

UPGRADING OF LOW-QUALITY EGYPTIAN PHOSPHATE ORE USNIG FLOTATION TECHNOLOGY

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ABSTRACT

Processes to upgrade the quality of low-quality phosphate ore are considered essential for sustainable development. Previous studies on exploitation and beneficiation of low-quality ores pointed out that anion flotation processes were considered one of the most appropriate methods for upgrading the quality of phosphate ores to a grade that render it suitable to produce phosphoric acid. In this study, a low-quality sample of phosphate ore was used which contained 21% phosphorus pentoxide and impurities of calcite and silicates. The sample was ground to a size of (-250 + 45 microns) using a rod mill. A mixture of fatty acids and kerosene was used as a collector during anionic flotation. The main factors that affect the flotation process, such as the collector dose, pH, and temperature of the medium, were studied and optimized to obtain the ideal separation conditions. An economically and environmentally suitable beneficiated ore was obtained containing 32.23% of phosphorus pentoxide, 4.67% (insoluble materials) representing silica and 10.10% loss of ignition (representing carbonates) with a recovery rate of 80.01% from an original containing 21.50% phosphorus pentoxide, 13.03% insoluble materials, and 13.13% loss of ignition.

Key words: Phosphate Ores, Flotation, Fatty acid.

INTRODUCTION

The surge in global population and the prevalence of famines in diverse regions, attributable to food shortages, have underscored the imperative of exploring phosphate ores as a pivotal source to produce phosphoric acid and fertilizers. The depletion of high-grade ores, driven by intensified agricultural activities and burgeoning crop yields, further accentuates the significance of this endeavor (Ahmed, 2007). High-grade phosphate ores, with a phosphorus pentoxide (P₂O₅) content of approximately 30% or higher, were extracted from the earth's crust and must undergo processing to become suitable for industrial applications. The mineralogical composition of these ores varies according to their type and origin. Notably, sedimentary ores, constituting around 80% of the global phosphate rock

production, show a multitude of impurities. Processing techniques for phosphate ores are contingent upon their type and the nature of associated gangue minerals. Marine sedimentary deposits, the most abundant source of phosphates globally, present variable compositions with carbonates and silicates. Approximately 95% of the world's produced phosphate is consumed within the fertilizer industry. Phosphate impurities predominantly manifest in very fine particle sizes (Sis and Chander, 2003; Abouzeid, 2008; Andronov *et al.*, 2016; Yehia *et al.*, 2019). Separation methods, such as washing and flotation, are employed to isolate valuable phosphate minerals from gangue in sedimentary rocks, while igneous deposits need flotation for separation (Ibrahim *et al.*, 2020). The subsequent dissolution of rich phosphate ores in sulphuric acid via wet and pyrogenic processes yielded intermediate products, essential for the production of diverse fertilizer types. Criteria for phosphate suitability in the wet phosphorus process encompasses P_2O_5 content exceeding 30%, CaO/P_2O_5 ratios below 1.6, and MgO concentrations below 1% (Sis and Chander, 2003). Various enrichment techniques, contingent upon ore type, mineralogy, gangue characteristics, and cost considerations, can be applied to upgrade phosphate ores (Guo and Li, 2010; Farzanegan and Mirzaei, 2015; Elbendary *et al.*, 2019). There are various alternative methodologies, encompassing attrition scrubbing to ameliorate clay impurities a concentrate assaying 28.26% P_2O_5 with a recovery of 80.37% (Ruan *et al.*, 2019). Electrostatic separation for pre-concentration by discarding a certain proportion of silica reduced the energy consumption of grinding, cost of chemical reagents and water usage a concentrate containing 30% P_2O_5 with a recovery of more than 85% (Sobhy and Tao, 2014). Magnetic separation pertinent to igneous apatite deposits a concentrate assaying 31.2% P_2O_5 with a recovery of 70% was obtained through the removal of dolomite gangue minerals (Blazy *et al.*, 1997). Gravity separation predicated on density disparities It is carried out according to the density difference between valuable minerals and gangue minerals a concentrate content 30.64% P_2O_5 and recovery 85.60% (Wei *et al.*, 2011). Calcination to enhance P_2O_5 content, collectively contribute to the expansive toolkit of phosphate beneficiation techniques (Watti *et al.*, 2016). Froth flotation stands out as a highly effective

and widely utilized beneficiation technique (Kawatra and Carlson, 2013; Karapetian and Dzhevaga, 2017; Ruan *et al.*, 2018; Aleksandrova, 2022). All the previous methods aim to upgrade the low-quality of phosphate ore to maximize its benefit and the ability to use it in industry.

