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CHARACTERIZATION AND DEVELOPMENT OF BENEFICIATION PROSPECTS OF SEDIMENTARY PHOSPHATE ORE FROM THE KEF ESSENOUN DEPOSIT (ALGERIA) BY REVERSE FLOTATION

Phosphate is one of the most important natural resources because it is so crucial to the production of fertilizers and phosphoric acid. However, many undesirable elements are present in raw phosphate ores, which need to be refined in some way to boost their market value is the objective of this work. The most efficient technique for upgrading phosphate ores by the selective removal of gangue minerals is reverse flotation. This study aims to determine the physicochemical properties and the flotation behavior of sedimentary phosphate ore from the Kef Essenoun deposit (Tebessa, Algeria), with the intention of developing an effective beneficiation process. To develop the processing parameters for the medium grade phosphate ore, a thorough investigation was conducted to ascertain its mineralogical composition, particle size distribution, and degree of impurity elimination. Particle size distribution measurements, XRF, XRD, SEM/EDS analysis, and petrographical evaluation were used to describe these phosphates. 69.64% of the total mass of the phosphate raw ore sample fell between the −0.5 and +0.1 mm range, which also showed a reasonably acceptable particle size distribution. In addition to the related impurities of MgO and SiO$_2$, chemical analysis revealed that the phosphates had P$_2$O$_5$ values ranging from 24% to 26%. According to mineralogical analysis, dolomite, calcite, and quartz were the related gangue minerals, whereas hydroxyapatite and fluorapatite were the predominant phosphate minerals. The traditional coprolitic and bioclastic characteristics of sedimentary phosphates were observed when examining the details on a smaller scale. Reverse flotation studies revealed that the produced concentrate reached 30% P$_2$O$_5$, confirm the effectiveness of this method for the perspective of valorization of sedimentary ore from the Kef Essnoun at Djebel Onk deposit and that this technique can increase the value of the ore. These findings support the idea that Algerian phosphate ore processing could benefit economically and environmentally from reverse flotation.

Keywords: particle size analysis, characterization, X-ray diffraction, beneficiation, phosphate, Djebel Onk, flotation.

1. Introduction

The expanding demands of the agriculture sector are the main driver of the ongoing increase in the global demand for phosphate fertilizer. With an average yearly growth rate of 2.4%, the International Fertilizer Association (IFA) predicts that this trend will continue. A number of variables contribute to this dynamic, such as the rising costs of agricultural commodities, especially cereals, which encourage farmers to increase their farmed acreage in order to increase their income. Increased need for fertilizers, such as phosphates, which are crucial for raising crop yields, is an inevitable consequence of the subsequent expansion of agricultural area [1–3].

With estimated deposits of more than 2 billion tons, Algeria has significant phosphate potential and is one of the most resource-rich countries in the world. But given the growing demand worldwide, its yearly production is still quite tiny, at only 1.5 million tons [4]. Algeria must overcome a number of obstacles in order to fully benefit from this natural richness, such as updating its industrial infrastructure, implementing cutting-edge technology, and creating sustainable plans.

Such a strategy would efficiently utilize these natural resources, increase production, and conform to market trends. Optimized phosphate exploitation could therefore boost the country's economy and improve its reputation abroad [5–8].

The mineralogical makeup of the ore has a direct impact on how well phosphate beneficiation techniques work. Simple mechanical procedures and complex strategies incorporating flotation, magnetic separation, electrostatic separation, dense media separation, and other techniques are examples of industrial techniques. For medium-grade phosphate ores, flotation is still the most used technique because it effectively separates phosphate particles from silicate gangue [9–11]. Direct flotation, which aims to extract rich minerals, and reverse flotation, which eliminates impurities, are the two primary techniques used. Nonetheless, there are still issues with technology, including as adjusting to complicated ores, cutting energy expenses, and improving chemical reagents. Advances in fine particle treatment and selective separation may increase yields and strengthen the sustainability of this vital sector [12].

The Djebel Onk deposit in northeastern Algeria, about 20 kilometers from the Algerian-Tunisian border, is the subject of the study

region (Fig. 1). The closest locality is Bir el Ater, which is almost 100 kilometers away and administratively under the Tébessa province (wilaya). Oued Btita, Bled el Hadba, Djebel Onk North, Djemi Djema, and Kef Essenoun are among the phosphatic zones that make up the deposit. Because of its geological and mining relevance, the Kef Essenoun deposit is the focal point of our investigation. There is a lot of interest in its development because of its strategic location and large phosphate reserves, which make it a crucial economic and scientific actor on a regional and national level [13].

