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ANALYSIS OF COMBINED PROCESSING METHOD OF PHOSPHATE MINERALS FROM THE KEF ESSENOUN DEPOSIT

Currently, the processing of beige and black phosphates only concerns the main sub-layers, while the other sub-layers (lower and upper for beige phosphate and the upper for black phosphate) are considered waste rock and stored near the complex. Therefore, the object of this research is the use of mixtures of three beige phosphate sub-layers and two black phosphate sub-layers as a feed fraction for the calcination and reverses flotation processes. This research work aims to characterise and treat two types of phosphate: one beige with the sub-layers (main, lower and upper) and the other black with the sub-layers (main and upper) in order to have better recovery of P_2O_5.

The working methodology consists of a series of sample preparation operations and characterization of the two types of beige and black phosphate. This characterization includes a petrographic study, a granulochemical analysis, an X-ray diffraction (XRD), a scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS), and an atomic adsorption spectrophotometry (SAA), an auto analyzer (AA), a Bernard calcimeter. These analyzes allow on the one hand to identify the mineral phases in quantitative and qualitative manner, and on the other hand to choose the process for treating phosphates from the Kef-Essenoun-Tebessa deposit.

The combined method of treatment by calcination followed by reverse flotation gave satisfactory results. The P_2O_5 content reaches 33.88 % for beige phosphate and 33.13 % for black phosphate.

The stored waste rock represents a problem for the company; this waste rock contains fairly high levels of phosphate. The treatment of the mixture of the main sub-layers with these waste rocks makes it possible to obtain a better recovery of P_2O_5, a better economic profitability of the Djebel Onk mining complex and an environmental impact in the region.

Keywords: *Beige phosphate, Black phosphate, phosphate characteristic, flotation method, calcination method, Tebessa, Algeria.*

1. Introduction

One of the main directions of development of the Algerian economy is the growth in the production of fertilizers containing phosphorus because the latter represents nearly 90 % of global demand and the food security of Algeria depends to a large extent on it [1]. To achieve this objective, it is necessary to produce concentrates adapted to the requirements of the chemical industry. These concentrates are obtained by processing phosphate ore which is the main source for manufacturing phosphate fertilizers and phosphoric acid [2].

However, phosphate rocks have three origins: sedimentary, metamorphic, and magmatic, the 80 % of world production is of sedimentary origin. Half of the proven phosphate reserves are located in North Africa (Morocco and Tunisia). China, Russia and the United States are among the major phosphate producers in the world [3].

The presence of organic matter in sedimentary phosphates negatively influences the production of phosphoric acid by the wet process [4]; in this regard knowledge of the chemical and mineralogical composition of phosphate ores is essential to anticipate the difficulties that could appear in the choice of recovery process. Researchers have described several phosphate processing methods such as mechanical methods, calcination, flotation, chemical enrichment with various acids and salts [5].

Research studies on the structural and stratigraphic plan of the Djebel Onk region were carried out by the National Mining Research Enterprise (EREM) [6]. The Algerian phosphate from the exploited Kef Essenoun deposit consists of two different types, one black and the other beige.

Algeria is one of the countries rich in phosphate in the world, but Algerian production is low compared to its significant reserves [7]. *For this aim*, in this research work

a physicochemical and mineralogical characterization of the phosphate ore of Kef Essenoun was performed, to determine the chemical composition, as well as the identification and distribution of the different mineralogical phases.

2. Materials and Methods

2.1. Study area. Algerian sedimentary phosphate is located in the South-East of the country, 100 km south of the city of Tebessa in the region of Djebel Onk (Bir El Ater); it is subdivided into five deposits: The deposit of Djemi-Djema, Djebel Onk North, Bled Hadba, Oued Betita and Kef Essenoun [8]. The studied phosphate ore in this article belongs to the Kef Essennoun deposit, the latter has 138 million tonnes of proven reserves [9], and it is composed of a thick layer of beige and black phosphorite of approximately 35 m which is divided into different sub-layers:

– lower sub-layer concerns only beige phosphate, composed of phosphorites and dolomites with a marl and clay matrix of medium and fine grain size;

– main sub-layer including beige and black phosphates composed of weak intercalations of marl and dolomite; it is friable due to the clay matrix, which is currently exploited;

– upper sub-layer encompassing the beige and black phosphates which are composed of a phosphatic dolomite with less abundant phosphates than dolomitic cement. The geological map of the Jebel Onk South region is shown in Fig. 1 [10].
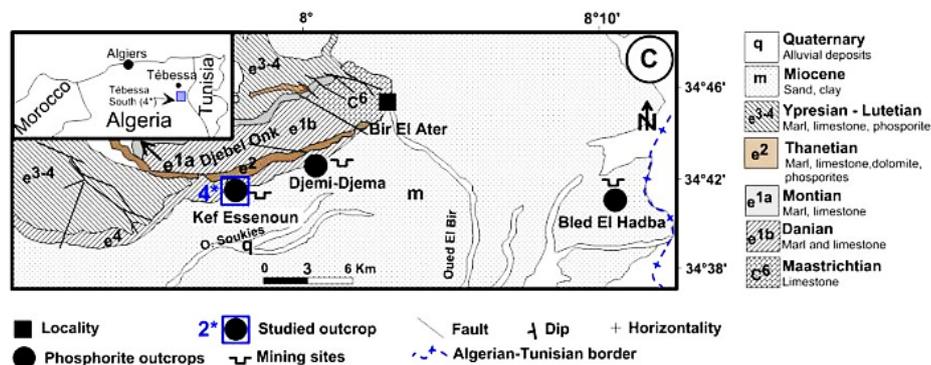

