64	4.40	1.10
–200 + 100	41.36	34.50	20.72	5.16
–100 + 63	28.47	23.82	12.02	6.32
–63 + 32	3.48	16.24	12.18	13.46
–32	8.72	17.80	50.68	73.96

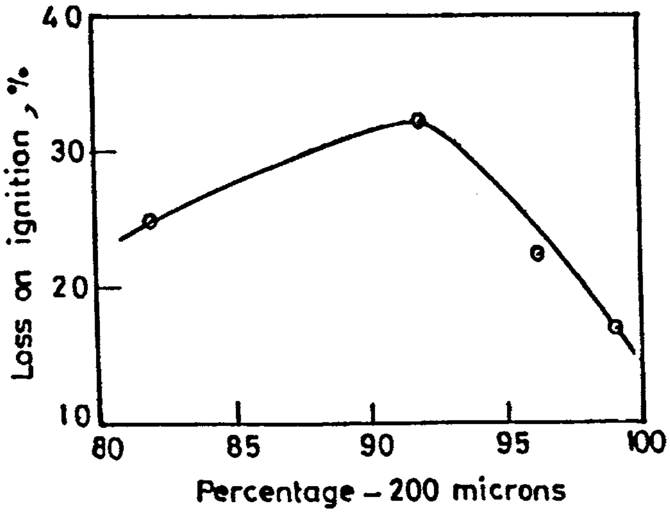

Fig. 2. Effect of particle size on loss on ignition of floated carbonates from the phosphate wastes (at 2.5 kg/t collector dosage)

From the above discussion, the size distribution of the sample 92% – 200 µm could be taken as an optimum grinding for flotation of the given material. Hence, all subsequent experiments were carried out with the material of that specific particle size distribution.

Effect of conditioning time

Studies to date have revealed that conditioning time plays a very important role in flotation. Tests conducted (at a collector dosage of 2.5 kg/t) to determine the influence of conditioning time on flotation behaviour of carbonates from phosphates yielded somewhat surprising results. It was found that the flotation recovery of carbonates remained relatively constant for all conditioning times tested. Also, the conditioning time appears to have no effect on the selectivity of carbonate flotation. Since the phosphate mineral is depressed by sulphuric acid–phosphoric acid mixture, it could be hypothesized that the collector (oleic acid) adsorbs immediately on the carbonate surfaces in a very short time and stays on the surface, as long as conditioning is on, without change. This may be attributed to the higher affinity of oleate collector to be adsorbed onto carbonate surfaces.

Effect of the mode of collector addition

Two sets of experiments were carried out at different modes of collector addition to the feed slurry to determine their effects on the flotation behaviour of carbonate gangue material. In the first set, the oleic acid (collector) was added to the feed slurry all at once (batch). While in the second set, the dosage of oleic acid was added in stages to the feed pulp. After each addition of collector amount and conditioning, the air valve was opened and the flotation continued until barren froth was observed. Then, another amount of collector was added, conditioned and floated until barren froth was obtained, and so on. In both sets of experiments, the required collector dosage was kept constant. The results of these experiments are given in Figures 3, 4 and Table 3. It can be seen from Figure 3 that beyond about 1.5 kg/ton of oleic acid addition, the flotation recovery of carbonate material was significantly enhanced through the addition of oleic acid in stages. For instance flotation recovery of only 23% was obtained when an oleic acid dosage of 2.5 kg/ton was added all at once as compared with 36% resulted from the same oleic acid dosage of 2.5 kg/ton added in

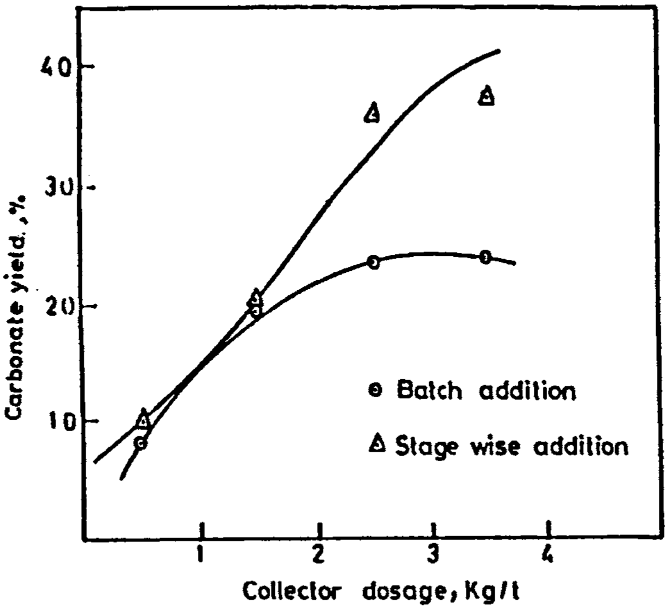

Fig. 3. Effect of mode of collector addition on the carbonate recovery

stages to feed pulp. The selectivity of carbonate flotation was also improved as shown in Fig. 4, when the collector dosage added to the feed slurry in stages. The superiority, in terms of carbonate flotation, of adding the collector dosage in stages could be interpreted as follows: The highly reactive surfaces of carbonate particles adsorb rapidly the collector (oleic acid) molecules. Moreover, there could be less share of collector between the highly reactive surfaces. Also, there will be less chance of reaction between collector ions and heavy metal ions (e.g., Ca^{2+} and Mg^{2+}) in the system which leads to the formation of heavy metal–oleate complexes. The phenomenon of forming heavy metal–oleate complexes, which will precipitate afterwards, may be more enhanced in the case of adding a large amount of collector to the feed slurry at once (batch).

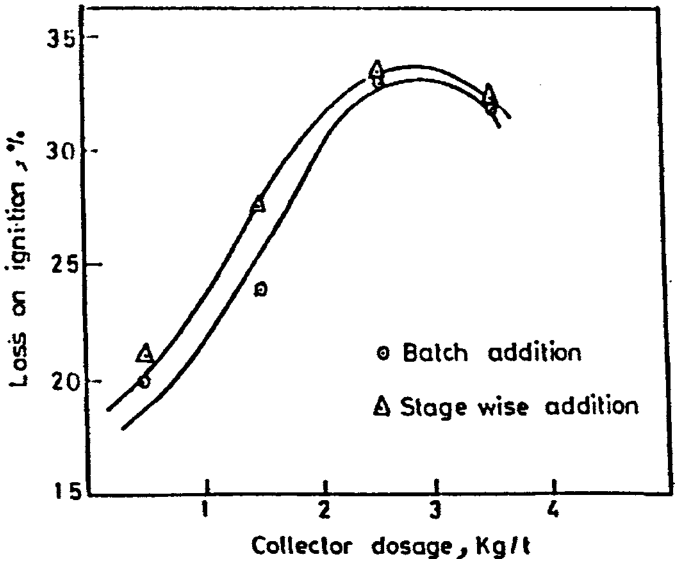

Fig. 4. Effect of mode of collector addition on the loss of ignition of floated carbonates from the phosphate wastes

Table 3 : Effect of the mode of collector addition on the flotation process

Collector dosage, kg/t	Batch addition				Stage wise addition			
	Floats		Non floats		Floats		Non floats	
	Yield, %	L.O.I%	Yield, %	L.O.I%	Yield, %	L.O.I%	Yield, %	L.O.I%
0.5	8.06	21.31	91.94	16.10	8.06	21.31	91.94	16.10
1.5	19.7	24.10	80.30	14.64	20.03	27.49	79.97	13.75
2.5	23.3	33.13	76.70	11.45	36.07	33.30	63.93	7.01

3.5	24.02	32.11	75.98	11.56	37.50	32.39	62.50	6.97

Effect of depressant type

To determine the effect of depressant type, a series of tests was performed. The results are presented in Figure 5, 6 and Table 4. It is obvious that the flotation performance of carbonate (in terms of grade) followed the order: H_3PO4 > mixture (1:1) of H_3PO_4 and H_2SO_4 > H_2SO_4 up to the reasonable collector dosage (2.5 kg/t).