Given the rising need for phosphate fertilizers, Algeria, which possesses substantial mineral deposits, needs to improve its industrial and technological capabilities in order to fully realize this potential. Therefore, the sedimentary phosphate ore from the Djebel Onk Kef Essenoun deposit, which is situated in the Tébessa region of Algeria, is the subject of this study.
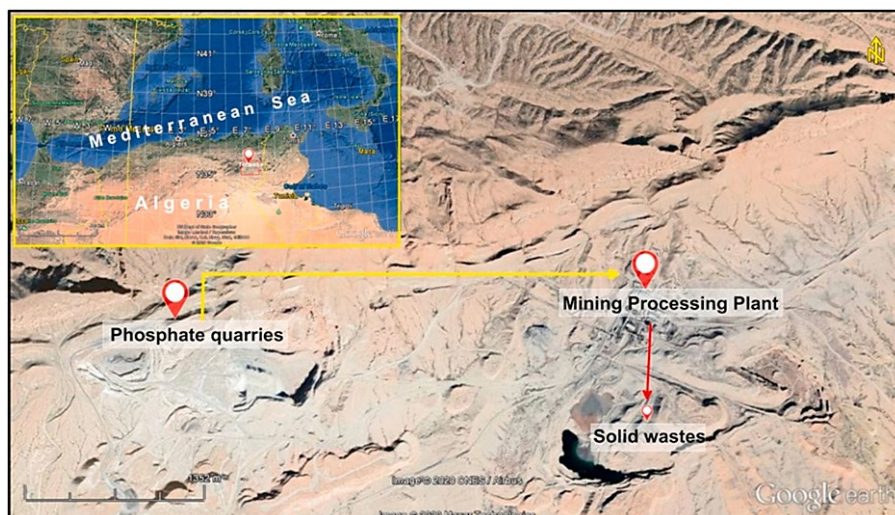

Fig. 1. Location of the phosphate deposit and its processing plant (satellite view-Google Earth) [14]

With creative enrichment methods and a better knowledge of the phosphate ore properties of the Djebel Onk deposit near Tébessa, Algeria may be able to establish itself as a major participant in the world phosphate market. Such advancements would support agricultural production and contribute to global food security in addition to boosting the local economy [15–17].

Thus, *the object of this study* is the Djebel Onk (Kef Essenoun) phosphate ore. *The aim* is to describe the content and characteristics of the ore. According to the findings, effective and economical enrichment techniques that satisfy industrial demands should be developed ($P_2O_5 \geq 30\%$, $MgO \leq 1\%$, $SiO_2 \leq 10\%$, $CaO \leq 50\%$). Enhancing ore processing could lead to increased global food security, local economic growth, and Algeria's position in the phosphate industry. In this context, samples were collected from the Djebel Onk phosphate mine and prepared for mineralogical and chemical characterization, as well as reverse flotation tests 18, 19.

2. Materials and Methods

2.1. Sample preparation

The first and one of the most crucial steps in characterization is sampling. Phosphate samples were taken every 30 minutes at the outlet of the grinder in the Djebel Onk processing plant to obtain a representative sample of the run-of-mine (ROM). A total of 25 kg of ore, consisting of a mixture of beige and black phosphate, was collected and sent to the quality control laboratory of Somiphos at Djebel Onk.

In the laboratory, the sample was homogenized and then divided using riffle splitters. One portion was used for particle size analysis, while the other was further subdivided to obtain a global representative sample of the ROM for chemical analyses. The remainder was kept as a reference sample. Finally, the samples were dried on a hot plate or in a drying oven.

2.2. Analysis

A particle size analysis was carried out using an electric sieve shaker equipped with sieves of 1, 0.5, 0.315, 0.1, and 0.08 mm. The raw ore was sieved for 15 minutes, and then each fraction was weighed and ground for chemical analyses. X-ray fluorescence (XRF) spectrometry was performed at the IUT of Angoulême (France) on the global sample and the particle size fractions to identify the major components and their distribution. A Bruker S1 TITAN portable XRF spectrometer, equipped with a rhodium-anode X-ray tube and an SDD detector, was used to enable rapid, non-destructive elemental analysis without sample preparation.