Fig. 1. Geological map of the Djebel Onk south region [10]

2.2. Samples collection. Sampling of the different types of phosphates is carried out on the three sub-layers (lower, upper, main) for the beige phosphate, and on the two sub-layers, upper and main, for the black phosphate. The mass of the collected sample is 100 kg for each type of phosphate; the objective of sampling is to obtain representative samples.

2.3. Physicochemical characterization of the samples. The samples of three beige phosphate sub-layers and two black phosphate sub-layers are mixed separately. Each mixture underwent a series of crushing, homogenization, quartering, and sieving operations in an electro-vibrating RETSCH type sieve: AS200 basic; Serial No. 22 2504 017 G; Voltage No: 230 V 50 Hz (manufactured in Germany) a quantity of 100 grams was grinded in an RM200 mortar grinder with Retsch brand ACC (manufactured in Germany) in order to carry out the following analyses:

– X-ray diffraction (XRD): which allows the identification of the mineral phases existing in the phosphate ore, in a diffractometer (OLYMPUS BTX-716 Benchtop XRD) (manufactured in USA), equipped with Cu-Kα radiation ($\lambda = 1.540598$ A°), operates at a voltage of 40 kV and a current of 30 mA; support and scanned from 0° to 55° (2θ angle range), a scanning speed of 2°/min;

– The scanning electron microscope/energy dispersive spectroscopy/X-ray spectrometry (SEM/EDS) is a tool for characterizing the chemical composition and determining the morphology of microstructures [11]; this type of analysis is conducted in a microscope: QUANTA 250 (manufactured in USA);

– Determination of major element contents by an atomic absorption spectrometer (AAS) type: Perkin Elmer precisely. AAnalyst 400; and an autoanalyzer 3 (AA): SEAL Analytical (manufactured made in USA) as well as the use of Bernard's method.

The petrography of phosphates in the sediments of Kef Essenoun has been carefully studied through the microscopic examination of thin sections taken from different stratigraphic levels of the deposit. This analysis made it possible to identify and characterize the main phosphate elements present in these facies.

2.4. Processing of phosphate ore by combined calcination – reverse flotation method

2.4.1. Calcination. The Kef Essenoun phosphate deposit contains dolomite and calcite; calcination is one of the most suitable heat treatment methods [12]. Calcination consists of removing carbon dioxide (CO_2) and decomposing carbonate minerals into calcium oxide and magnesium oxide at a temperature of 950 °C [13].

The calcination tests are carried out in a Nbertherm GmbH muffle furnace (manufactured in Germany). A quantity of 100 g of beige phosphate and 100 g of black phosphate with a particle size (-500 μm$+63$ μm) are calcined at three different temperatures, namely 800, 900, and 1000 °C for 15 minutes.

To reduce the content of organic matter contained in the Kef Essenoun phosphates, the phosphates calcined at 1000 °C are then washed using a magnetic stirrer on the 2 mag magnetic motion type machine, MIX 8 XL (manufactured in Germany).

2.4.2. Samples reverse flotation tests. To reduce the MgO and CaO contents and eliminate clays from the sample, washing the non-calcined phosphates by attrition and on a 63 μm sieve is essential.

Direct flotation is only suitable for ores with low phosphate content because it consumes a high quantity of reagents for medium to low content ores on the one hand and on the other hand it sometimes requires high temperatures as well as the difficulty of reusing recycled water [13].

The application of the reverse flotation process proves to be very effective for the processing of sedimentary phosphate rocks [2].

The flotation tests are carried out in a metso minerals serial No. P5007260 type cell (manufactured in USA), under the following conditions: pH=9, pulp density: 25 %, rotation speed: 1200 rpm; pulping time 3 minutes; conditioning time 10 minutes. The reagents used are: oleic acid as a collector (2500 g/t), sodium and potassium double tartrate as a depressant (1200 g/t), ethanol as an activator (800 g/t), sodium hydroxide as a foaming agent (2000 g/t), the pH regulators used are sodium hydroxide and phosphoric acid; the dosage of the reagents is taken by [14].

Each of the two types of phosphates (black and beige) has undergone a reverse flotation operation, for each operation the flotation cell is feed with a proportional mixture composed of two products, namely the washed raw phosphate and the washed calcined phosphate.

3. Results and Discussions

3.1. Petrographic study. Microscopic observation of the thin sections is carried out under-analyzed natural polarized light (LPNA).

Within the beige phosphates (Fig. 2, *a*, *b*, *c*) let's observe a fragile or friable composition, characterized by carbonate cement and a low organic matter content. Homogeneous phosphate grains, approximately 0.25 mm in size, represent around 50 % of the total matrix. This matrix incorporates a diversity of grains, including organic matter, coprolites, glauconite, and pellets. A late calcitic filling is also present, with some grains cemented by sparitic cement and others by microsparitic cement. Coprolites vary in shape and size, ranging from cylindrical to irregular, and measuring between

50 and 150 μm. The pellets, oval and rounded in shape, have dimensions between 40 and 70 μm.

Black phosphates (Fig. 2, *e*, *f*) have a dark gray tint due to the presence of organic matter and phosphate elements. These facies include rounded pellets, coprolites, and glauconite grains. The present elements of various sizes and shapes are cemented by sparitic and microsparitic cements. The organic matters retain a black color, while the coprolites, from automorphic to cylindrical or elongated in shape, vary in size from 50 μm to 1 mm. The glauconite, green in color and ovoid in shape, measures approximately 50 μm. The pellets, small in size, complete this composition.

The dolomite phosphate ores (Fig. 2, *d*), present in the upper layer of the deposit, are distinguished by a high concentration of dolomite cement compared to the phosphate grains. This feature gives the facies a harder and more compact consistency.

3.2. Phosphate chemical composition by atomic absorption. The obtained results from chemical analyses of beige and black phosphate samples are presented in Table 1.

Currently, the upper sub-layers of beige and black phosphates with a P_2O_5 content of 18.44 %, and 19.57 % respectively, and the lower sub-layer of beige phosphate with a P_2O_5 content: of 27.78 % are considered to be a poor ore, they are stored near mine as waste rock.

To this end, in order to minimize phosphate losses from the Djebel Onk mining complex, the characterization and treatment tests will focus on the mixture of the three beige phosphate sub-layers (lower, main, upper), also for black phosphate (main and upper).
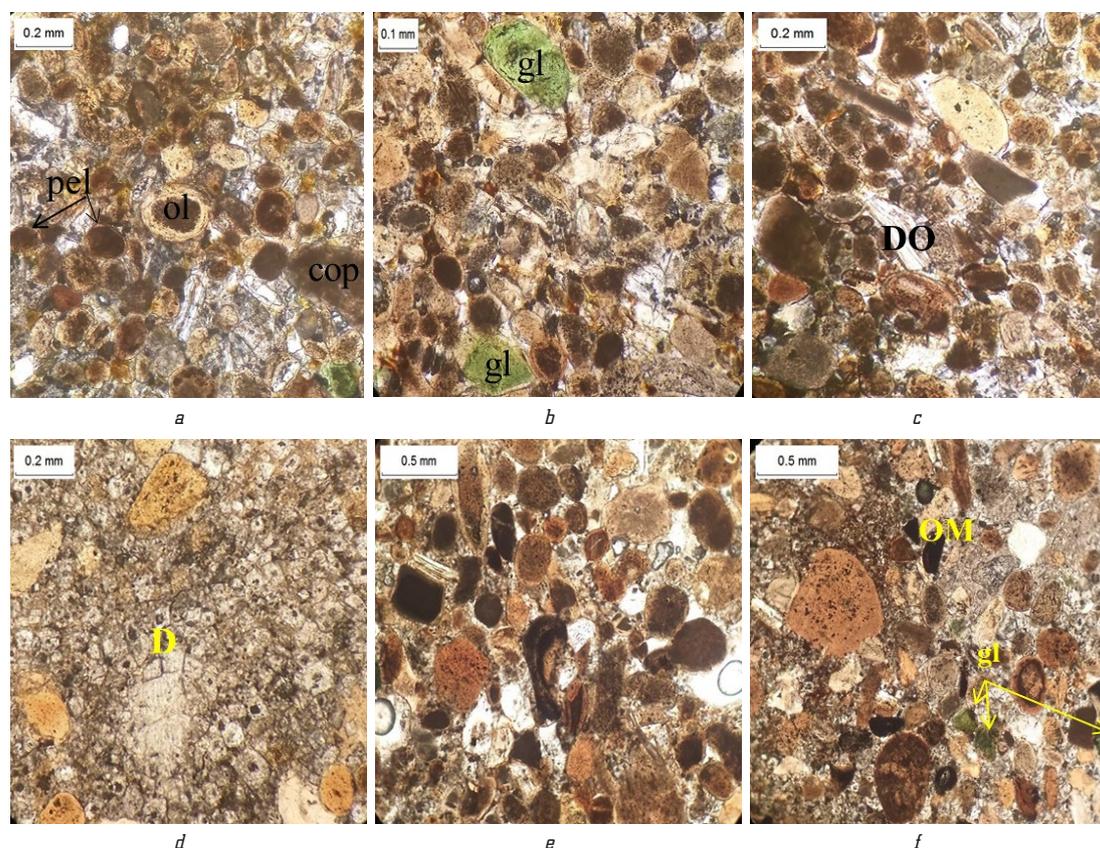

