Processing of low-grade phosphate ores of Djebel Onk mine (Algeria) with electrostatic separation method

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Abstract: Phosphate ore is an important raw material for manufacturing phosphorous fertilizers and chemicals. Although most phosphate resources cannot be marketed directly as raw material, including that from the Djebel Onk mine, due to their low P_2O_5 content and high impurity content. To obtain a quality phosphate concentrate, the enrichment of low-grade phosphate ore is therefore of great necessity. The objective of this research was to explore the use of mixtures consisting of three beige phosphate sub-layers and two black phosphate sub-layers as feed material. The objective of this study was also to identify the chemical composition and mineral phases of the ore by various analysis techniques (X-ray diffraction, X-ray fluorescence, SEM / EDS scanning electron microscope, and optical microscope). Many beneficiation techniques can be used to improve the P_2O_5 content of phosphate ores depending on their characteristics. This technique of electrostatic separation gave excellent results in terms of content and recovery for both calcined and non-calcined beige and black phosphates. The removal of organic matter and calcite is crucial in the phosphate industry, wherefore high-quality phosphoric acid can be produced more economically, while reducing acid consumption. The best-obtained results from the calcination process followed by electrostatic separation from an initial ore with a content of 31.15 % of P_2O_5 were achieved for calcined beige phosphate (under conditions: rotation speed: 30 rpm, electrical voltage: 30 kV), with content of P_2O_5 in concentrate 36.89 % and recovery of 93.91 %. For calcined black phosphate the best results were achieved under conditions: rotation speed: 50 rpm, electrical voltage: 35 kV, with content of P_2O_5 in concentrate 36.65 % and recovery of 93.88 % from an initial ore (with a content of 30.85 % P_2O_5). This study highlights the importance of the calcination treatment before the electrostatic separation operation.

Key words: beige phosphate, black phosphate, low-grade phosphate, calcination, enrichment, electrostatic separation, Djebel Onk mine, Kef Essennoun

Graphical abstract
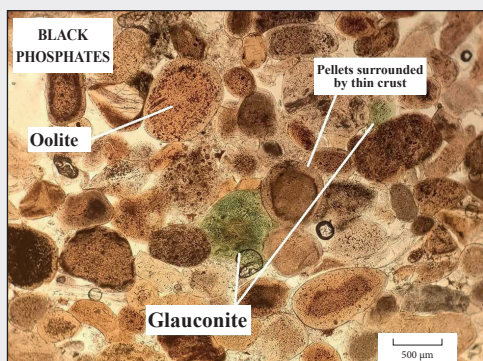
Highlights

- The study focuses on the characterization of treatment by electrostatic separation of low-grade phosphate ore from the Djebel Onk mine (Kef Essennoun – Algeria).

- The originality of presented research resides in the possible application of a combined method for the phosphate ore enrichment by calcination and electrostatic separation, which is an effective strategy for concentrating ore from a sub-arid region. The phosphate ores treated by these processes meet industrial requirements and international standards.

1   Introduction

Phosphorus is an essential element in the agricultural sector, used in the form of fertilizers and animal feed. Algeria has a significant mining industry with vast potential. In terms of Algeria's economy, mining products will certainly dominate the future. Algerian phosphate, notably that of the Tebessa region (Eastern Saharan Atlas) is particularly important, with the Djebel Onk deposit being the largest in the country, estimated at 2.2 billion tons of phosphates (USGS, 2020). The Kef Essennoun deposit is characterized by a thick layer of approximately 35 m of Upper Thanetian phosphorites, divided into three sub-layers according to the P_2O_5 and MgO contents, and is presented in two different forms, one black and the other beige (Kechiched et al., 2016). The phosphate beneficiation industry faces the challenge of economically and efficiently exploitinglow-grade phosphate ores. It is confronted with a decline in the quality of phosphate rocks, unpredictable quantities of gangues, and the need to combine different enrichment techniques (Zafar et al., 1996). Recently, many reports have been published on the improvement of low-grade phosphate ores, using both wet and dry beneficiation techniques (Bada et al., 2012).

This study focuses on the physicochemical and mineralogical characterization of phosphate ore from the Kef Essennoun mine, using various analysis techniques (X-ray diffraction, X-ray fluorescence, SEM / EDS scanning electron microscope, and optical microscope). The objective is to identify the chemical composition and mineral phases of the ore.

Depending on the climatic conditions of the region, a study was carried out to test the possibility of high-tension electrostatic separation treatment on representative samples. This technique gave excellent results in terms of content and recovery for both calcined and non-calcined beige and black phosphate. The process makes it possible, on the one hand, to reduce the volumes of waste and, on the other hand, to protect the environment from the dangers of phosphate waste.

Phosphate ore deposits containing a large amount of carbonates and significant chemical substitutions, respectively in the gangue and throughout the ore, make it difficult to concentrate phosphate elements (Kechiched et al., 2020).

The calcination of phosphate ore is a well-known processing method that increases the phosphorus (P_2O_5) content of the ore (Zieliński et al., 2023).

Natural phosphate is the fundamental component for the manufacture of phosphoric acid and phosphate fertilizers. Kef Essennoun phosphate reacts to heat treatment, which allows the purity of raw and calcined phosphates to be estimated. The P_2O_5 content increased from 28.39 % in the raw phosphate to 31.09 % in the calcined product (Bounemia et al., 2023). At elevated temperatures, fluorapatite can undergo thermal decomposition resulting in changes to its chemical and crystal structure.

Using an electrostatic separator can improve the recovery of minerals, including phosphate. The efficiency of the process allows a content and recovery of 35 % and 90.1 % respectively to be obtained (Stencel et al., 2003).

Electrostatic separation is a method used to sort materials based on their electrical properties. This technique has been applied to various materials, including granular mixtures of different sizes and compositions, such as non-conductive particles, non-conductive and conductive particles, as well as conductive particles (Fig. 3) (Zhu et al., 2023; Bendilmi et al., 2022).

The conductive particles discharge rapidly onto the grounded electrode. Simultaneously, the particles also become charged by electrostatic induction. The electric field extracts the charge carriers from the electrode and transfers them to the particles (Dascalescu et al., 1994; Idres et al., 2016).

It is fascinating to note that electrostatic ore processing offers significant benefits, both in terms of concentrate recovery and reducing stored volumes, and protecting the environment (Tiour et al., 2022; Idres et al., 2017, 2014).

2   Study area

2.1 *Geological setting and deposit location*

The Djebel Onk phosphate deposit near Bir El Ater (Fig. 1) is located in the transition zone between the eastern part of the Saharan Atlas to the north and the Saharan platform to thesouth.The boundary between these two tectonic units is marked by the southernmost Atlas fault or flexure. The Atlas Mountains belong to the Alpine belt that was formed by the convergence of the Eurasian and African plates during the Miocene period (Notholt, 1980).

The Kef Essennoun phosphate deposit belongs to the Djebel Onk mining field in northeastern Algeria. It is located approximately 10 km southwest of the town of Bir El Ater, 100 km south of the town of Tebessa and 20 km from the Algerian-Tunisian border (Fig. 1; Gadri et al., 2015).