The petrographic study was conducted on thin sections prepared at the geology laboratory of Badji Mokhtar University and subsequently examined using a polarizing microscope. Scanning electron microscopy (SEM) analyses were performed at the laboratories of the National School of Technology and Engineering in Annaba (ENSTI) using a FEI Quanta 250 microscope equipped with an energy-dispersive X-ray spectroscopy (EDS) system. The instrument operated in low vacuum mode, allowing both high-resolution imaging and elemental analysis of uncoated phosphate ore samples.

The identification of mineralogical phases in the finely ground phosphate ore was performed using an X-ray diffractometer (PANalytical Empyrean), equipped with a Cu-Kα radiation source ($\lambda = 1.5406$ Å). This device allows precise identification of crystalline phases through $\theta - 2\theta$ scans, making it suitable for detailed characterization of mineralogical compositions.

2.3. Beneficiation process

Reverse flotation tests were carried out on the Kef Es Sennoun ore (–500 + 63 µm fraction) at the LAVAMINE laboratory of Badji Mokhtar University using a DENVER flotation cell. Before flotation, 10-minute attrition was performed, followed by wet desliming at 63 µm and drying at 100°C to remove impurities (clays, silicas) and reduce the MgO content. This preparatory phase ensured optimal flotation conditions, significantly improving mineral separation efficiency. The systematic methodology not only enhanced the recovery of valuable minerals but also contributed to overall process optimization, demonstrating the critical role of pre-treatment in beneficiation performance.

Each sample was then prepared as a pulp with 30% solids, stirred for 10 minutes at 1100 rpm, and conditioned with reagents for 10 minutes. The reagents used were:
  – oleic acid and potassium oleate as collectors;
  – sulfuric acid, sodium silicate, and sodium-potassium tartrate as depressants.

The Methyl Isobutyl Carbinol MICB as a frothing agent. The pH regulators used were hydrochloric acid (HCl) and sodium hydroxide (NaOH). The reagents were added at different dosages and times (Table 1).

During flotation, air is injected to form a froth that carries the gangue minerals, which are recovered by skimming. The valuable minerals remain in the pulp. The floating material corresponds to the tailings, while the sinking material constitutes the concentrates. This method enables efficient separation of the target minerals.

Table 1

Reverse flotation test parameters

Test	Particle Size (μm)	pH	pH regulator	Reagents	Dosage (g/t)	Volume (ml)	Time (min)
1	−500 + 63	5	HCl	Sulfuric acid	1000	6	5
				Potassium oleate	1000	6	3
				MICB	17	1	2
2	−500 + 63	9	NaOH	Sodium-potassium tartrate	1000	6	5
				Oleic acid	1500	6	3
				MICB	17	1	2
3	−500 + 63	6–7	–	Potassium oleate	1000	6	5
				Sulfuric acid	500	3	3
				MICB	50	0.5	2

3. Results and Discussions

3.1. Chemical composition

The results of X-ray fluorescence (XRF) chemical analysis of a raw phosphate sample and its different particle size fractions resulting from dry sieving are summarized in Table 2. Significant differences in chemical composition based on particle size are shown by the results. An important metric for determining the quality of phosphate, the P_2O_5 content, peaks at 29.21% in the (−0.5 + 0.315 mm) fraction and decreases in the fraction (> 1 mm) and ultrafine (< 0.08 mm). MgO is mainly detected in the coarser (+1 mm) and finest (−0.08 mm) fractions, with minimal values in the intermediate fractions. The finer particles, on the other hand, include more silica (SiO_2), most likely as a result of contaminants connected to clay. In the (−0.1 + 0.08 mm) range, calcium oxide (CaO) exhibits the highest concentration (35.46%), demonstrating relative stability throughout fractions and confirming its close relationship with phosphate minerals.

larly the −0.5 + 0.315 mm and −0.315 + 0.1 mm fractions, perform best combining a high mass yield with elevated P_2O_5 levels and reduced amounts of MgO and SiO_2.