Fig. 2. Microscopic appearance of the studied phosphate minerals;
a, *b*, *c* – beige phosphates (LPNA X10); *d* – dolomitic phosphates (LPNA X10); *e*, *f* – black phosphates (LPNA X 5); cop – coprolite; pel – pellet;
gl – glauconite; BD – bone debris; D – dolomite crystals; OM – organic matter

Table 1

Chemical analysis results for beige and black phosphates

Beige phosphate				
Samples	Content (%)			
	CO_2	P_2O_5	MgO	CaO
Lower sub-layer	8.40	27.78	1.66	45.84
Main sub-layer	8.40	27.91	4.48	46.05
Upper sub-layer	20.08	18.44	1.39	30.43
Mixture of 3 sub-layers	12.05	24.58	2.98	40.56
Fraction −500 µm+63 µm	09.30	25.80	4.43	42.57
Black phosphate				
Upper sub-layer	18.40	19.57	4.64	32.29
Main sub-layer	8.40	27.91	1.94	46.05
Mixture of 2 sub-layers	13.05	23.51	3.48	38.79
Fraction −500 µm+63 µm	13.29	22.98	5.90	37.92

3.3. Granulochemical analysis of the samples. The sieved fractions of two types of beige and black phosphate are evaluated by SAA, and the results are presented in Fig. 3.

The P_2O_5 contents increase gradually with the reduction of the sieve mesh until they have maximum values in the ranges of: −0.5 mm+0.25 mm; −0.25 mm+0.125 mm; −0.125 mm+0.063 mm.

Microscopic observation of the grains of the different particle size slices of beige and black phosphate confirms that 90 % of the phosphate grains are released in these three slices and therefore this fraction is considered as the release mesh of two types of phosphate.
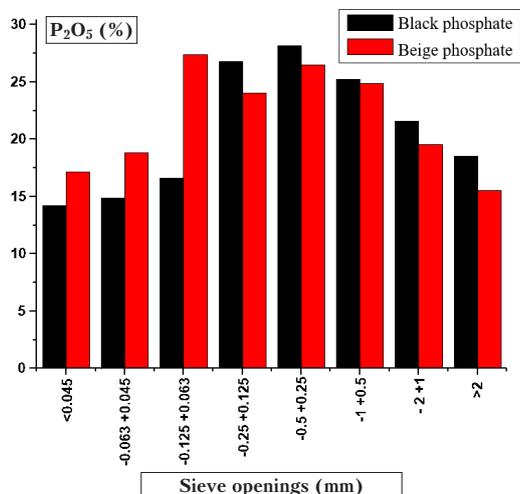

Fig. 3. Variation in P_2O_5 content according to particle size ranges

3.4. Scanning electron microscopy and EDS. The analysis of the sample of the −500 µm+63 µm fraction by SEM-EDS is carried out for beige phosphate and black phosphate. The use of SEM/EDS allows on the one hand to identify the different mineralogical phases presented in the beige and black phosphate, namely: oval and rounded pellets and cylindrical to elongated coprolites as well as dolomite crystals and bone debris and on the other hand it makes it possible to clearly determine and specify the contents of chemical elements (P, Si, Mg, Al, Na, F, C, O, S, and Ca).

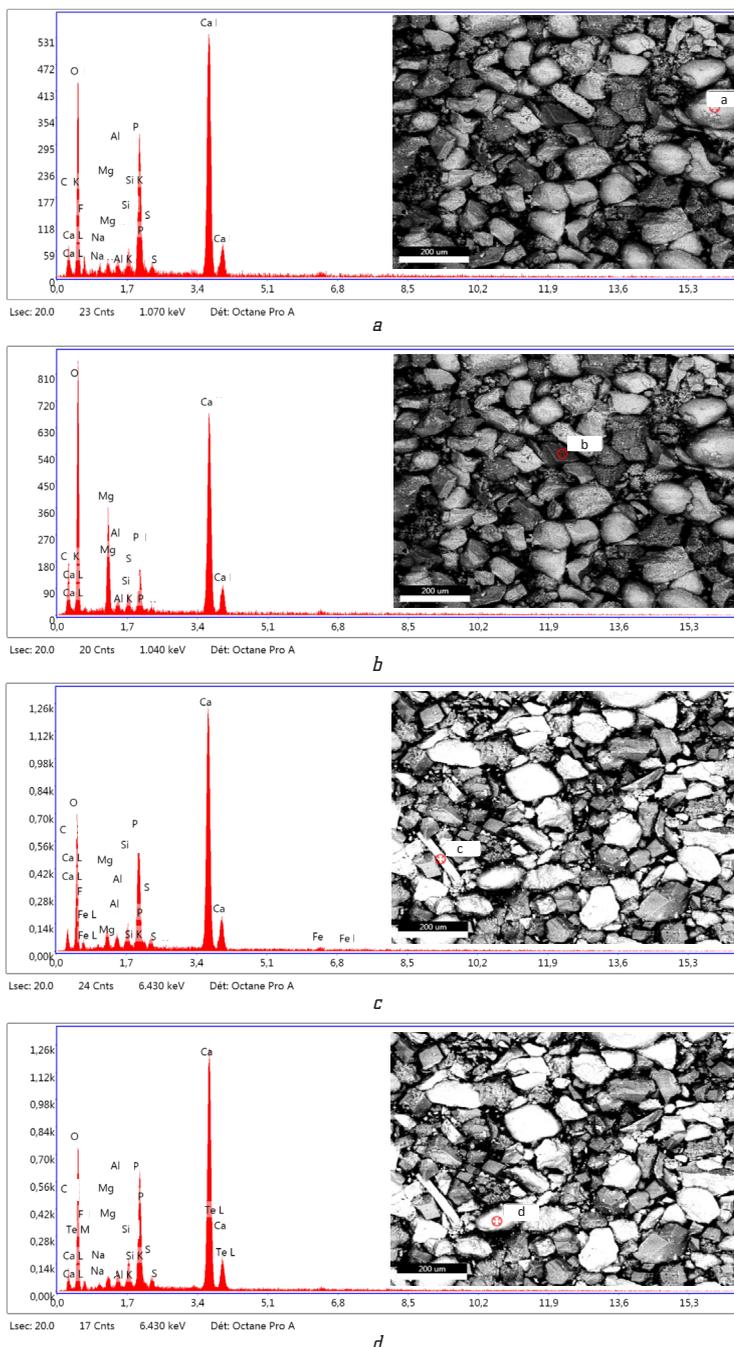The SEM/EDS results are illustrated in Fig. 4.