The Kef Essennoun deposit site presents substantial differences with the Gafsa-Metlaoui basin (Tunisian phosphorite basin), Stratiform deposits of marine phosphate are well developed in the northeastern territory of Algeria. These Tertiary phosphate deposits (late Paleocene-early Eocene) belong to the large Mediterranean phosphogenic province, particularly with regard to lithologies and succession, especially in the phosphorite formation. The sedimentary lithologies consist of a 500 m thick succession dating from the Upper Cretaceous (Maastrichtian) to the
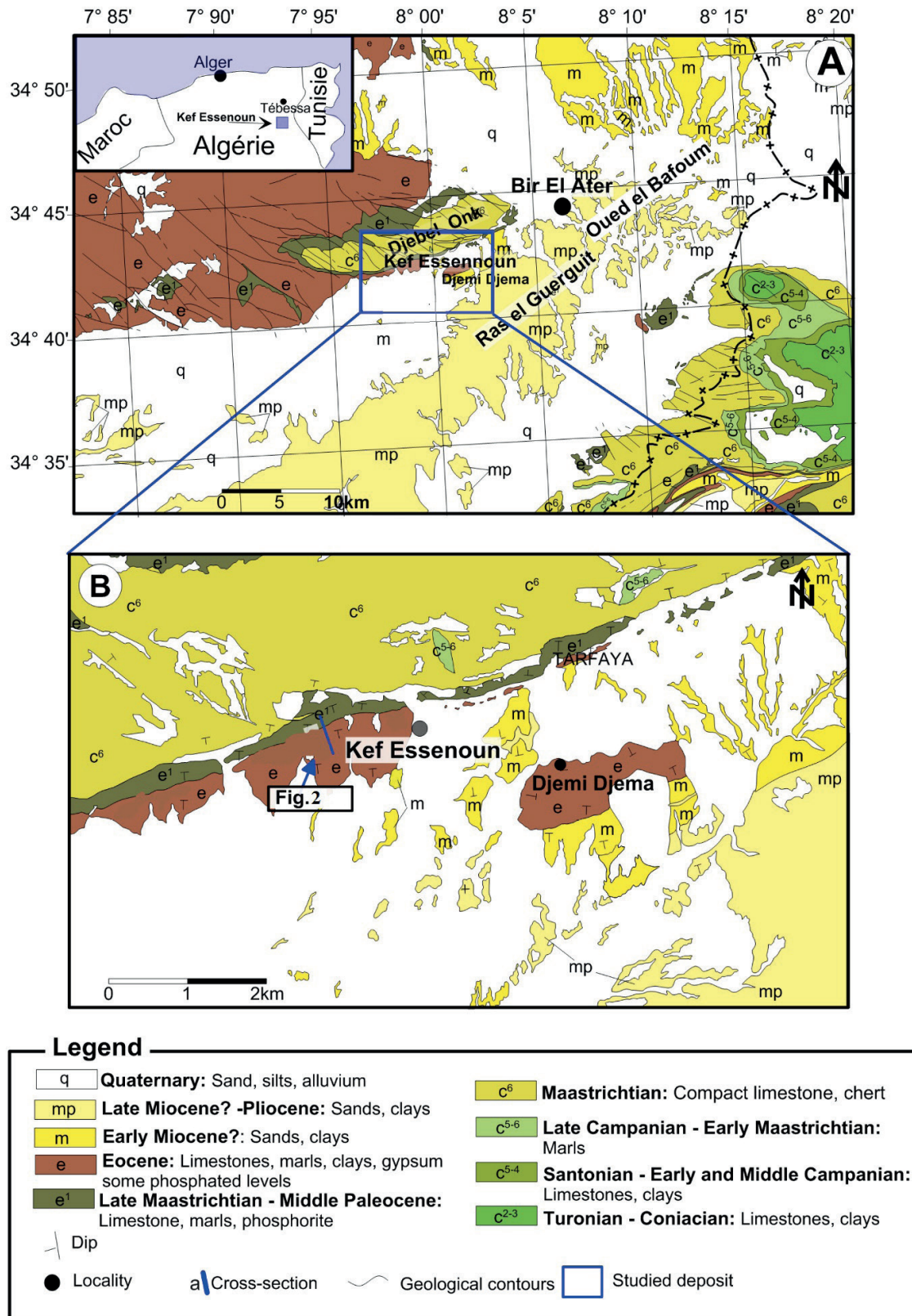
Fig. 1. (A) Geological map of the Djebel Onk mining basin including Kef Essenoun, the studied deposit in southern phosphorites (modified after ORGM, 2000). (B) (Farhaoui et al., 2022).
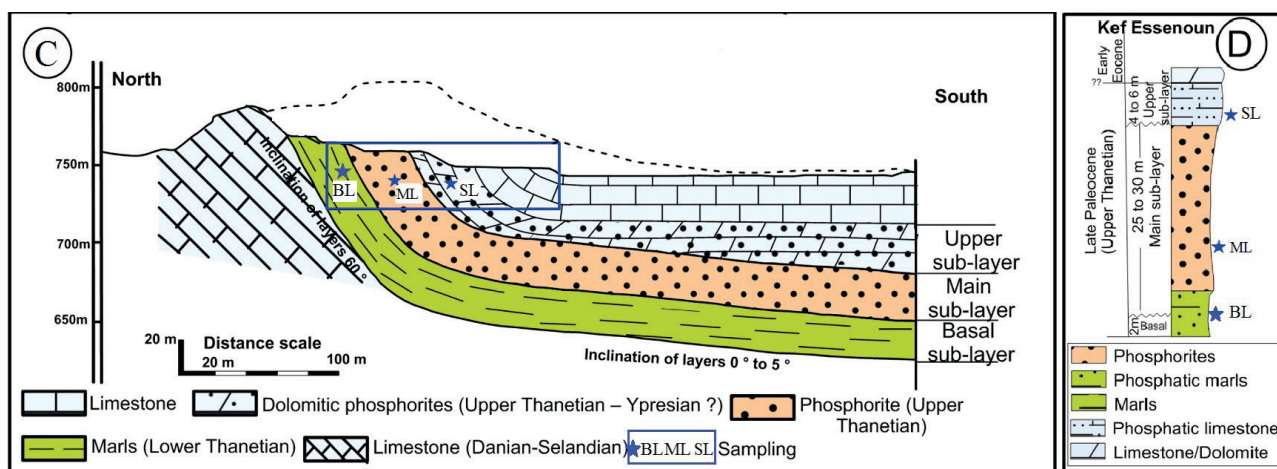
Fig. 2. Simplified geological section of the phosphorite deposit of Kef Essennoun (C); With SL: Somital sub-layer, ML: main sub-layer, BL: The basal sub-layer sample collectionpoints; Simplified lithological column from the Kef Essennoun deposit (D) (Farhaoui et al., 2022).

Middle Eocene (Lutetian) (Farhaoui et al., 2022).

The deposit is characterized by a thick layer (~ 35 m) of Upper Thanetian phosphorites, which is itself divided into three sub-layers known throughout the Djebel Onk district according to the P_2O_5 and MgO contents. From bottom to top, these sub-layers are:

- The basal sub-layer (BL): It is made up of alternating marls, phosphorites, and dolomites, with a thickness of approximately 2 m. The heterogeneous phosphorite grains are cemented by a matrix of marl and clay.
- The main sub-layer (ML): It has a thickness of 25 to 30 m and is exploited for phosphorites. The homogeneous particles of phosphorite are cemented by clay or carbonaceous cement.
- The somitalsub-layer (SL): It consists of a layer of phosphated dolomite with a relatively low P_2O_5 content (16 to 24 %) and a high MgO content (6 to 11 %). Phosphorite particles have heterogeneous particle sizes (Kechiched et al., 2016).