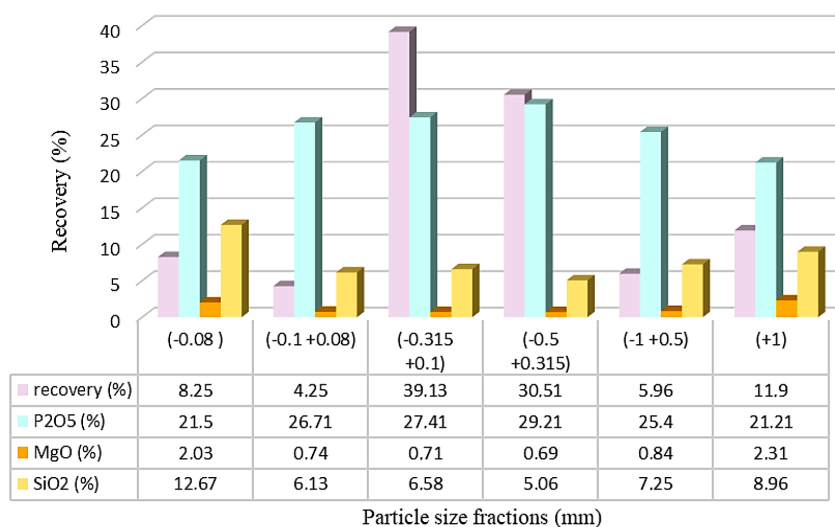| Particle size fractions (mm)	(-0.08)	(-0.1 +0.08)	(-0.315 +0.1)	(-0.5 +0.315)	(-1 +0.5)	(+1)
recovery (%)	8.25	4.25	39.13	30.51	5.96	11.9
P2O5 (%)	21.5	26.71	27.41	29.21	25.4	21.21
MgO (%)	2.03	0.74	0.71	0.69	0.84	2.31
SiO2 (%)	12.67	6.13	6.58	5.06	7.25	8.96

Fig. 2. Particle-size distribution of key elements in the raw phosphate sample

Table 2

Chemical analysis by XRF (run-of-mine) of different particle size fractions

Particle size fraction (mm)	P_2O_5 (%)	MgO (%)	SiO_2 (%)	CaO (%)	Al_2O_3 (%)	Fe_2O_3 (%)	TiO_2 (%)
+1	21.21	2.31	8.96	34.25	1.14	0.64	0.04
−1 + 0.5	25.40	0.84	7.25	34.55	1.07	0.50	0.04
−0.5 + 0.315	29.21	0.69	5.06	34.73	0.68	0.42	0.02
−0.315 + 0.1	27.41	0.71	6.58	34.86	0.84	0.47	0.03
−0.1 + 0.08	26.71	0.74	6.13	35.46	0.81	0.44	0.02
−0.08	21.50	2.03	12.67	31.84	1.70	0.91	0.04
Global sample	24.59	1.27	6.41	35.15	0.86	0.49	0.03

The impurities (AlO_3, FeO_3, and TiO_2) are present in trace levels and show slight variations that are associated with the range of particle sizes. A stable phosphate matrix is indicated by the bulk sample's homogenous composition, which averages 24.59% P_2O_5, 1.27% MgO, 6.41% SiO_2, and 35.15% CaO. Phosphate recovery might be improved and waste could be reduced with such a method.

As shown in Fig. 2, there is a noticeable relationship between grain size and chemical composition. The intermediate size ranges, particu-

This suggests that, in these fractions, phosphate minerals are more effectively liberated from the surrounding gangue. In practical terms, these sizes are ideal for beneficiation processes. On the other hand, both the finest particles (−0.08 mm) and the coarsest ones (+1 mm) show higher impurity levels and lower phosphate grades. That likely means the phosphate is either not fully liberated or still locked within gangue minerals. The finer material might include clay-rich particles where phosphate is mixed with impurities, while the coarser grains could be complex aggregates of multiple minerals both of which are more challenging to process efficiently.

3.2. Petrographic characterization

A complex microstructure with diverse particle morphologies and size distributions is displayed by Kef Essennoun phosphates, according to petrographic study of thin sections. Three different phosphate phases were identified:
1) pellets;
2) coprolites;
3) bioclasts.

These phases were closely linked to exogangue minerals such as calcite and dolomite (Fig. 3).

A complicated, polyphase depositional history marked by repeated periods of sedimentary reworking and diagenetic overprinting is reflected in the observed textural and compositional heterogeneity.