Fig. 4. SEM/EDS of phosphate samples: Beige:
a – Pellet, *b* – Dolomite; Black: *c* – Bone debris, *d* – Coprolite

3.5. X-ray diffraction (XRD). The use of the diffractometer makes it possible to determine the existing minerals in the beige and black phosphate, the samples are mainly composed of apatite minerals: carbonated fluorapatite $(Ca_{10}(PO_4)_5CO_3F_{1.5}(OH)_{0.5}$, fluorapatite $(Ca_5(PO_4)_3F)$, hydroxylapatite $Ca_5(PO_4)_3(OH)$, as well as the gangue minerals: dolomite $CaMg(CO_3)_2$, calcite $CaCO_3$ and quartz SiO_2.

The results of the XRD analysis of the mixture of the three beige phosphate sub-layers and two black phosphate sub-layers are presented in Fig. 5.

The results of chemical analyses of beige and black phosphates calcined at different temperatures are presented in Table 2.
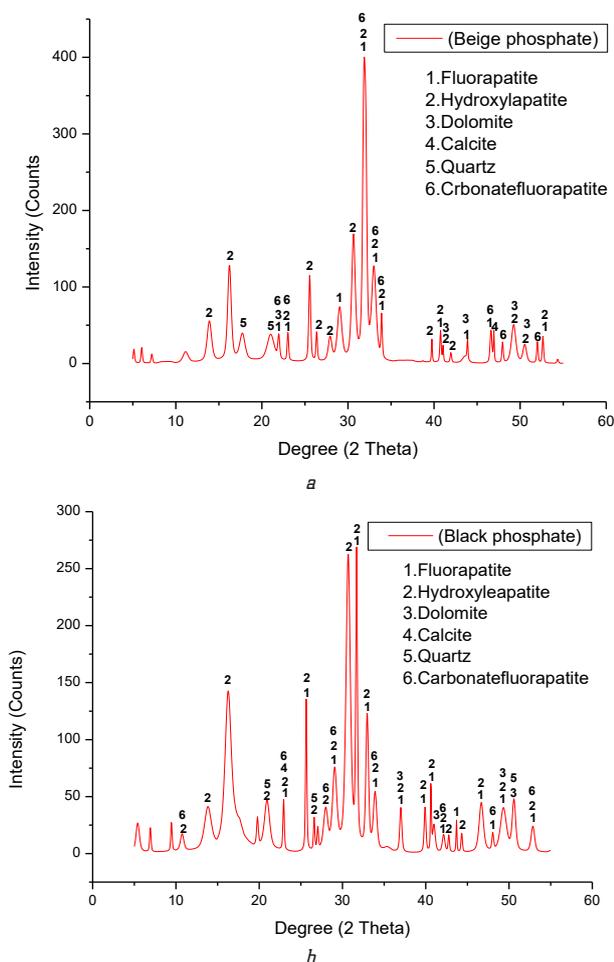
Fig. 5. XRD results of: *a* – Beige phosphate; *b* – Black phosphate

ted fluorapatite). For the gangue minerals, let's note the presence of quartz peaks in small quantities as well as the decrease in dolomite phases and the appearance of CaO and MgO peaks at 2 theta=37.36° and 42.91° for phosphate beige. For black phosphate, it is possible to observe the presence of dolomite peaks in considerable quantity as well as those of quartz and calcite, with the presence of CaO and MgO peaks at 2 theta=37.36° and 42.90°.

The presence of CaO and MgO peaks confirms the decomposition of calcium and magnesium carbonates into oxides and the disappearance of carbon dioxide CO_2.