3 Methodology

3.1 Sample preparation

The sampling of the different types of phosphates was carried out on the three sub-layers (somital, main, basal) for beige phosphate, and on the two sub-layers (somital and main) for black phosphate, taken from the different sites. Each phosphate sample weighs 100 kg to ensure its representativeness. In this experimental study, representative samples were taken and subjected to detailed characterization.The sampling points were marked by (Fig. 2) and the samples were labeled, stored in plastic bags, then crushed to a size less than 4 mm,

air dried, homogenized, and ground for ease of further handling.

3.2 Physicochemical characterization of the samples

The samples of the three beige phosphate undercoats and the two black phosphate undercoats were mixed separately, with proportions determined according to the length of each layer. Each mixture then underwent a series of operations: homogenization, grinding, quartering, crushing to 4 mm, and sieving using a RETSCH AS200 basic electro-vibrating sieve (Serial No. 22 2504 017 G, Voltage: 230 V, 50 Hz, manufactured in Germany) the vibration amplitude used is equal to 60 and the sieving time is equal to 25 minutes. A quantity of 200 grams of each mixture was crushed in a Retsch ACC brand RM200 mortar grinder (made in Germany); the grinding time is equal to 15 minutes, to carry out the following analyses:

X-ray diffraction (XRD) can identify the mineral phases present in phosphate. This analysis is performed with an OLYMPUS BTX-716Benchtop. The bracket scans from 0° to 55° (2θ angle range) at a speed of 2°/min. The obtained diffractograms are processed using XpertHighScorePlus V3.0d software.

The petrographic analysis of phosphates in the sediments of Kef Essennoun was meticulously carried out by the microscopic examination of thin sections taken at different stratigraphic levels of the deposit.

Determination of major element contents using a Perkin Elmer atomic absorption spectrometer (AAS), model AAnalyst 400; as well as a SEAL Analytical AA3 auto-analyzer (made in the United States). Methods used include automatic spectrophotometry with the Technicon NF U42-201 auto analyzer and the high-performance M4 TORNADO micro-XRF spectrometer.

3.3 Processing of phosphate ore by the combined method of calcination and electrostatic separation

3.3.1 Calcination

The calcination tests are carried out in a muffle furnace from Nabertherm GmbH (manufactured in Germany). A quantity of 100 g of beige phosphate and 100 g of black phosphate are used, with a particle size of (–1 000) μm.

Beige and black phosphates with a particle size (–1 000 + 125) μm are calcined at three different temperatures: 850 °C, 950 °C and 1 050 °C. For each temperature, the calcination duration was 15 minutes. The samples were placed in porcelain crucibles, which were inserted into a preheated muffle furnace. This procedure aims to reduce the content of organic matter contained in the phosphates of Kef Essennoun. After the selected duration, the samples were removed and cooled in a desiccator. The calcined products were then ground in a Retsch AG brand RM200 mortar grinder (manufactured in Germany), for 15 minutes, to obtain a fraction less than 45 μm.

3.3.2 Electrostatic separation

The high-tension separator used in these tests was a Carpco panel type HP16-ll4 model. This device has been specifically designed for high-tension separation studies. Conditions, such as rotor diameters, electrode positions, voltage gradients, polarities, and field shapes, can be varied to study the effects of these variables. Each of the two types of phosphates (black and beige) was subjected to an electrostatic separation operation. The samples were dried under a vacuum between 80 and 100 °C before being introduced into the separator by a vibrating feeder. For each test, 200 g of the representative sample was introduced by a vibrating feeder and transported into the rotor separator, where the particles were subjected to different voltages between the separator electrodes 25, 30 and 35 kV, with different rotation speeds of 30, 40 and 50 rpm.

4 Results and discussions

4.1 Petrographic analysis

The most abundant phosphatic elements appear in the form of rounded to sub-rounded pellets embedded in microsparitic cement with sizes ranging from 150 to 250 μm. These elements are surrounded by a thin, clear cortex; the granules (pel) are slightly rounded and have different colors: white-gray to dark brown in the three different sublayers (Fig. 4).Glauconite grains in the main sublayer of Kef Essennoun are more abundant and have well-rounded shapes, measuring approximately 200 μm. Additionally, other features observed in the thin sections include elongated "C" shaped bone debris.

The exogangue is generally carbonated, showing phosphate grains surrounded by dolomite rhombohedra "A", while the endogangue is present either as a silica grain or as a carbonate, occurring as "A", respectively, as quartz or calcite included in the grains of phosphate and Oolites
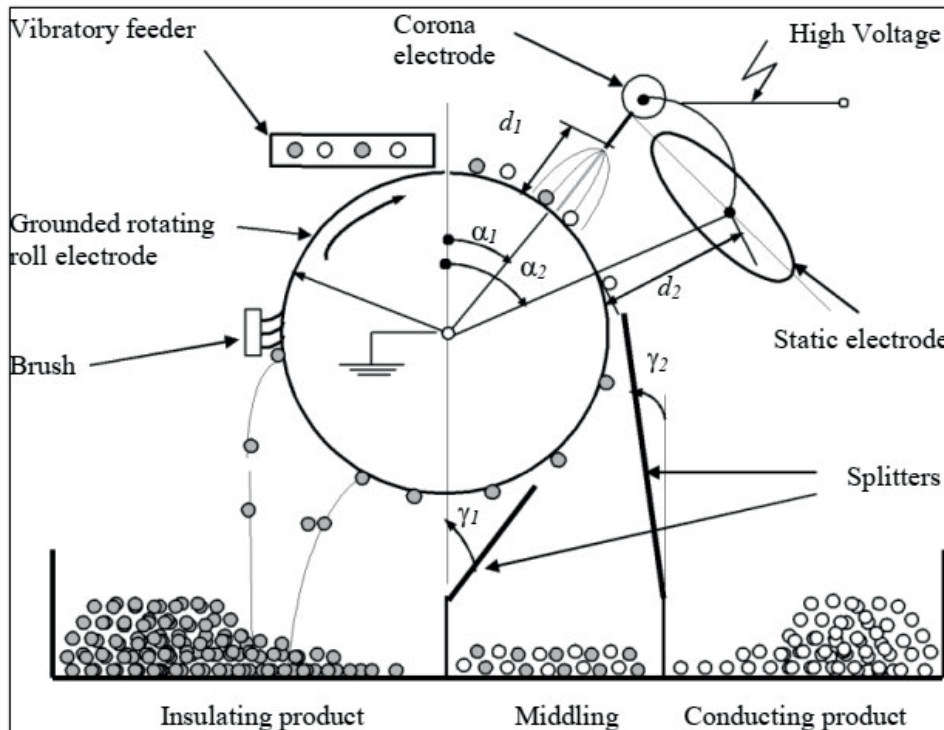

Fig. 3. Loading mechanism in the ring field α_1 and d_1 – angular and radial positions of the corona electrode; α_2 and d_2 – angular and radial positions of the electrostatic electrode; γ_1 and γ_2 – angular positioning of the dividers (Idres et al., 2016).

formed by growth and crystallization of micrometric layers of phosphate "B", "D" and "E" also show grains of glauconite (Gl) green in color at 500 μm.