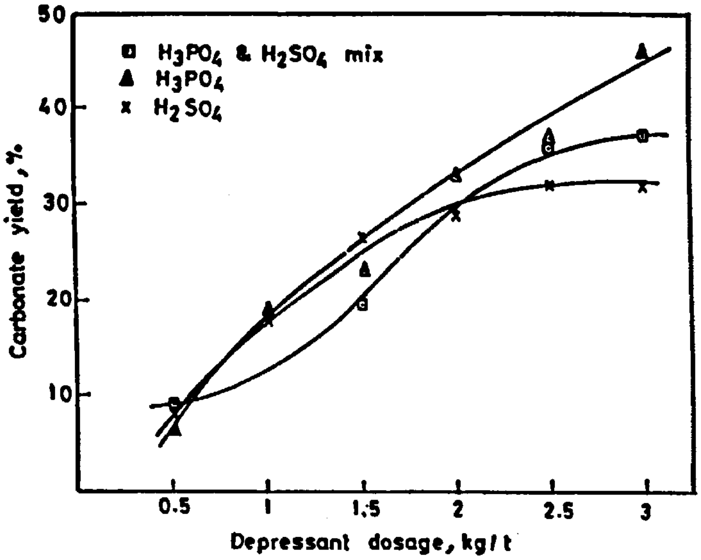

Fig. 5. Effect of depressant type on the carbonate recovery

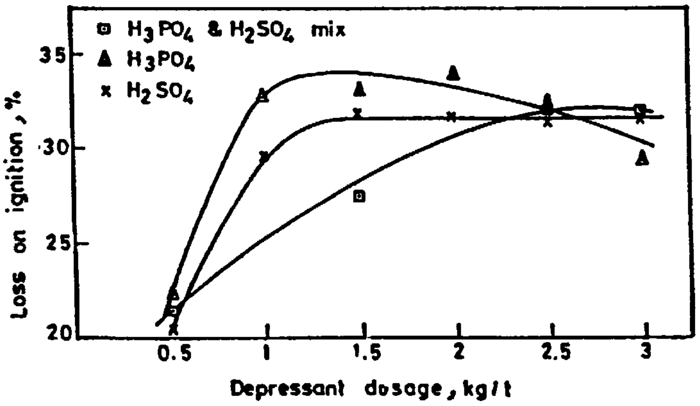

Fig. 6. Effect of depressant type on the L.O.I. of the floated carbonates from the phosphate wastes

Similar results (Abouzeid et al., 1992; Abdel, 1994; Taha et al., 1977, Abdel-Hamid, 1989; Ramadan et al., 1995) concerning other calcareous and/or dolomitic phosphate ores were obtained. It is suggested that the aqueous $CaHPO_4$ is most probably the species responsible for apatite depression. Using H_2SO_4 as a depressant will result in precipitating or complexing part of the free dissolved calciumion. This will reduce the concentrations of aqueous $CaHPO_4$ and depression of phosphate in the presence of H_3PO_4 will be better than their depression action in the presence of H_2SO_4 (Elgillani, Abouzeid, 1993). In practice, mixed solutions of H_3PO_4/H_2SO_4 could be used during conditioning and flotation in order to reduce the cost of using the expensive H_3PO_4 alone.

Table 4. Effect of depressant type on the flotation process

Collector dosage, kg/t	Floats		Non floats	
	Yield, %	L.O.I., %	Yield, %	L.O.I.,%
Depressant type: sulphuric acid				
	8.17	20.18	91.83	16.17
	17.89	29.74	82.11	13.62
	26.39	31.77	73.61	11.75
	29.08	31.62	70.92	10.3
	31.6	31.68	68.4	9.48
	31.8	31.65	68.2	9.43
Depressant type: phosphoric acid				
0.5	7.38	22.45	92.26	16.03
1.0	19.01	32.7.3	80.99	12.69
1.5	23.42	33.33	76.58	11.35
2.0	33.6	34.0	66.4	7.64
2.5	37.05	32.05	62.95	7.35
3.0	46.55	28.77	53.45	5.8
Depressant type:mixture of sulphuric and phosphoric acids (1:1)				
0.5	8.06	21.31	91.94	16.1
1.0				
1.5	20.03	27.49	79.97	13.74
2.0				
2.5	36.07	32.32	63.93	7.57
3.0	37.5	32.40	73.81	6.96

In our case of using H_3PO_4 alone a phosphate concentrate assaying 30.1% P_2O_5 (at P_2O_5 recovery of 75.3%) and 0.75% MgO was obtained from a feed material assaying 23% P_2O_5 and 3.77% MgO. The phosphate concentrate results from using H_2SO_4 as depressant, assayed 25.5% P_2O_5 with P_2O_5 recovery of 77%. When 1:1mixture of

H_3PO_4 and H_2SO_4 was used the resulted phosphate concentrate assayed 27% P_2O_5 with P_2O_5 recovery of 75%. XRD analyses of the phosphate product and the tailings produced from flotation tests conducted using H_3PO_4 as an apatite depressant showed that ankerite is almost removed from the feed material. This is a further confirmation of the chemical analysis of test products.

In summary, the test sample was ground to attain different size distributions. The optimum grind size was that of 92% – 200 microns. Oleic acid was used as a collector at various dosage levels and the optimum dose was about 2.5 kg/t. Several acidic depressants were tested to enhance the flotation response of carbonates from the phosphate values. All of the depressants improved the yield significantly, but the influence on the grade was different. On the basis of the above flotation conditions, it was possible to obtain a final phosphate concentrate with grade of 30.1% P_2O_5 and 0.75% MgO and recovery of 75.3% P_2O_5 from undeslimed feed assaying 23% P_2O_5 and 3.77% MgO.

CONCLUSIONS

The main features revealed in this study can be summarized as follows:

The amenability of reprocessing the phosphate wastes of Abu-Tartur mine generated from the magnetic separation process is very low when the wastes are leached with acetic acid solutions due to the poor reactivity of ferriferous dolomite with acetic acid.

Selective flotation of carbonate gangue material (mainly ankerite) from Abu-Tartur phosphate wastes (magnetic fraction from HGMS) using oleic acid as a collector has been successfully attempted by depressing the phosphates with phosphoric acid, sulphuric acid or a mixture of phosphoric and sulphuric acids.

For efficient use of flotation process to upgrade the phosphate waste, grinding to about 92% – 200 μm is required for adequate liberation of phosphates from its associated gangue minerals.

It was found that the mode of collector addition to slurry has an important effect on the performance of carbonate flotation. When compared with adding the collector dosage all at once, stage wise addition of the collector dosage gave superior flotation results.

Conditioning time has no influence on carbonate flotation. The optimum conditions under which an acceptable phosphate concentrate was obtained are : 92% – 200 μm particle size, two minutes conditioning time, 2.5 kg/ton collector dosage at pH value of 5.8. It was possible to obtain a phosphate concentrate of grade of 30% P_2O_5 and 0.75% MgO with recovery of 75% P_2O_5 from the feed containing 23% P_2O_5 and 3.77% MgO.

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Seifelnassr A., Ahmed A., Wzbogacanie odpadów rudy fosforytowej z Abu-Tartur w Egipcie. *Fizykochemiczne Problemy Mineralurgii*, 32, 135–147 (in English)

Badano możliwość wtórnego wzbogacania odpadów powstałych z separacji magnetycznej fosforytów ze złoża Abu-Tatur przez zastosowanie ługowania kwasem octowym i na drodze flotacji. Odpad charakteryzuje się tym, że zawiera związki żelaza oraz dolomit. Dla zoptymalizowania procesu ługowania badano wpływ takich parametrów jak wielkość ziarn, stężenie kwasu octowego, stosunek ilości kwasu do zawartości części stałych oraz czas ługowania. Miarą przebiegu procesu była wielkość strat prażenia. Przeprowadzone badania ługowania nie dały zadawalających wyników. Dobre wyniki uzyskano przy separacji fosforytów od towarzyszących zanieczyszczeń na drodze flotacji. Dla zoptymalizowania warunków prowadzenia procesu przebadano wpływ następujących parametrów: skład ziarnowy, czas mieszania, sposób dozowania odczynników oraz rodzaj stosowanych depresorów. Wyniki badań wskazują, że z nadawy o zawartości 23% P_2O_5 i 3,77% MgO istnieje możliwość otrzymania koncentratu fosforytowego zawierającego 30% P_2O_5 przy zawartości MgO 0,75% i uzysku 75% P_2O_5.