The XRD analysis results of the calcined beige and black phosphates are presented in Fig. 6.
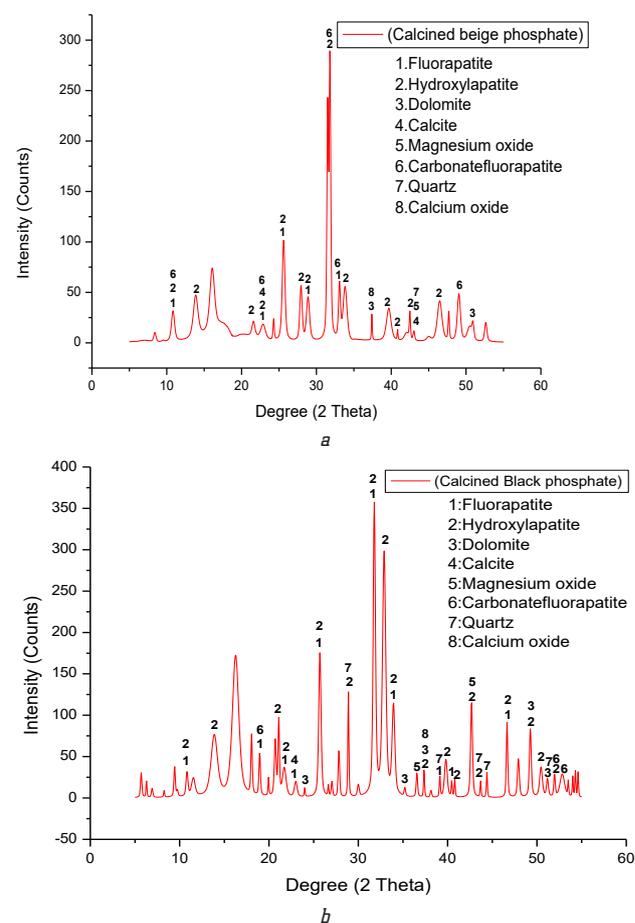

Fig. 6. XRD analysis results at 1000 °C of: *a* – Calcined beige phosphate; *b* – Calcined black phosphate

Table 2

Calcination results of beige and black phosphates

Tempera-tures (°C)	LOI (%)	Content (%)			
		P_2O_5	MgO	CO_2	CaO
Beige phosphate					
800	14.38	29.88	4.68	2.00	49.30
900	12.18	29.78	4.53	1.66	49.14
1000	14.68	29.88	4.76	1.00	49.70
Black phosphate					
800	18.03	27.08	6.83	2.16	44.68
900	17.72	27.16	7.06	1.66	44.81
1000	18.47	27.28	7.49	1.16	45.19

The best calcination results are obtained at a temperature of 1000 °C (Table 2). The P_2O_5 content for beige and black phosphate is 29.88 % and 27.28 % respectively. Let's note that with increasing temperatures the contents of MgO and CaO oxides also increase, while the CO_2 contents are gradually reduced until reaching a value of 1 % for beige phosphate and 1.16 % for black phosphate with the same temperature of 1000 °C.

The analysis of the calcined samples shows that the beige and black phosphate have similar mineral phases with different intensities, the majority of the peaks existing in the two types of phosphate reveal the presence of phosphate elements: (fluorapatite, hydroxylapatite, carbona-

The washing results of the calcined beige and black phosphates are shown in Table 3.

Table 3

Washing results of calcined products at 1000 °C

Samples	Content (%)			
	P_2O_5	MgO	CO_2	CaO
Beige phosphate	30.12	2.98	1.49	49.30
Black phosphate	27.39	4.63	1.63	45.01

Washing made it possible on the one hand to reduce the MgO contents from 4.76 % to 2.98 % and from 7.49 % to 4.63 % and on the other hand to reduce the CaO contents

from 49.70 % to 49.30 % and from 45.19 % to 45.01 % respectively for beige and black phosphates.

The washing results of the raw phosphate mixtures are shown in Table 4.

Attrition washing results of raw beige and black mixtures

Mixture of raw beige phosphate				
Sample	Content (%)			
	P_2O_5	CaO	CO_2	MgO
Unwashed beige phosphate	25.80	42.57	9.30	4.43
Washed beige phosphate	28.17	46.48	9.49	2.06
Mixture of raw black phosphate				
Unwashed black phosphate	22.98	37.92	13.29	5.90
Washed black phosphate	27.23	44.93	9.63	2.31

The results of the reverse flotation tests of the beige and black phosphates are given in Tables 5 and 6.