These microstructural characteristics act as important genetic markers for reconstructing the paleoenvironmental circumstances that prevailed during phosphate formation and offer vital limits for determining the deposit's paragenetic history. All things considered, the results greatly advance our knowledge of phosphorite genesis in similar geological settings and depositional conditions.
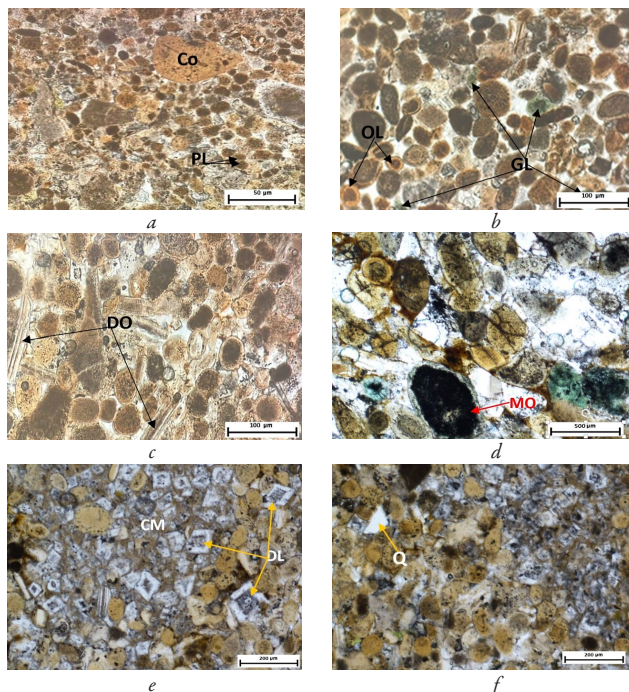
Fig. 3. Thin section microphotograph. Key: (CO) Coprolite; (Pl) Pellet; (GL) Glauconite; (DO) Bone fragment; (DL) Dolomite crystals; (CM) Microsparitic dolomitic cement; (Q) Quartz. Photomicrographs of thin sections of phosphate rocks observed under an optical microscope: *a* – shows the presence of coprolites (Co) and pellets (Pl) within a fine-grained matrix; *b* – illustrates ooliths (OL) associated with glauconite grains (Gl); *c* – shows numerous ooliths accompanied by bone debris (DO); *d* – presence of organic matter (MO); *e* – shows microsparitic dolomitic cement (CM) surrounding dolomite crystals (Dl); *f* – presence of quartz (Q)

3.3. Mineralogical analysis

The identification of the mineralogical phases in the ground phosphate ore confirmed the presence of target minerals (Fig. 4).

The analytical examination of the Kef Es Sennoun phosphate ore revealed the following phases:

1. The main phosphate-bearing components include: hydroxyapatite $Ca_5(PO_4)_3(OH)$; carbonate hydroxyapatite $Ca_5(PO_4,CO_3)_3(OH)$; apatite $Ca_5(PO_4)_3$; fluorapatite $Ca_5(PO_4)_3F$; and carbonate fluorapatite $Ca_{10}(PO_4)_5CO_3F_{1.5}(OH)_{0.5}$.

2. The gangue components are mainly represented by carbonate minerals such as calcite $(CaCO_3)$ and dolomite $(CaMg(CO_3)_2)$, as well as siliceous minerals like quartz (SiO_2) and clays. This mineralogical composition is consistently found across all particle size fractions, though in varying proportions.
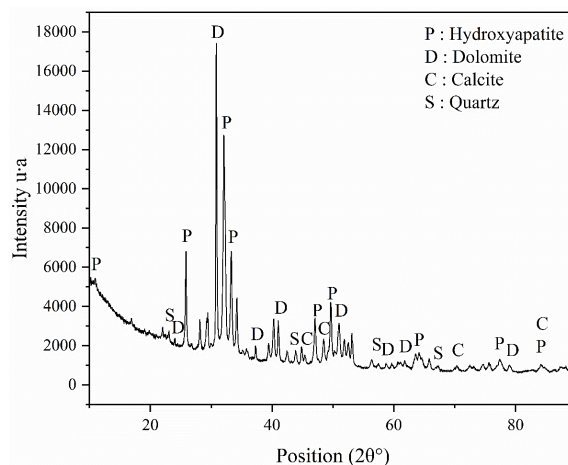

Fig. 4. X-ray Diffractogram of run-of-mine (ROM) phosphate ore

3.4. Scanning electron microscopy (SEM) analysis

Energy-dispersive X-ray spectroscopy (EDS) in conjunction with scanning electron microscopy (SEM) demonstrated intricate microstructural characteristics in the phosphate samples. Polymorphic phosphate phases with diverse particle morphologies ranging from 5 to 200 μm were found by the analysis (Fig. 5). Quantitative EDS mapping revealed that 60–70% of the particles were composite grains with close phosphate-dolomite intergrowths. These textural connections point to two possible processing issues because of micron-scale mineral locking and diagenetic recrystallization under changing geochemical circumstances. The paragenetic origin of the deposit and the necessary beneficiation techniques – specifically, liberation size optimization and reagent selection for efficient phosphate-dolomite separation – are both strongly supported by the observed microstructural complexity.

The following is displayed in Fig. 6, which shows the mapping of the regional variation in the chemical element concentrations of the raw phosphate ore sample from Kef Essenoun using SEM.

The presence of apatite $(Ca_5(PO_4)_3(F,Cl,OH))$, a feature of phosphate ores, is confirmed by the preponderance of Ca, P, and O. Silicate impurities like quartz or feldspar are probably the cause of the comparatively high Si content (9%). On the other hand, the sample's overall compositional complexity may be enhanced by the existence of secondary mineral phases, such as clays, metal oxides, or sulfates, as indicated by the low quantities of Al, Fe, and S.
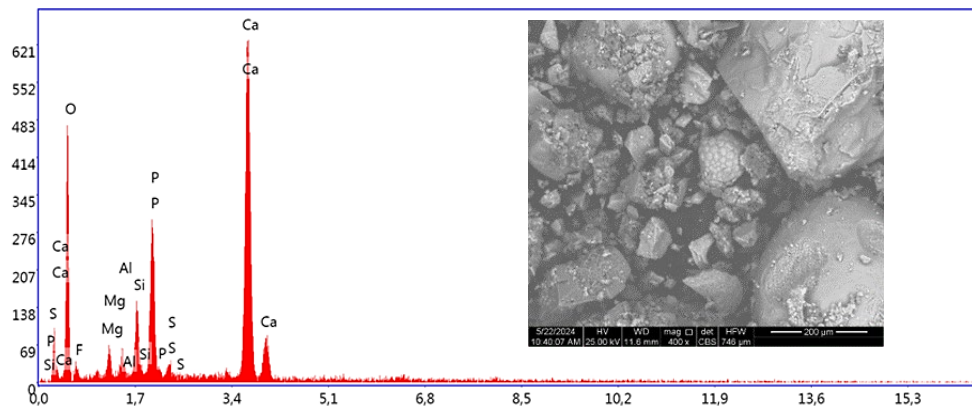
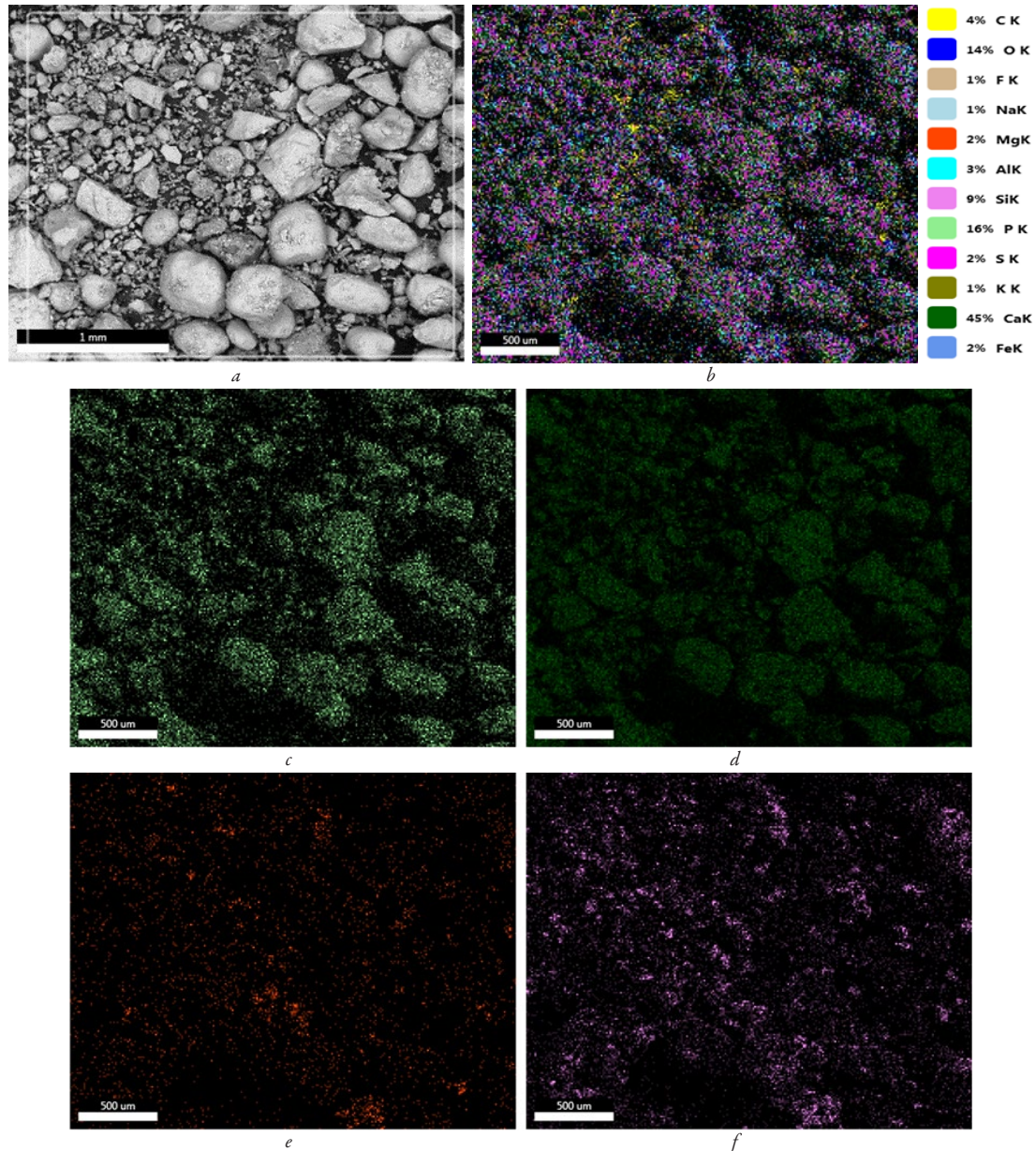**Fig. 5.** SEM/EDS of raw phosphate ore

Fig. 6. Scanning electron microscope (SEM) elemental mapping of raw phosphate ore: *a* – SEM image showing the morphology and texture of the raw phosphate ore particles; *b* – Combined elemental mapping (EDS) an overlay of all detected elements, with colors corresponding to the legend; *c, d, e, f* – are respectively elemental maps of Phosphorus (P), Calcium (Ca), Magnesium (Mg) and Silicon (Si)

All target species, including dominant apatite with related dolomite, calcite, quartz, and clay minerals, were confirmed to be present by mineralogical investigation. The phosphate particles had a variety of shapes, including spherical, cylindrical, and oval shapes. They were frequently intergrown with calcite and quartz as accessory minerals. These microstructural studies show comparable mineralogical properties across analytical techniques, validating both previous petrographic results and bulk chemical experiments. The findings offer a thorough comprehension of the composition and textural correlations of the ore, which are essential for designing the beneficiation process.

3.5. Reverse flotation

Table 3 displays the reverse flotation test results, which show the performance attained. The main information is compiled in this table, which makes it possible to compare the many parameters that were examined throughout the experiment.

Table 3

Results of chemical analyses of reverse flotation products

Tests	Products	Weight recovery (%)	Grade (%)			Metal recovery (%)			
			P_2O_5	MgO	SiO_2	CaO	P_2O_5	MgO	SiO_2
1	Feed	–	26.5	4.5	10.3	35.15	–	–	–
	Floating	4	18.13	4.95	16.01	30.85	19.6	46.53	86.53
	Sinking	96	29.87	4.17	3.13	39.26	71.4	53.4	13.47
2	Feed	–	26.5	4.5	10.3	35.15	–	–	–
	Floating	15	21.71	5.51	12.1	31.28	27.7	34.74	91.63
	Sinking	85	28.95	4.1	3.91	29.66	72.3	65.23	8.35
3	Feed	–	26.5	4.5	10.3	35.15	–	–	–
	Floating	11.73	20.28	5.77	11.98	29.84	27.8	56.5	93.54
	Sinking	88.26	30.05	3.5	3.4	30.97	72.2	43.5	6.46

The results of the tests show that the concentrate (sinking fraction) has a high P_2O_5 content, ranging from 28.95% to 30.05%, P_2O_5 recovery rate of up to 72%, and weight yields exceeding 85%. Even though the levels of impurities, particularly MgO (< 4.17%) and SiO_2 (< 3.91%), are comparatively lower, they are still more than what is needed to meet industrial standards. Additionally, the reject (floating fraction) retains a notable amount of P_2O_5, particularly under alkaline conditions(Test 2), suggesting incomplete separation of fine phosphate particles. pH has a major impact on separation efficiency: neutral to alkaline circumstances increase the total mass yield of the floating fraction, while acidic conditions (Test 1) increase the concentration of P_2O_5 in the sinking fraction. These results demonstrate how important pH regulation is for maximizing concentrate grade and fines recovery.

Nevertheless, our product can be applied in various sectors: Industrial, agricultural, pharmaceutical and even in the materials construction manufacturing, such as cement and composite. It may be used in the manufacture of medium-grade fertilizers, as a soil amendment for nutrient-deficient agricultural land, or as a raw material in certain phosphoric acid production processes where high purity is not necessary. It also holds potential for use in the production of phosphates for livestock feed and in chemical applications where moderate phosphate content is acceptable. Furthermore, it can be incorporated as an additive in the fabrication of composites to produce high-quality materials that align with sustainable development principles. However, the implementation of these applications remains subject to prevailing regulations and the specific technical requirements of each process, highlighting the importance of tailoring the material's treatment to its intended use.

More research should concentrate on improving pH conditions in conjunction with the use of more selective flotation reagents in order to reach industrially acceptable standards. This could allow for a more effective and balanced beneficiation process.

4. Conclusions

The physico-chemical analyses (XRD, XRF, SEM-EDS, and thin section microscopy) conducted on the raw phosphate ore from Kef Es Sennoun enabled the characterization of its granulochemical, mineralogical, and petrographic properties. The ore displays a fine particle size distribution, with the majority of the mass (69.64%) concentrated in the (–0.5 + 0.1 mm) fraction, which is rich in P_2O_5 and low in impurities, representing the optimal liberation size. The phosphate content ranges between 24% and 26% P_2O_5, along with impurities such as MgO, SiO_2, and CaO. Principal component analysis identified two main chemical associations: apatite and gangue phases. Microscopically, the ore is composed of pellets, coprolites, bioclasts, and gangue minerals such as dolomite, calcite, and quartz. XRD results revealed the presence of phosphate phases like hydroxyapatite and fluorapatite, along with a silico-carbonate gangue. SEM observations confirmed these findings. Our research on Kef Essennoun phosphate ore confirms that using reverse flotation gives a valuable opportunity to produce a rich concentrate with over 30% P_2O_5. However, this product remains doesn't has commercial standards because it contains too many impurities such as MgO and SiO_2 which exceed industry limits. Because of this, further purification is needed to make the concentrate commercially viable and meet market requirements.

When Kef Essennoun's phosphate ore was subjected to reverse flotation, a concentrate enhanced reached 30% P_2O_5 was generated. Nevertheless, the product still fails to meet commercial requirements because of high impurity levels, especially MgO and SiO_2, which beyond permissible industrial limits. To improve the concentrate's quality and make it fit for commercial use, more purification procedures are therefore required. Meeting the strict requirements needed for commercial and industrial applications would require additional refining.

Several strategies might be investigated to enhance flotation performance and the resulting concentrate's quality. To minimize P_2O_5 losses in the floating material, one way to maximize reagent selection is to evaluate collectors and depressants that provide higher selectivity. To increase recovery and reduce phosphate-rich particles in the tailings, desliming efficiency must be increased. Furthermore, a thorough analysis of pH levels might help in determining the ideal balance between yield and grade. By using these strategies, operating conditions might be improved and technical and financial goals could be better met.

Conflict of interest

The authors declare no financial, personal, professional, or authorship-related conflicts of interest that could have affected the research design, execution, analysis, or reporting. They affirm the study's integrity and objectivity, ensuring that all findings and conclusions are free from bias or external influence.

Data availability

There is no data associated with the manuscript.

Use of artificial intelligence

The authors confirm that artificial intelligence technologies were not utilized in the creation of the current work.

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