MATERIALS & METHODS

A representative phosphate sample, weighing approximately 200 kilograms, was procured from the run-of-mine deposits in the Sabyia West region. Post-classification, the sample was categorized as low grade. Zeta potential measurements were conducted on both pure apatite and silica components. The reagent employed as a collector in these experiments was oleic acid (cis-9-octadecenoic acid) with a purity level of 99.4%, obtained from Ward's Natural Science Establishment in New York, USA, and supplied by Alfa Products. All other chemical reagents, including sodium hydroxide and hydrochloric acid of analytical grade (Aldrich), were utilized as pH regulators. These chemicals were sourced from El-Nasr Company for Chemical Industries in Egypt and were used without any additional purification steps. The preparation of all solutions was conducted freshly at room temperature immediately before their utilization in measurements. Bi-distilled water, meeting the requisite standards, was employed in the formulation of all solutions to ensure the accuracy and reliability of the experimental procedures. (Vogel, 1961)

1. X-Ray Diffraction (XRD analysis)

A -200 mesh finely ground sample, mixed with 2 grams of wax as a binder, and was compacted into an aluminum cup to form a disk. This disk was then subjected to Qualitative X-ray diffraction (XRD) analysis using a PAN analytical X'Pert PRO with a Secondary Monochromator. Cu-radiation ($\lambda=1.542 \text{ \AA}$) was employed at 45 kV and 35 mA, with a scanning speed of 0.04 degrees per minute. (Zwell and Danko, 1975)

2. Chemical Analysis

Quantitative chemical analysis of oxide content in phosphate samples was conducted through X-ray fluorescence analysis (XRF) using a Phillips PW 2404 instrument from the Netherlands. In this procedure, 10 grams of the dried sample was blended with 2 grams of

wax as a binder in an aluminum cup, forming a disk. This disk was then exposed to X-rays for analysis. Phosphorus content was specifically analyzed and quantified as P_2O_5 using a UV/Vis Spectrometer, Model Lambda 2500, at a wavelength of 420 nm. For the determination of silicates and any undissolved clays, gravimetric analysis was employed, while the content of carbonates was assessed through loss of ignition (LOI) by firing 10 grams of the phosphate sample at 1000°C for 2 hours. The subsequent gravimetric analysis facilitated the calculation of the loss of ignition.

3. Flotation Experiment

Direct phosphate flotation was executed employing the "Denver D12" flotation machine, with a collector consisting of a 1:1 mixture of kerosene and oleic acid. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were utilized as pH regulators to ensure optimal conditions for the flotation process. The operational parameters for flotation included a rotational speed of 1800 rpm, a solid/liquid ratio of 60%, and a flow rate of 30 cm/min.

4. Zeta Potential

Zeta potential measurements were conducted using a laser Zeta Meter, specifically the 'Malvern Instruments Model Zeta Sizer 2000.' The experimental setup involved measurements taken both before and after the introduction of the collector, with the ionic strength maintained at 2×10^{-2} M KNO_2 . Sodium hydroxide (NaOH) served as the pH modifier throughout the experimental procedure.

5. Surface Tension Measurement

Surface tension measurements for the solutions were conducted at room temperature (20-25°C) utilizing the Krüss Tensiometer K9, employing the Wilhelmy plate method. The experimental procedure involved the preparation of various concentrations of sodium oleate, followed by thorough agitation to ensure homogeneity. Subsequently, surface tension was measured at distinct pH levels, considering the oleate-induced reduction in surface tension at the water-mineral interface. (Shaw, 1992)

6. Adsorption Measurement

The procedure involved adding 0.5 g of the pure sample to a 50 ml oleate solution of the desired concentration. The mixture was conditioned for 30 minutes to facilitate interaction between the sample and oleate. Subsequently, centrifugation was performed at 15,000 rpm for 15 minutes to separate the solid components from the solution. The determination of total organic carbon (TOC) content was carried out using a Total Carbon Analyzer, specifically the Phoenix8000 model.

RESULTS AND DISCUSSION

A comprehensive chemical analysis of the original sample was conducted utilizing X-ray fluorescence (XRF) techniques, and the obtained results were detailed in Table (1). The analysis revealed a phosphorus pentoxide (P_2O_5) content of 21.03%, alongside other impurities such as insoluble residue (I.R.) measuring 13.03%, carbonates (L.O.I.) at 13.31%, and 44.49% calcium oxide (Cao). These findings collectively indicate that the phosphate sample falls within the category of low-grade material (Leikam & