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REVERSE FLOTATION PROCESS IN DOUBLE STAGE ON THE ALGERIAN PHOSPHATE ORE TREATMENT

Purpose. In this work, the influence of flotation reagents on the separation yield of the phosphate-carbonate system is studied. The investigation is conducted by the reverse flotation process in double stage of the phosphate ores from the Jebel Onk deposit-Algeria.

Methodology. The experimental part can be divided into three aspects, a mechanical preparation of the samples, and flotation tests in the first stage followed by a double flotation and, finally, characterization of the flotation products by different physical-chemical analysis techniques.

Findings. Reverse flotation double stage process was applied on two classes of phosphate ores (C1: 40–250 and C2: 250–500 μm). The samples are constituted by phosphatic elements (apatite), cemented by a mixture of a great amount of dolomite with low amounts of quartz, clay and calcite. Flotation results showed that P_2O_5 recovery was higher for C2 compared to C1. Thus, oleic acid and double tartrate of sodium-potassium could be considered as the best reagent since the P_2O_5 recovery content corresponds to 29.27 and 31.25 % for C1 and C2, respectively. Compared to the oleic acid and pine oil reagent, the recoveries of P_2O_5 were 28.85 and 30.55 % for C1 and C2, respectively. However, when other reagents as (oleic acid + phosphoric acid) and (oleic acid + diesel fuel) were used, the recovered amount of P_2O_5 was found to be less important, 27.80 and 27.10 % for class C1, and 30.14 and 29.15 % for C2, respectively.

Originality. During this study two main axes were discussed: the first one was the influence of flotation reagents on flotation of the first and second stages, the second one was to study the effect of grain size on the flotation yields. The flotation process has been shown to be effective both in terms of depressed phosphate elements in the phosphate concentrates and in gangue floated in the flotation tailings. It can be seen that the concentrate contents of around 32 % is achieved in P_2O_5 with superior recoveries of 99 %.

Practical value. The results of analyses carried out on the flotation products showed, in most cases, that the streams are richer in phosphate elements (fluorapatite, carbonate hydroxylapatite, carbonate fluorapatite) and a low amount of carbonates (dolomite and calcite), unlike floating, mainly made up of dolomite with a small amount of phosphated elements.

Keywords: *natural phosphate, reverse flotation, carbonates, reagents*

Introduction. The phosphate ores are used mainly as fertilizers [1, 2], animal feed supplements and in the manufacturing of phosphoric acid and used in chemical, and pharmaceutical industries [3, 4], which constitutes around 80 to 90 % of the world consumption of phosphates.

Most of phosphate ores are sedimentary deposits, in the form of fluorine apatite ($3\text{Ca}_3(\text{PO}_4)_2\text{CaF}_2$), hydroxylapatite and chlorapatite, cemented by a gangue in the form of carbonates and silicates [5, 6]. Igneous rock deposits consist of fluorapatite ore, mainly carbonatites and other types of alkaline intrusions. The magmatic ores are widely of lower grade though give higher-quality enriched products with low contents of impurities [7, 8].

The development of cost-efficient enrichment processes to eliminate this gangue (silicates, carbonates, and clays) is mandatory to enhance the quality of the considerable reserves [9, 10]. The application of these enrichment methods (calcination, acid leaching, and magnetic separation) in the industry presents certain limitations and disadvantages compared to the flotation process. Froth flotation method has been identified as an important stage of the concentration process since the 1920s. It is well known that more than half of the world's phosphate in the market is produced by froth flotation [10].

Several types and structures of reagents are used in the direct and reverse flotation processes of phosphate ores such as collectors, depressants, and activating reagents. In direct flotation, phosphate ores are floated at a $\text{pH} < 7$ with commonly anionic collector and depressant/modifier for gangue elements [11]. However, in inverse flotation, siliceous gangues are float-

ed in phosphate ore at neutral pH using a cationic amine type collector [10, 12]. The phosphate treatment encounters various difficulties subdivided in three leading factors: i) PO_4^{3-} ions are highly substituted with CO_3^{2-} , OH^- , F^- , and Cl^- ions in the apatite lattice; ii) the phosphate particle surface is inhomogeneous and irregular; and iii) phosphate ores contain organic matter, require the use of higher amounts of reagents during the processing process [3]. Despite efforts to improve the efficiency of the phosphate ore flotation process, including direct flotation, which has low efficiency and high consumption of flotation reagents. Therefore, this leads to the use further efficient flotation process (reverse flotation). Reverse flotation is an effective way for obtaining a phosphate concentrates (by floating dolomite and calcite from phosphate ore) [10, 13, 14]. In low grade yield cases, the double flotation process can give an alternative solution for yielding the phosphate concentrate with a high recovery rate. In addition, phosphorite is pre-concentrated by a direct anionic flotation step, subsequently subjected to a reverse flotation step where silicates are floated in the froth fraction by using cationic collectors. The Crago double float is typically employed in Florida, USA [12, 15].

It is well known that the fatty acids are most widely used as collectors to separation apatite from dolomite by flotation [8, 13, 16]. However, the effects of different components of vegetable oil fatty acids have been examined on phosphate flotation [17]. In addition, it was demonstrated that the use linoleic acid and palmitic acid as mixed collectors facilitate phosphate flotation, achieving a high flotation efficiency for the oleic acid to linoleic acid ratio of 1.5 [13]. Moreover, the structural analysis of fatty acid collectors indicated that the flotation properties depend on the alkyl carbon chain length, unsaturated bonds number, and the bonds relative positions.

The main aim of this work consists in the treatment of the phosphate ore by the separation process of phosphates-carbonates system by reverse flotation in double stages. Besides, the influence of the flotation reagents compared to the yield separation has been examined.

Materials and methods. Materials and sample preparations.

Three main steps are performed during the preparation of raw phosphate ore before proceeding to the flotation tests: mechanical preparation, putting in pulp, and attrition/desliming.

Step 1: The raw ore extracted from Kef ES Sennoun deposit (Djebel Onk – Algeria) is subjected to crushing up to 2 mm for reducing the grain size. The obtained product is sieved through 40, 250 and 500 μm meshes. The fact that the classes (> 500 μm) and (< 40 μm) are rich in silicates and carbonates, these considered as rejects, only C1 and C2 classes are concerned in the treatment process (reverse flotation).

Step 2: The preparation of the pulp is mainly based on setting in suspension phosphate ore particles of the two classes C1 and C2 in water, with a mass ratio of 25 wt%. This quantity of solid (25 %) allows a perfect distribution of particles in the flotation cell, besides, a better adsorption of flotation reagents on the phosphate particles surface. This will allow a perfect distribution of particles in the flotation cell, hence ensuring a better and total adsorption of flotation reagents on the phosphate ore surface [6].

Step 3: The obtained samples are introduced into attrition cell in order to liberate the surfaces of oxidation layer which coats the phosphate grains. Indeed, this process allows eliminating the argillaceous and siliceous coatings of phosphate grains that influence directly to higher consumption of the flotation collector. The obtained samples in the previous step are carefully sieved by wet method on both 40 and 250 μm meshes for C1 and C2 classes, respectively.

Flotation process. Flotation reagents. The treatment scheme by reverse flotation is applied for four reagent types (R1, R2, R3 and R4) where the corresponding mass is given in Table 1. The choice of these reagents is particularly motivated by technical and economic aspects, as well as with the aptitude to the efficient and selective enrichment process [18].

Flotation tests. Reverse flotation tests in double stages of both classes C1 and C2 with contents 24.77 and 27.83 % of P_2O_5 , respectively, are realized in flotation cell acquired from Motso Mineral Industries, Inc type. Darville, PA U.S.A, which is equipped with a reservoir provided with agitation axis, fixed by the helix.

The conditioning of flotation is performed on the flotation cell in thick pulp to 25 wt% of solid. All flotation reagents were added at room temperature. After conditioning, the pulp is fed with air through the bottom of the cell; the phosphatic elements are depressed at pH = 9 (Table 1). While the carbonated gangue is floated by the oleic acid collector. The alkalinity of the pulp is maintained with adding hydroxide. However, the acidity was fixed by adding of phosphoric acid, which is generally used as a depressant for apatite.

Foam loaded of carbonates is recovered by overflow at the top of the cell. The obtained products (the floating and the flowing) constitute the carbonated waste and phosphatic concentrates, respectively, as illustrated in Fig. 1.

Investigation methods. The mineralogical phases were identified by X-ray diffraction (XRD) technique using X'Pert MPD PANalytical diffractometer equipped with $\text{CuK}\alpha$ radiation source ($\lambda = 1.5418 \text{ \AA}$) in the 2θ range 5–80° with a step size 0.025° and accounting time of 10 s. The chemical analysis of the different oxides was carried out by X-ray fluorescence (XRF) method using Philips Magix Pro PW-2440. The functional groupements were identified with Fourier-transformation infrared (FTIR) Spectroscopy analysis which was carried out by Shimadzu 8400 spectrophotometer in the range of 400–4000 cm^{-1} . The microstructure of the samples was characterized by a high-resolution scanning electron microscopy (HRSEM) using JEOL SM 840 operating at 20 kV and equipped with an energy dispersive X-ray (EDX) spectrometer for elemental chemical analysis.

Results and discussion. Reverse flotation tests in the first stage. The obtained results of reverse flotation tests in first stage for both classes C1 and C2, as well as, the depressed phosphate and floated carbonates recovery for each reagent type are summarized in Table 2. It appears that the recoveries in P_2O_5 are relatively high for class C2 compared to class C1. The best reagents corresponding to the high recovery rate in P_2O_5 for both classes are the reagents R1 and R2, reaching flowing values around 92 %. In contrast, P_2O_5 recovery rate for the flowing of reagents R3 and R4 are relatively lower around 83 % for both classes C1 and C2. From these results, it appears that the flotation concentrates during the first stage, for both classes C1 and C2 achieve reasonable contents in P_2O_5 with a mean value of about 28 and 30 %, respectively as produced from the reagents R1 and R2. Therefore, it can be highlighted that the utilization of these reagents allows obtaining a good yield, which demonstrates the remarkable role that the depressing process plays, namely double tartrate of sodium-potassium and pine oil.

Reverse flotation tests in the second stage. According to the obtained reverse flotation tests in the second stage, as given in Table 3, it is found that the recovery rate in P_2O_5 is higher for

Table 1

Dosage of different flotation reagents

Reagents	Dosage (g/t)	Reagent types			
		R1	R2	R3	R4
Oleic acid	2500	x	x	x	x
Ethanol	800	x	x	x	x
Sodium hydroxide	2000	x	x	x	x
Tartrate of sodium-potassium	1200	x	–	–	–
Diesel fuel (d = 0.82)	500	–	x	–	–
Phosphoric acid	1000	–	–	x	–
Pine oil	1000	–	–	–	x

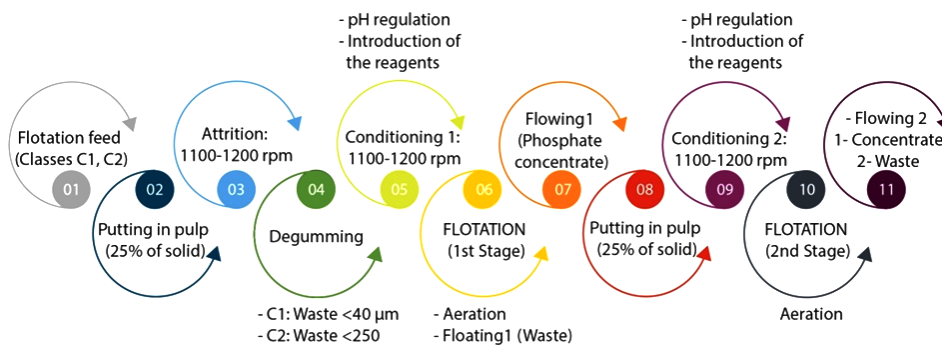


Fig. 1. Reverse flotation experimental protocol scheme of Kef ES Sennoun phosphate ores

Table 2

Variation of P_2O_5 content and recovery (Re) in the first stage of flotation products for both classes C1 and C2 with different reagents

Reagents		R1		R2		R3		R4	
Products		Floating	Flowing	Floating	Flowing	Floating	Flowing	Floating	Flowing
Mass (%)	C1	10.89	89.11	18.62	81.38	19.04	80.96	11.15	88.85
	C2	09.01	90.99	18.36	81.64	17.09	82.91	10.31	89.69
P_2O_5 (%)	C1	20.46	28.85	23.30	25.87	22.89	26.52	21.55	27.33
	C2	23.83	30.96	25.11	28.68	24.88	29.03	24.00	29.56
Re (%)	C1	92.03		82.92		83.13		90.99	
	C2	92.92		83.55		84.98		91.47	

class C2 than that for class C1. Besides, the reagent progress is well noticed by reagent R4 with P_2O_5 recovery rate reaching 94.71 % for class C1 and 95.75 for class C2. It reveals that the reagent R4 is more appropriate to reverse flotation during the second stage because of its depressing effect (Diesel fuel). Thus, the phosphate concentrates produced during the second stage of flotation from the reagents R1, R2, R3 and R4 for both classes C1 and C2 record comparable contents in P_2O_5 as obtained during the first stage, setting in the range 28–31 %. The corresponding recovery rate of P_2O_5 ranges from 85 up to 95 % for class C1 and slightly higher 88–96 % for class C2. Indeed, the presence of double tartrate of sodium-potassium in the reagent R1 and pine oil in the reagent R4, as quality of phosphate depressing, are favorably affecting the recovery of P_2O_5 . Thus, P_2O_5 recovery in flowing produced from the reagent R1 is greater than that obtained from the reagent R2.

The results of reverse flotation tests realized on four reagents in two stages showed that the best separation performance of phosphates-carbonates system can be obtained from reagents R1 and R2. This can be explained by the preponderant role playing the oleic acid collector as well as the depressing like powerful agents.

The performance of the reagents R3 and R4 is far to be significant, due to the low recovery rate of P_2O_5 . This is due to the presence of organic matters (petroleum) in the diesel fuel (re-

agent R3) and dolomitic and siliceous compounds in the phosphoric acid (reagent R4). According to these experiments, it can be concluded that reverse flotation in double stages is quite favorable and presents an excellent yield for the reagents R1 and R2 in the first stage and for the reagent R4 in the second stage. The results of reverse flotation tests in double stage obtained with both classes C1 and C2 (Tables 2 and 3), have shown that the P_2O_5 recovery rate is relatively higher in class C2 than in class C1. This can be due to the sequence time of elementary steps during the contact particle-bubble (the particle-bubble collision time, the particle-bubble attachment time and the particle-bubble aggregate stability time). Indeed, it has been shown that the drainage rate of coarse particles can differ from that of the finer particles in the upper regions of well-drained froths, since they can be trapped in the bubble films more easily. Furthermore, coarse particles can be dislodged from the bubbles more easily than fine particles for their weight. However, drainage velocity of coarse particles from the froth can also differ from that of fine particles because of interactions between the particles and the walls of the plateau borders where the drainage occurs. Thus, the behavior of certain species, influenced by the characteristics of the froth, can obscure the possible detachment of other species (like quartz).

Physico-chemical and mineralogical characterization of flotation products. Mineralogical analysis. Fig. 2 showed a large

Table 3

Variation of P_2O_5 content and recovery (Re) in the second stage of flotation products for both classes C1 and C2 with different reagents

Reagents		R1		R2		R3		R4	
Products		Floating	Flowing	Floating	Flowing	Floating	Flowing	Floating	Flowing
Mass (%)	C1	20.24	79.76	06.54	93.46	19.58	80.42	12.46	87.54
	C2	17.54	82.46	04.95	95.05	15.37	84.63	14.94	85.06
P_2O_5 (%)	C1	18.16	29.27	21.61	27.10	20.79	27.80	19.15	28.85
	C2	20.73	31.25	24.82	29.15	22.19	30.55	22.37	30.14
Re (%)	C1	85.07		94.71		84.60		91.37	
	C2	87.64		95.75		88.35		88.46	

difference among flowing and floating. Indeed, the qualitative and quantitative variation of phosphatic elements and carbonates. This variation is in agreement with the enrichment degree evolution. A net decrease in peaks intensity of carbonate phases is noticed from the reagent R1 (class C1 and C2) in the flowings, whereas the peaks intensity of phosphatic phases is much higher. Moreover, the mineralogical analysis results illustrated by the XRD patterns of Figs. 2, *a*, *b*, indicate that the flowings are richer in phosphatic phases such as fluorapatite, carbonate hydroxylapatite, carbonate fluorapatite and poorer in carbonates. However, the floating contains principally dolomite and low quantity of phosphatic phases. As shown in Fig. 2 the disappearance of carbonate phases in the flowings in favor of the appearance of these phases in the floatings is noticed. Moreover, the presence of phosphatic phases is clearly observed on the XRD patterns of the floatings. According to the results shown in Figs. 2, *a*, *b* for the reagent R1, the peaks belonging to the phosphatic phases (32.03 and 32.33°), carbonate phase derived from the calcite (30.83°) and of dolomite (30.98°) are much more pronounced in the floatings than in the flowings. This difference is associated to good separation performance of carbonates and phosphatic phases with this type of reagent.

Infrared spectroscopy analysis (FTIR). The flotation products for both classes C1 and C2 were analyzed with FTIR method, in order to show the evolution of the mineral phases depressed and floated. The FTIR spectra in Fig. 3 exhibit the phosphatic elements and carbonates bands. Both flowing and floating present absorption bands located at 1043.88 and 473.72 cm^{-1} attributed to and ions, respectively. It can be observed that the flowing presents broad bands of and . The absorption bands observed at 1432 cm^{-1} (strong) and 862 cm^{-1} (weak) are characteristic of apatitic ions, which are higher in flowings. Two absorption bands are observed at 1650 and 872 cm^{-1} (weak) and are characteristic of endogangue carbon-

ates, which are more pronounced in the floatings. A large band observed at 3444.76 cm^{-1} (strong) attributed to the OH groups, is more important in the flowings. This can be related to either structural water in different mineralogical forms ($\text{Ca}_3(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Mg}_5\text{Si}_8\text{O}_{20}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$) and/or to the presence of hydroxyapatite [19]. The spectra of class C1 bring out a weak bands among 2500 to 2600 cm^{-1} , which is attributed to the stretching bands C—H and O—H, can be related to hydrocarbons constituting the organic matter present in this class [19]. Finally, the weak peak observed at 2927.89 cm^{-1} is attributed to $-\text{CH}_2-\text{CH}_3$; this suggest the adsorption of oleic acid collector on the phosphatic particles [19, 20].

Morphological observations and elemental analysis. SEM images (Figs. 4, *c*, *d*) of flowing products show clean grains, well pruned of the gangue and in different sizes. Indeed, elemental analysis by EDX shows that the majority of grains are phosphatic elements. On the other hand, SEM images (Figs. 4, *a*, *b*, *e*, *f*) of floating products show a more pronounced heterogeneity compared to the flowings and the irregular dolomitic elements in different sizes. The latter are arranged next to the spherical grains of phosphate. The EDX elementary analysis presented in Fig. 5 shows the presence of calcic gangue (EDX-01), dolomitic (EDX-02), gypsum grains (EDX-05) and the siliceous gangue (EDX-06) well pronounced in the images (*a*, *b*, *e*, *f*) with the presence of phosphatic elements (EDX-03, and EDX-04) in the images (*c*, *d*). The flowings, particularly obtained from the reagents R1 and R2, are marked by a high proportion of phosphate grains and a well-developed exogangue. However, the floatings where the proportion of phosphatic grains is low are mainly constituted of carbonated dolomitic aggregates. This implies that these reagents favor the flotation of fine grains. On the other hand, the flowings obtained particularly from the reagents R3 and R4 are marked by the lower proportion of phosphate grains and more

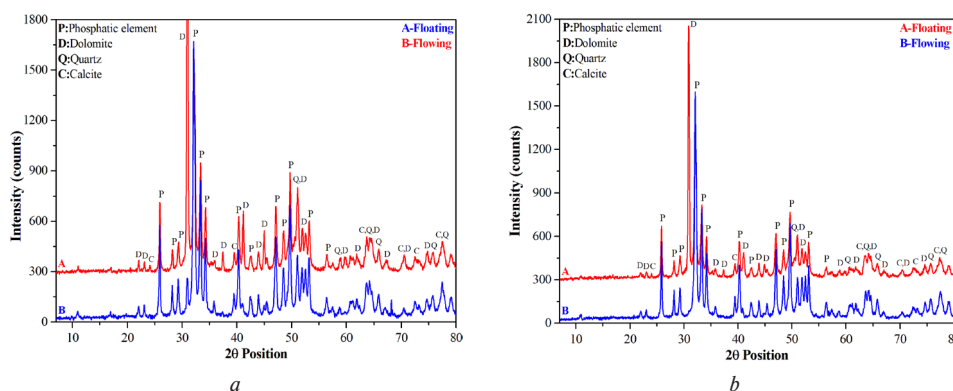


Fig. 2. X-ray diffraction patterns of flotation products obtained with the reagent R1 for both classes C1 and C2: *a* – C1, R1; *b* – C2, R1

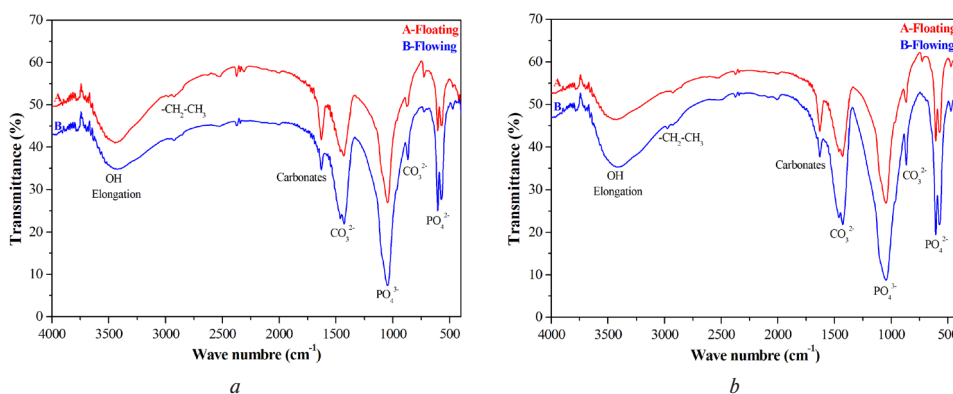


Fig. 3. C1 and C2 classes FTIR spectra of flotation products obtained with the reagent R1: *a* – C1, R1; *b* – C2, R1

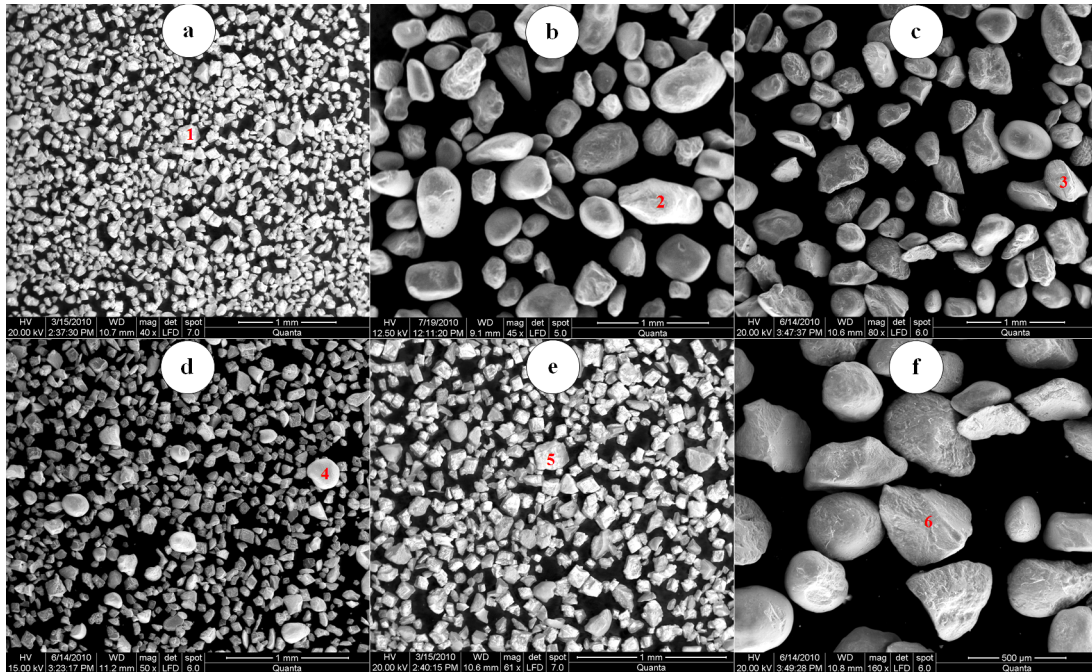


Fig. 4. SEM micrographs of flotation products obtained with different reagent types

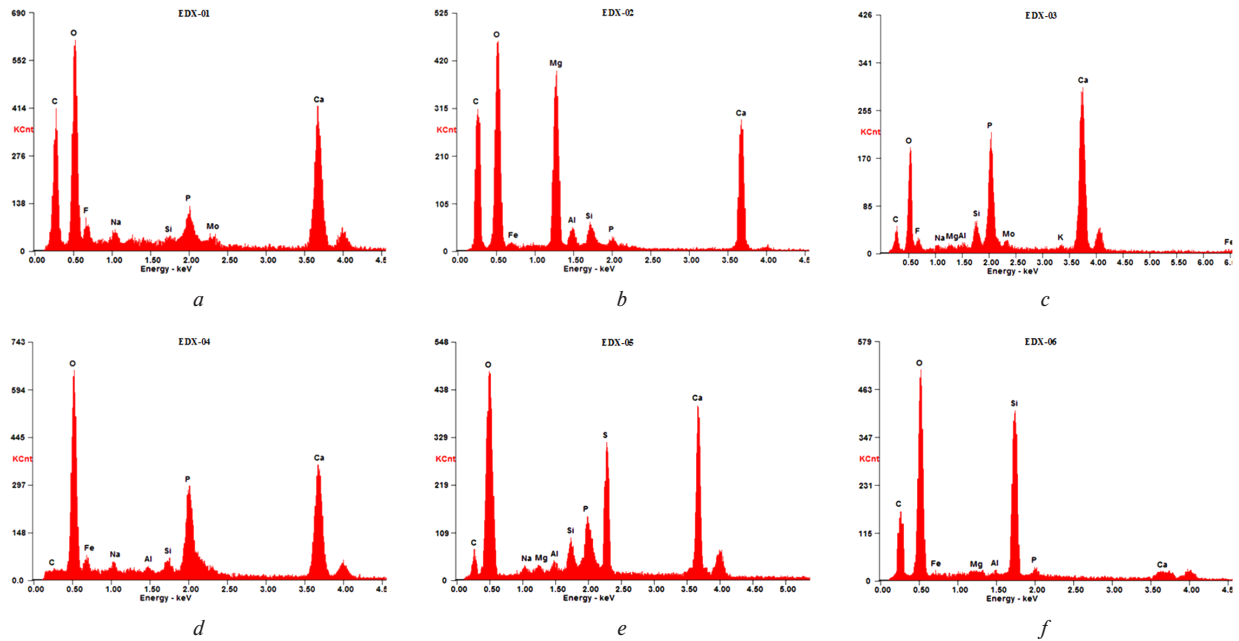


Fig. 5. EDX spectra of micrographs of Fig. 4:

EDX-01: image a (grain 1); EDX-02: image b (grain 2); EDX-03: image c (grain 3); EDX-04: image d (grain 4); EDX-05: image e (grain 5); EDX-06: image f (grain 6)

developed exogangue, which can be explained by the presence of phosphate grains at the surface of the exogangue.

Conclusions. The obtained results indicated that the flowings are rich in phosphatic elements (fluorapatite, carbonate hydroxylapatite, fluorapatite carbonate) and poor in carbonates (calcite and dolomite) and quartz. However, floating is composed of dolomite with a low amount of phosphatic elements. The results of flotation tests in double stages with both classes C1 and C2 showed that P_2O_5 recovery rate was relatively high for class C2 compared to class C1. The best reagent with high recovery rate in P_2O_5 is the reagent R1, i. e. flowings 29.27 % for class C1 and 31.25 % for class C2. The average recovery rate achieved from the reagent R2, exhibited P_2O_5 content of 28.85 and 30.55 % for the flowings of classes C1 and C2, respectively. The reagents R3 and R4 used show a low re-

covery rate, with P_2O_5 content of 27.80 and 27.10 %, respectively, in the flowings for class C1. Also, 30.14 and 29.15 % for class C2, respectively. The flotation process exhibits a high efficiency in depressed phosphatic elements in the flowings as well in floated carbonates in the floatings. Indeed, the phosphate concentrate content achieves 32 % in P_2O_5 with a high recovery rate of 95 %. Thus, the results confirm that the reverse flotation in double stages was favorable and could be an excellent economic impact.

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Двостадійний процес зворотної флотації під час переробки алжирських фосфатних руд

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Мета. У роботі досліджено вплив флотаційних реагентів на результат сепарації фосфатно-карбонатної системи. Дослідження проведено на основі двостадійного процесу зворотної флотації фосфатних руд родовища Джебель-Онк (Алжир).

Методика. Експериментальна частина може бути розділена на три аспекти: механічна підготовка зразків і флотаційний випробування на першій стадії, наступна подвійна флотація і, нарешті, визначення характеристик продуктів флотації різними методами фізико-хімічного аналізу.

Результати. До двох класів фосфатних руд (С1: 40–250 та С2: 250–500 мкм) застосовували двостадійний процес зворотної флотації. Зразки являють собою фосфатні елементи (апатити), зцементовані сумішшю великої кількості доломіту з низькою кількістю кварцу, глини й кальциту. Результати флотації показали, що вилучення Р₂О₅ було вищим для С2 у порівнянні із С1. Таким чином, олеїнову кислоту й подвійний натрієво-калієвий тарtrat можна вважати найкращими реагентами, оскільки вміст вилученого Р₂О₅ відповідає 29,27 та 31,25 % для С1 та С2 відповідно. У порівнянні з реагентами олеїнової кислоти й оснкової олії, вилучення Р₂О₅ становило 28,85 та 30,55 % для С1 та С2 відповідно. Однак, при використанні інших реагентів, таких як (олеїнова кислота + фосфорна кислота) і (олеїнова кислота + дизельне паливо), обсяг вилученого Р₂О₅ виявився менш значним – 27,80 і 27,10 % для класу С1, а також 30,14 і 29,15 % для класу С2 відповідно.

Наукова новизна. У ході цього дослідження розглядалися два основних напрями: перший – вплив флотаційних реагентів на процес флотації першої та другої стадії, другий – вивчення впливу крупності фракції на результат флотаційного випробування. Виявлено, що процес флотації є ефективним як у відношенні вилучення фосфатних елементів у фосфатних концентратах, так і у відношенні вилучення шламу, що спливає у хвості флотації. Видно, що вміст Р₂О₅ у концентраті близько 32 % досягається при вилученні 99 %.

Практична значимість. Результати аналізів продуктів флотації показали, що в більшості випадків потоки мають більший вміст фосфатних елементів (фторопатит, карбонатгідроксиapatит, карбонатний фторопатит) і менший вміст карбонатів (доломіт і кальцит), на відміну від флотації, що складається в основному з доломіту з невеликою кількістю фосфатованих елементів.

Ключові слова: природний фосфат, зворотна флотація, карбонати, реагенти

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