Results of reverse flotation tests of calcined and non-calcined beige phosphate mixtures

Calcined proportion (%)	Proportion not calcined (%)	Initial contents (%)				Contents in the flowing (%)			
		P_2O_5	MgO	CaO	CO_2	P_2O_5	MgO	CaO	CO_2
/	100	28.17	2.06	46.48	9.49	29.46	1.72	48.61	8.47
20	80	28.12	1.97	46.40	7.81	29.41	1.92	48.53	7.15
40	60	30.55	1.74	50.41	4.48	30.59	1.56	50.47	6.15
60	40	31.53	1.51	52.00	6.31	33.88	1.26	55.9	3.67
80	20	31.89	1.44	52.62	8.64	32.80	1.14	54.12	2.33
100	/	30.12	2.98	45.19	1.49	33.83	1.11	55.82	1.33

Results of reverse flotation tests of calcined and non-calcined black phosphate mixtures

Calcined proportion (%)	Proportion not calcined (%)	Initial contents (%)				Contents in the flowing (%)			
		P_2O_5	MgO	CaO	CO_2	P_2O_5	MgO	CaO	CO_2
/	100	27.23	2.31	44.93	9.63	28.15	1.99	46.45	9.47
20	80	28.05	2.34	46.28	3.16	29.01	1.61	47.87	7.47
40	60	29.21	2.09	48.20	6.31	29.59	1.94	48.82	6.83
60	40	30.14	1.94	49.73	5.00	33.00	1.84	54.45	5.16
80	20	30.86	2.06	50.92	3.65	32.20	1.82	53.13	3.32
100	/	27.39	4.63	45.19	1.63	33.13	1.33	54.66	1.66

The reverse flotation tests of the calcined and non-calcined beige mixtures made it possible to increase the P_2O_5 contents and reduce the organic matter contents.

The best results are achieved for 100 % calcined phosphate and the mixture with a proportion of 60 % calcined and 40 % non-calcined. For black phosphate, it is possible

to see that the best P_2O_5 and MgO contents are obtained in the case of 100 % calcined phosphate.

The results of XRD analysis of flowing reverse flotation product (concentrate) show the absence of the majority of peaks of gangue minerals (dolomite, quartz, calcite) as well as the dominance of peaks of phosphate phases: fluorapatite, hydroxylapatite, fluorapatite carbonated; among these peaks, the most significant are at 2 theta: 31.77°, 31.94°, 33.25° (Fig. 7).
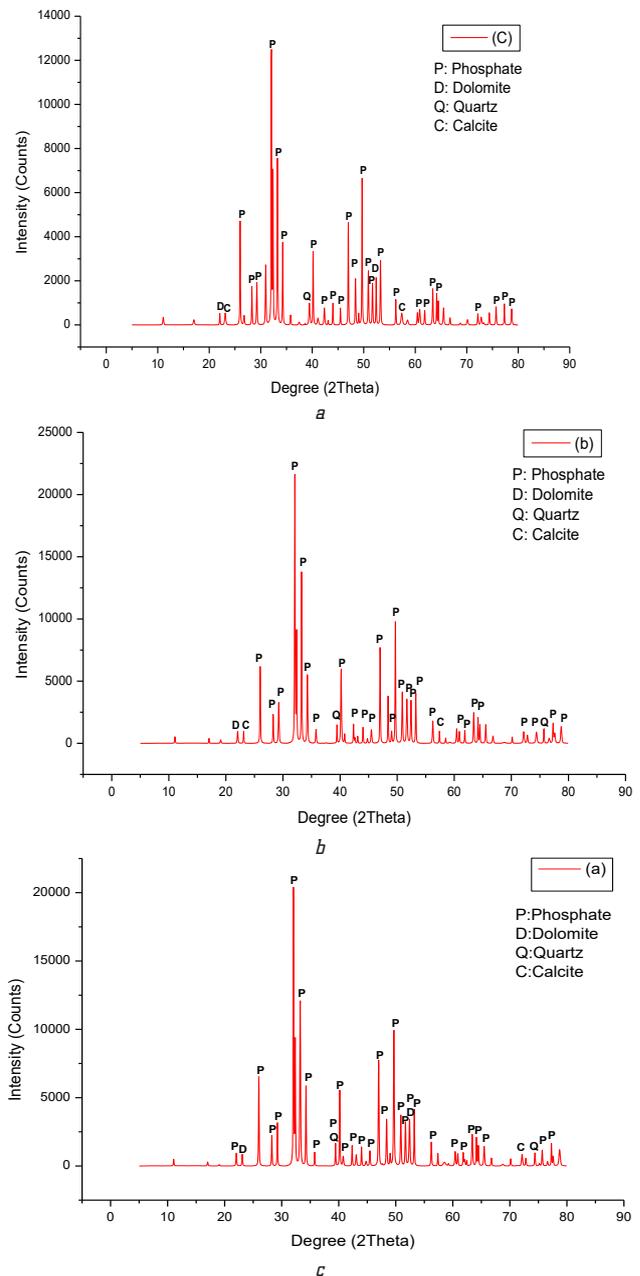

Fig. 7. XRD results of phosphate flotation products: a – 100 % calcined black phosphate; b – 100 % calcined beige phosphate; c – beige phosphate (60 % calcined, 40 % non-calcined)