Aggregates of small dolomitic "A" rhombohedra constitute the carbonate bonding phase, which generally indicates the beginning of crystallization of the micritic material containing all the impurities of the original mud. The coprolites (Cop) have a cylindrical, elongated morphology "A" and "C".

4.2 Chemical composition of phosphates

It is interesting to note how analytical techniques have made it possible to reveal relationships between petrographic and mineralogical phases, as well as the particle size distribution and the evolution of chemical contents in minerals. The homogenization method developed for the three sub-layers offers a variety of mixtures meeting the usability criteria. This approach aims to provide a higher quality phosphate concentrate.
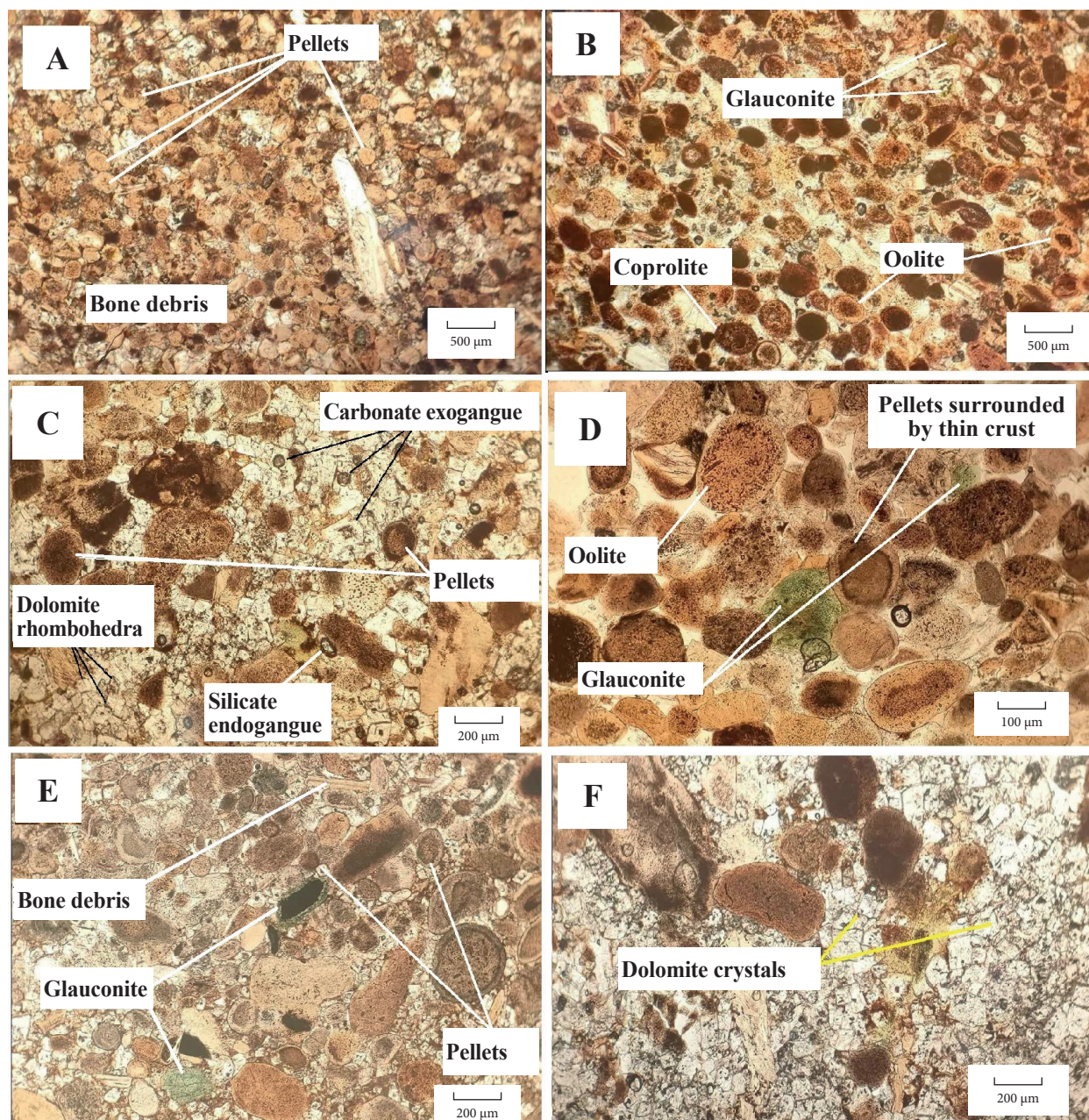

Fig. 4. Microscopic observation (LPNA X10) of the phosphate minerals studied; A, B, C – beige phosphates (somital, main, and basal); D, E – black phosphates (main and somital layer) F – dolomitic phosphates of the summit layer: dolomite crystals; glauconite, Oolite; E – bone debris; coprolite; pellet; organic material.

In our study, a collective homogenization of beige and black phosphates is proposed according to the percentage of reserves, covering the entire deposit. This approach could have a significant impact on the efficiency of mining and on the socio-economic and environmental aspects related to phosphate extraction.

The results of thechemical analysesof the beige and black phosphate samples are presented in tables 1 and 2.

In order to minimize phosphate losses from the Djebel Onk mining complex, the characterization and processing tests will focus on the mixture of the three beige phosphate sub-layers (basal, main, somital), as well as on black phosphate (main and somital).

Tab. 1

Basic chemical composition (P_2O_5, CO_2 and MgO) according to particle size ranges (SL, ML and BL) in beige phosphate

Slices [μm]	P_2O_5 [%]			MgO [%]			CaO [%]			CO_2 [%]		
	SL	ML	BL	SL	ML	BL	SL	ML	BL	SL	ML	BL
> 2 000	17.03	24.74	22.42	4.64	4.44	3.65	28.10	40.82	36.99	20.60	11.04	15.28
−2 000 + 1 000	21.09	23.56	20.96	3.81	2.82	3.98	34.80	38.87	37.88	15.28	12.29	16.61
−1 000 + 500	25.14	24.64	23.31	2.82	2.32	4.98	41.48	40.66	51.15	12.96	11.04	13.32
−500 + 250	25.26	28.51	23.42	2.82	1.33	1.82	41.68	47.04	38.64	10.30	7.31	13.62
−250 + 125	20.00	29.37	24.14	3.64	1.03	1.99	33.00	48.46	46.43	10.96	6.64	8.31
−125 + 63	17.89	23.96	24.66	4.64	1.99	2.98	29.52	39.53	40.69	16.61	8.31	9.97
−63 + 45	16.31	21.41	20.29	4.64	2.98	2.49	26.91	35.33	33.48	15.95	11.63	12.29
−45 + 0	18.75	18.29	16.47	3.98	2.49	2.49	30.94	30.18	27.18	14.62	9.97	11.04

Tab. 2

Basic chemical composition (P_2O_5, CO_2 and MgO) according to particle size ranges (SL and ML) in black phosphate

Classes [mm]	P_2O_5 [%]		MgO [%]		CaO [%]		CO_2 [%]	
	SL	ML	SL	ML	SL	ML	SL	ML
> 2 000	**17.49**	**22.53**	4.47	3.48	28.86	37.17	19.27	14.62
−2 000 + 1 000	**20.17**	**25.53**	3.98	3.98	33.28	42.12	16.94	10.30
−1 000 + 500	**22.71**	**27.45**	3.48	1.66	37.47	45.29	14.62	8.31
−500 + 250	**25.37**	**29.23**	2.82	0.96	41.86	48.23	10.96	7.31
−250 + 125	**26.21**	**26.85**	1.99	1.99	43.25	44.30	14.62	7.64
−125 + 63	**27.34**	**18.85**	1.56	3.32	45.11	31.10	20.60	11.96
−63 + 45	**22.23**	**18.95**	3.65	3.81	36.68	31.26	23.59	13.28
−45 + 0	**27.32**	**16.67**	1.82	2.82	45.08	27.50	23.25	9.96

Tab. 3

Basic chemical composition of particular grain fractions of sub-layer mixture (somital, main, basal) for beige phosphate and for black phosphate (somital and main)

Slices [µm]	Beige phosphate				Black phosphate			
	P_2O_5 [%]	MgO [%]	CaO [%]	CO_2 [%]	P_2O_5 [%]	MgO [%]	CaO [%]	CO_2 [%]
> 2 000	**21.04**	4.24	35.30	15.64	**20.01**	3.98	33.02	16.95
–2 000 + 1 000	**21.87**	3.54	37.18	14.71	**22.85**	3.98	37.70	13.62
–1 000 + 500	**24.36**	3.37	44.43	12.44	**25.08**	2.57	41.38	11.47
–500 + 250	**25.73**	1.99	42.39	10.41	**27.30**	1.89	45.05	9.30
–250 + 125	**25.84**	2.22	42.63	8.63	**26.53**	1.99	43.76	11.13
–125 + 63	**22.17**	3.20	36.58	11.63	**23.53**	2.44	38.11	16.28
–63 + 45	**19.34**	3.37	31.91	13.29	**20.59**	3.73	33.97	18.35
–45 + 0	**17.84**	2.99	29.43	11.88	**22.00**	2.32	36.29	16.61

Tab. 4

Basic chemical composition of sub-layer (somital, main and basal) for beige phosphate and for black phosphate (somital and main)

Beige phosphate				
Sample	Contents [%]			
	P_2O_5	MgO	CO_2	CaO
Somital (SL)	**24.60**	2.58	12.36	39.60
Main (ML)	**27.07**	2.10	9.64	44.66
Basal (BL)	**20.86**	3.32	12.79	34.41
Mixture of the three sub-layers	**27.02**	3.68	9.83	35.40
Slice	**30.20**	2.35	9.60	55.40
Black phosphate				
Somital	**23.05**	3.65	8.64	38.03
Main (ML)	**27.93**	1.94	8.40	46.08
Mixture of the two sub	**26.59**	3.44	9.97	34.83
Slice	**30.8**	2.82	9.30	56.0

*Slice (–1 000 + 125) µm = mixture of three slice fractions (+ 500 µm + 250 µm + 125 µm).
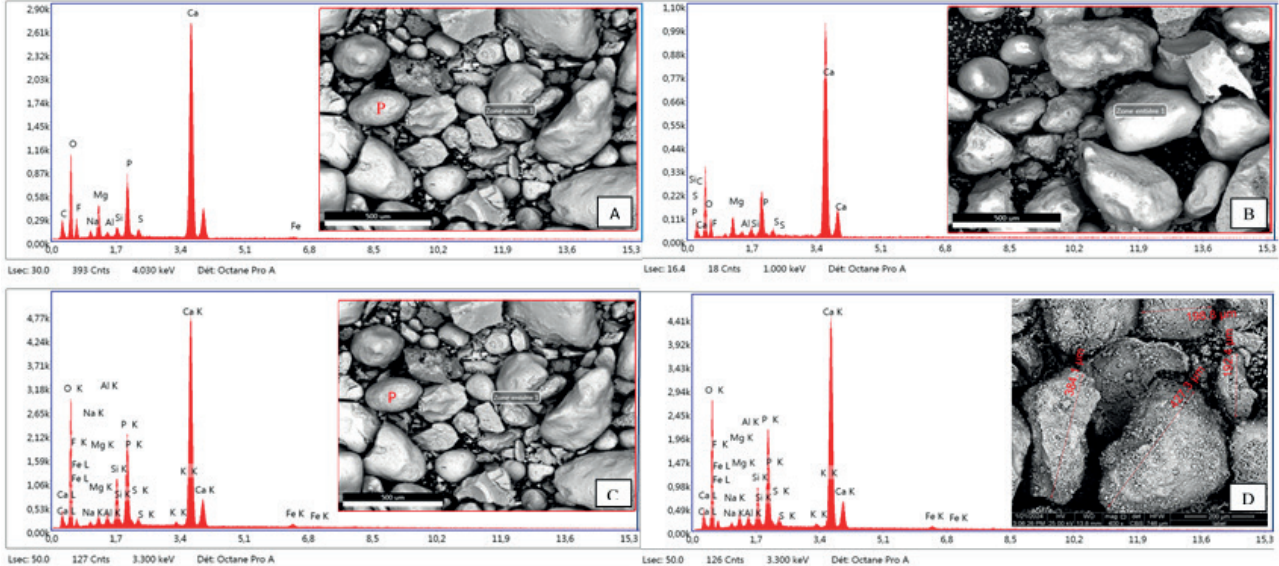

Fig. 5. SEM / EDS observation of beige "A" and black "B" phosphate before calcination and "C" and "D" after calcination (A; B). EDS spectrum showing the abundance of Ca, P and F in the phosphate particles and the high contents of O and Si in the matrix (C; D). EDS analysis showing that the abundance of Ca, O, Si, decrease after calcination. P – pellets, Cop – Coprolite, Ol – Oolite.

The upper sub-layers (somital) of the beige and black phosphates have a P_2O_5 content of 24.60 % and 23.05 %, respectively. The basal sub-layer of the beige phosphate has a P_2O_5 content of 20.86 %. These layers are considered poor ore and are stored near the mine as waste rock.

4.3 Effect of calcination process by sem / eds analysis

Analysis of the sample of beige and black phosphate by SEM is carried out for raw beige phosphate and black phosphate after calcination. The use of SEM / EDS is essential to characterize raw beige and black phosphates,

as well as to evaluate the efficiency of the calcination process.

The obtained EDS spectra for the released phosphate sample show the constituent elements of the released phosphate sample such as C, O, Mg, Al, Si, P, Mo, Ca, and Fe in different ratios. The content of calcium, oxygen, and phosphorus is the highest; however, the EDS spectrum shows that the abundance of Ca, O, and Si is reduced after calcination. The SEM / EDS results before and after calcinations are shown in (Fig. 5).

4.4 X-ray diffraction (XRD)

The mineralogical study carried out by XRD revealed that apatite is a highly substituted francolite, in OH ions (hydroxylapatite) and F ions (fluorapatite). In total, the intensity of the peaks is characteristic of the phosphatic elements and those of the gangue. The results are reported in (Fig. 6).The XRD results shows the presence of fluorapatite and dolomite as main minerals. Apatite, carbonated fluorapatite $(Ca_{10}(PO_4)_5CO_3F_{1,5}(OH)_{0,5}$ as well as the gangue minerals dolomite $CaMg(CO_3)_2$ calcite $CaCO_3$ and quartz SiO_2 have been discovered and form the matrix of the phosphorite grains, however, are present in small quantities. These findings reveal significant differences for the three phosphate sub-layers.

Indeed, the ore of the main layer is the richest in fluorapatite, hydroxylapatite, and the poorest in calcite compared to the two other layers. However, the lower layer is the poorest in phosphate minerals and richest in dolomite compared to the upper and main layers. The presence of dolomite is evident in the upper and lower layers. Quartz is more abundant in the upper layer than in the main and lower layers. Identifying the mineralogical composition and quantity of each mineral in the different phosphate layers can suggest the most appropriate ore processing.

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.

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°.These results therefore clearly confirm the presence of phosphate in our samples.
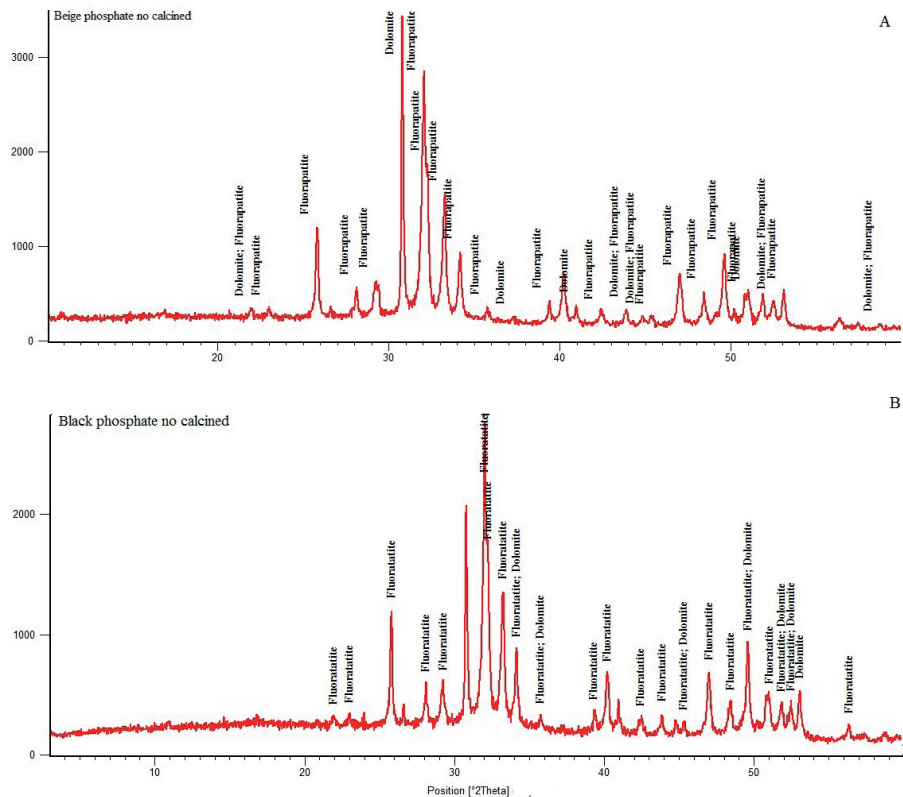

Fig. 6. XRD results of: A – beige phosphate, B – black phosphate (before calcination).
The chemical analysis results of the major elements of the calcination of beige and black phosphates are shown in Tab. 5.

Tab. 5

Results of chemical analysis of the major elements of the calcination of beige and black phosphates

Beige phosphate					
Temperature [°C]	L.O.I [%]	Contents [%]			
		P_2O_5	MgO	CaO	CO_2
0	/	27.02	3.68	35.40	9.97
850	13.61	30.31	3.365	39.70	2.49
950	14.60	31.03	3.979	40.65	1.33
1	15.19	31.15	3.782	40.80	1.00
Black phosphate					
0	/	26.59	3.44	34.83	9.30
850	15.20	26.59	3.448	34.83	9.30
950	16.30	30.79	3.680	40.33	1.49
1	17.37	30.85	3.40	40.41	1.16

Note that with the increase in temperature, the contents of MgO and CaO oxides also increase for beige phosphate (3.78 % and 40.80 %) andfor black phosphate (3.40 % and 40.41 %), respectively. On the other hand, the CO_2 contents gradually decrease until reaching a value of 1 % for beige phosphate and 1.16 % for black phosphate.

The removal of organic matter and calcite is crucial in the phosphate industry. By doing this, high-quality phosphoric acid can be produced more economically, while reducing acid consumption. This fits perfectly into an approach to sustainable development and environmental preservation.

4.5 Treatment using electrostatic separation

Two types of calcined and non-calcined samples were used for the electrostatic separation experiments; with a content of P_2O_5 approximately 31.5 % for beige phosphate and 30.85 % for black phosphate after calcination and 27.02 % and 26.59 % P_2O_5 for beige and black phosphate, in order, without calcination step. A representative sample of the calcination concentrate was prepared for electrostatic separation.

The results of the high-tension electrostatic separation tests (beige and black phosphate calcined and without calcination) are shown in Tabs. 6 and 7.

The best results in X-ray fluo-rescence (XRF) are obtained with the following parameters:
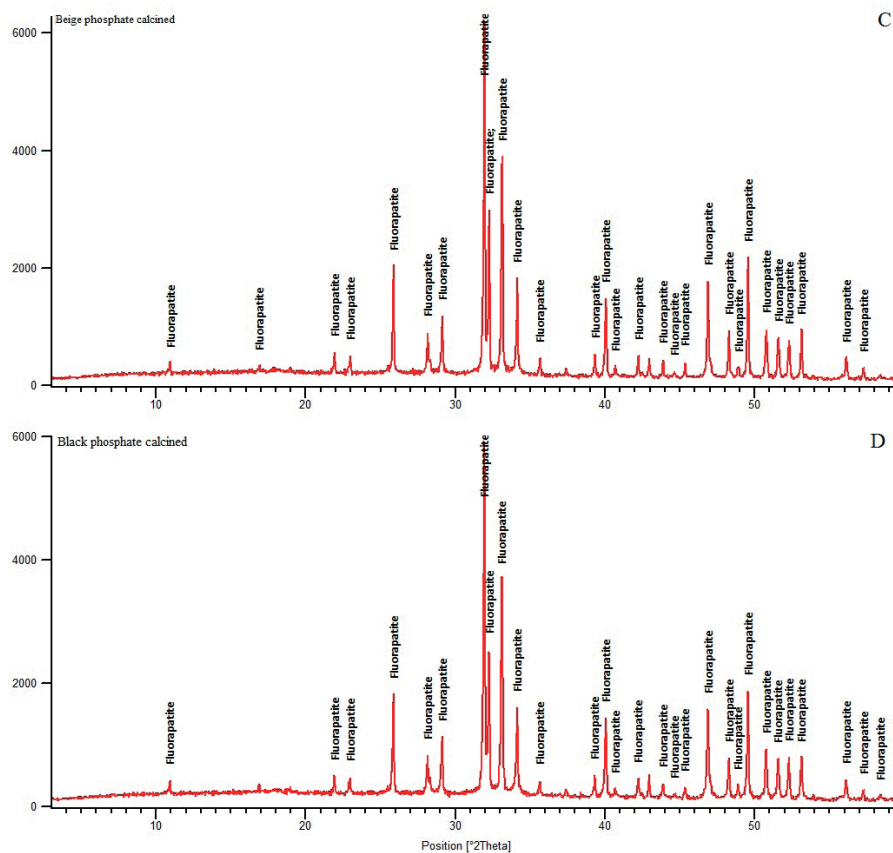

Fig. 7. XRD results of: C – beige phosphate calcined at 1 050 °C, D – black phosphate calcined at 1 050 °C.

Tab. 6

High voltage electrostatic separation test results for non-calcined and calcined beige phosphate

Round / min	Tension [kV]	Conductivity	Non-calcined beige phosphate				Calcined beige phosphate			
			P_2O_5 [%]	MgO [%]	CaO [%]	SiO_2 [%]	P_2O_5 [%]	MgO [%]	CaO [%]	SiO_2 [%]
30	25	conductor	**33.20**	4.42	67.16	6.80	**34.85**	5.05	75.08	6.11
		non-conductor	**28.84**	6.18	53.7	15.06	**26.77**	6.43	68.78	11.56
	30	conductor	**33.22**	4.17	66.28	6.56	**36.89**	4.42	75.85	5.20
		non-conductor	**27.43**	4.85	57.3	17.81	**27.13**	7.08	62.9	9.47
	35	conductor	**29.51**	2.45	60.22	4.94	**28.77**	6.43	68.78	5.56
		non-conductor	**20.84**	4.05	54.3	11.70	**22.33**	8.08	65.90	9.17
40	25	conductor	**32.58**	3.38	65.38	5.49	**32.37**	7.31	75.31	5.84
		non-conductor	**17.81**	6.13	62.36	14.79	**27.15**	8.08	62.9	7.37
	30	conductor	**30.65**	3.32	63.03	5.88	**34.64**	5.20	73.17	5.47
		non-conductor	**24.83**	3.98	58.08	11.05	**26.87**	8.43	63.78	12.56
	35	conductor	**30.24**	3.49	62.5	5.86	**35.56**	4.70	73.83	5.64
		non-conductor	**27.18**	8.17	60.01	11.63	**26.57**	6.46	67.18	11.16
50	25	conductor	**32.95**	4.21	66.43	6.33	**36.68**	6.28	78.15	5.64
		non-conductor	**28.57**	6.33	53.6	14.01	**27.45**	7.08	66.9	9.38
	30	conductor	**30.75**	3.33	60.45	5.34	**30.86**	6.86	71.38	5.34
		non-conductor	**26.94**	5.05	58.44	12.32	**25.94**	7.95	65.44	11.22
	35	conductor	**32.03**	3.29	63.94	5.86	**31.32**	2.20	72.17	5
		non-conductor	**29.60**	6.3	63.36	13.79	**24.33**	5.08	65.9	9.17

Tab. 7

High voltage electrostatic separation test results for non-calcined and calcined black phosphate

Round / min	Tension [kV]	Conductivity	Non-calcined beige phosphate				Calcined beige phosphate			
			P_2O_5 [%]	MgO [%]	CaO [%]	SiO_2 [%]	P_2O_5 [%]	MgO [%]	CaO [%]	SiO_2 [%]
30	25	conductor	**33.77**	3.18	66.89	4.17	**31.85**	7.89	73.47	4.74
		non-conductor	**27.63**	4.95	57.73	16.91	**24.5**	11.08	62.9	9.37
	30	conductor	**35.63**	3.03	69.91	4.23	**33.29**	8.45	76.27	4.79
		non-conductor	**27.5**	4.08	52.9	7.47	**26.53**	9.30	65.12	9.77
	35	conductor	**32.90**	3.03	63.71	3.95	**32.67**	7.07	74.14	6.09
		non-conductor	**17.64**	3.73	41.54	9.35	**26.54**	8.18	64.7	13.06
40	25	conductor	**34.09**	3.53	66.15	4.12	**32.81**	6.97	73.79	4.57
		non-conductor	**26.32**	4.08	53.9	8.41	**25.44**	8.18	68.7	12.16
	30	conductor	**36.06**	3.79	70.37	4.57	**33.52**	10.22	78.97	5.02
		non-conductor	**27.15**	5.08	56.9	9.47	**27.84**	12.18	63.7	14.06
	35	conductor	**35.42**	3.11	69.41	4.27	**29.12**	8.20	70.39	4.79
		non-conductor	**28.15**	4.58	54.9	9.47	**23.60**	9.13	63.36	12.79
50	25	conductor	**34.43**	2.85	67.23	4.38	**33.91**	6.31	71.20	4.7
		non-conductor	**26.5**	5.08	54.9	7.87	**27.93**	5.95	61.73	8.91
	30	conductor	**34.09**	3.46	67.27	4.55	**32.91**	8.62	76.73	5.06
		non-conductor	**25.35**	4.18	53.9	9.47	**17.63**	10.73	41.64	9.75
	35	conductor	**35.31**	3.08	68.16	4.06	**36.65**	9.94	82.16	5.62
		non-conductor	**27.50**	4.48	54.9	8.47	**27.25**	12.58	83.9	9.87

- For calcined beige phosphate and without calcination – rotation speed: 30 rpm, electrical voltage: 30 kV, P_2O_5 content: 36.89 % (calcination) and 33.22 % (without calcination) and recovery: 93.91 % (calcination) and 90.85 % (without calcination).
- For calcined black phosphate – rotation speed: 50 rpm, electrical voltage: 35 kV, P_2O_5 content: 36.63 % and recovery: 93.88 %.
- For black phosphate without calcination – rotation speed: 30 rpm, electrical voltage: 30 kV, P_2O_5 content: 35.65 % and recovery: 91.94 %.

5 Technological scheme of phosphate processing

The obtained treatment results made it possible to develop a treatment scheme for beige and black phosphates from the Kef Essennoun deposit of the Djebel Onk phosphate mining complex (Fig. 8).

X-ray diffraction (XRD) and scanning electron microscopy (SEM / EDS) results confirm that the main mineral phases in the sample are apatites, including fluorapatite, hydroxyapatite, and carbonate fluorapatite. On the other hand, the gangue minerals present include quartz and dolomite.

Calcination breaks down the carbonates into calcium oxide and magnesium oxide and eliminates most of the carbon dioxide (CO_2). The best results of chemical analysis obtained at the temperature 1 050 °C for beige phosphate are content of P_2O_5 31.15 % amount of MgO 3.78 %, and 40.80 % CaO, while for black phosphate was obtained 30.85 % of P_2O_5, 3.40 % MgO and 40.41 % CaO.

The beneficiation of phosphate ore by a combined method of calcination and electrostatic separation is an effective approach to concentrate ore from a sub-arid region. Then, electrostatic separation separates the particles based on their electrical properties, which facilitates the concentration of the phosphate.
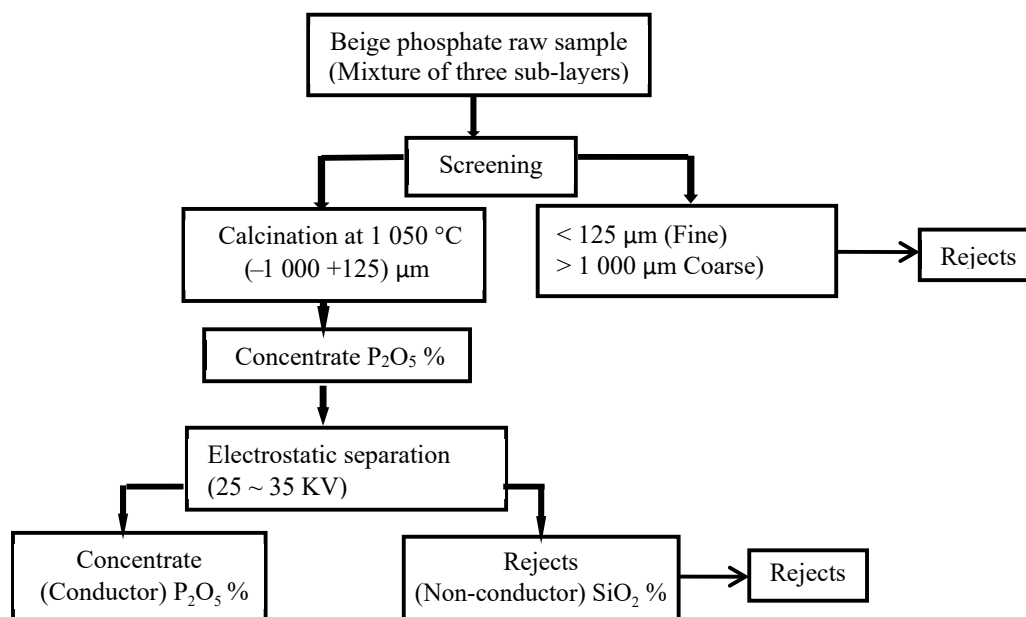

Fig. 8. Technological scheme for processing of Kef-Essennoun phosphate ores by electrostatic separation.

6 Conclusion

The Kef Essennoun deposit has sedimentary origins; it is composed of two types of phosphate: beige and black.

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

Indeed, particle size fractions between 1 000 μm and 125 μm are particularly rich in phosphate and display a significant yield of P_2O_5.

The calcination process allowed us to break down the carbonates into calcium oxide and magnesium oxide and remove most of the carbon dioxide (CO_2).

The best-obtained results from the calcination process followed by electrostatic separation with corona effect from an initial ore with a content of 31.15 % of P_2O_5 were achieved for calcined beige phosphate (rotation speed: 30 rpm, electrical voltage: 30 kV), with content of P_2O_5 in concentrate 36.89 % andrecovery of 93.91 %. For calcined black phosphate (rotation speed: 50 rpm, electrical voltage:

35 kV where content of P_2O_5 in concentrate reached 36.65 % with recovery of 93.88 % from an initial ore with a content of 30.85 % P_2O_5.

Tests of enrichment of calcined and non-calcined beige and black phosphate ore from the Kef Essennoun deposit by a combined method of calcination and electrostatic separation make it possible to obtain very satisfactory results. This study highlights the importance of the calcination treatment before the electrostatic separation operation.

Acknowledgments

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Spracovanie fosfátových rúd s nízkym obsahom úžitkovej zložky z bane Djebel Onka (Alžírsko) metódou elektrostatickej separácie

Výskumná práca sa zaoberá problematikou technologického spracovania nízkokvalitných fosfátových rúd z bane Djebel Onk (Alžírsko) metódou elektrostatickej separácie. Cieľom výskumu bolo overiť použitie zmesí pozostávajúcich z troch vzoriek suroviny odobratých zo svetlých (béžových) fosfátových vrstiev (bazálnej, hrubej približne 2 m, hlavnej strednej podvrstvy s hrúbkou asi 25 – 30 m a povrchovej vrstvy, ktorá obsahovala dolomit s nízkym obsahom fosfátov) a dvoch vzoriek suroviny odobratých z čiernych fosfátových podvrstiev povrchovej a hlavnej strednej vrstvy.

Odobraté vzorky boli podrvené na veľkosť zŕn menšiu ako 4 mm, voľne sušené a homogenizované. Pred elektrostatickou separáciou sa skúmané vzorky so zrnitosťou 125 – 1 000 µm kalcinovali pri teplote 850, 950 a 1 050 °C počas 15 minút s cieľom redukcie obsahu organických zložiek vo fosfátoch. Elektrostatická separácia sa realizovala použitím vysokonapäťového separátora typu Carpo HP16-114. Pred elektrostatickou separáciou sa vzorky sušili vo vákuu pri teplote 80 – 100 °C. Na každý test sa použilo 200 g vzorky, ktorá bola podrobená separácii pri napätí 25, 30 a 35 kV s rýchlosťou rotácie 30, 40 a 50 otáčok za minútu.

Chemické a mineralogické zloženie vstupnej suroviny, ako aj produktov technologickej úpravy bolo stanovené použitím rôznych analytických metód (röntgenová difrakčná analýza, röntgenová fluorescencia, SEM/EDS rastrovací elektrónový mikroskop a optický mikroskop).

Na základe uvedených analýz sa na elektrostatickú separáciu zvolili vstupné nekalcinované vzorky a vzorky termicky upravené pri teplote 1 050 °C.

Najvyššia výťažnosť pri použití elektrostatickej separácie v prípade béžového fosfátu sa získala pri kalcinovanej vzorke za týchto podmienok: rýchlosť rotácie 30 otáčok za minútu, elektrické napätie 30 kV. Zo vstupnej kalcinovanej rudy s obsahom 31,15 % P_2O_5 sa separáciou získalo 93,91 % P_2O_5. Z nekalcinovanej vstupnej vzorky s obsahom 33,22 % P_2O_5 sa za rovnakých podmienok získalo 90,85 % P_2O_5.

V prípade čierneho fosfátu po separácii kalcinovanej vstupnej vzorky s obsahom 36,63 % P_2O_5 pri napätí 35 kV a 50 otáčkach za minútu najvyšší získaný podiel P_2O_5 predstavoval 93,88 %. Z nekalcinovanej vstupnej vzorky čierneho fosfátu s obsahom 36,65 % P_2O_5 bola najvyššia výťažnosť separácie pri napätí 30 kV a 30 otáčkach za minútu, keď sa podarilo odseparovať 91,94 % P_2O_5.

Na efektívne získavanie P_2O_5 z fosfátových rúd v závislosti od ich charakteristík možno použiť viaceré technologické postupy. Elektrostatická separácia bola overená ako vhodná ekologická a efektívna metóda získavania P_2O_5 z kalcinovaných, ale aj nekalcinovaných béžových a čiernych fosfátov. Kalcinácia pri teplote 1 050 °C okrem rozkladu organických zložiek umožnila navyše rozklad karbonátov na oxidy vápnika a horčíka, ako aj odstránenie väčšiny obsahu CO_2. Odstraňovanie organických látok je rozhodujúce aj vo fosfátovom priemysle, kde tento spôsob úpravy vstupnej suroviny umožňuje vyrábať vysokokvalitnú kyselinu fosforečnú ekonomickejšie.