3.6. Technological schema of phosphate processing. The obtained processing results allowed to develop a treatment plan for beige and black phosphates from the Kef Essenoun deposit of the Djebel Onk phosphate mining complex (Fig. 8).
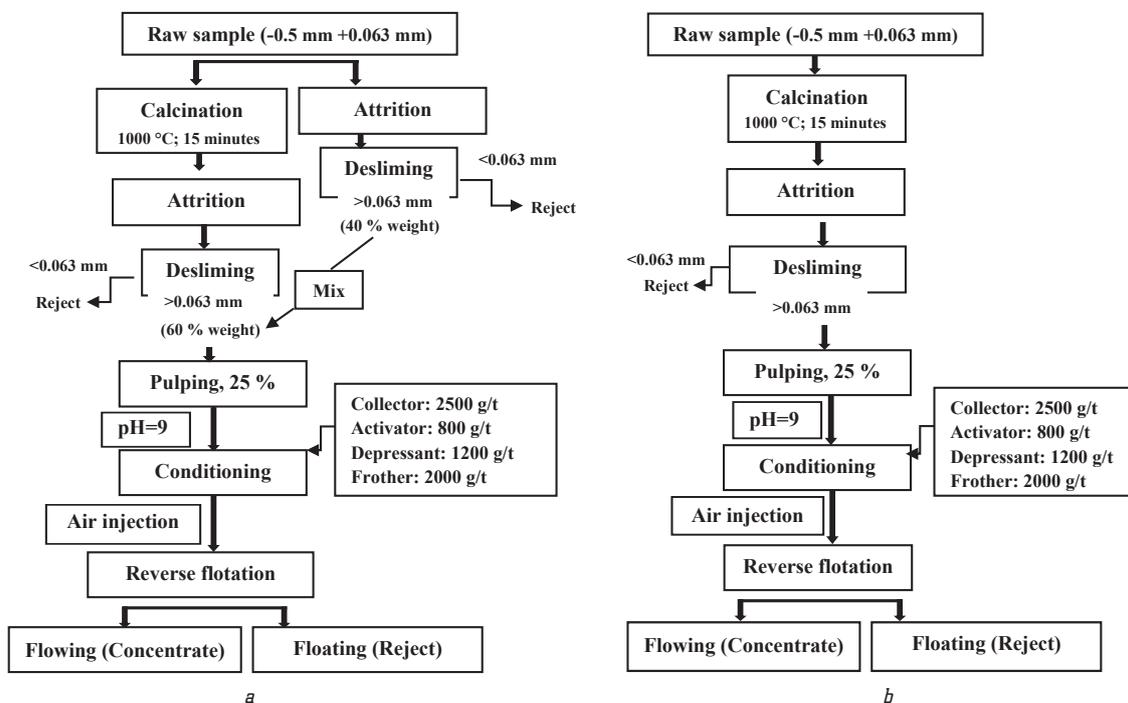
Fig. 8. Technological schema for processing of the Kef Essenoun phosphate ores: *a* – effective for the proportional mixture of beige phosphate 60 % calcined and 40 % non-calcined; *b* – effective for: 100 % calcined black phosphate; 100 % calcined beige phosphate

3.7. Limitation and directions of research development. The practical experiments performed on Algerian phosphates are based on the main layers only. The novelty of our work relates to the treatment of mixtures of phosphates from the main sub-layers processed at the mining complex with the upper and basal sub-layers currently stored and considered as poor ores, allowing to obtain high-quality concentrates. The quality of the obtained phosphate concentrate can economically impact the Djebel Onk mining complex.

The proposed method represents some negative environmental impact due to the use of chemical reagents on the one hand and the investment cost on the other hand. To remedy these inconveniences, the wastewater can be treated and recycled in the treatment circuit. The quality of the concentrate can allow to recoup the investment.

4. Conclusions

The Kef Essenoun deposit is of sedimentary origin; these proven reserves are estimated at 138 MT; it is composed of two types of phosphate, namely beige and black.

Microscopic observation of thin sections shows that the phosphate ore is mainly composed of pellets, coprolites and glauconite as well as quartz, dolomite and calcite as cements.

The particle size analysis shows that the slices (-0.5 mm$+$ $+0.25$ mm; -0.25 mm$+0.125$ mm; -0.125 mm$+0.063$ mm) present a considerable yield and are rich in P_2O_5. For this purpose, they are considered as the liberation mesh.

The analysis of the samples by SEM/EDS confirms the results of XRD, these two analyzes show that the main minerals are associated with the apatite family (fluorapatite, hydroxylapatite, carbonated fluorapatite), while the gangue minerals contain quartz, dolomite and calcite. The use of calcination allows these carbonates to be broken down into calcium oxide and magnesium oxide and to eliminate the majority of carbon dioxide (CO_2).

The valorization of phosphate ore by a combined method of calcination and reverse flotation gives encouraging results making it possible to obtain a P_2O_5 content of 33.13 % and 33.88 % from the initial contents of 22.98 % and 25.80 % respectively for black phosphate and beige.

Conflict of interest

The authors declare that they have no conflict of interest concerning this research, whether financial, personal, authorship or otherwise, that could affect the study and its results presented in this paper.

Data availability

The paper has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating this work.

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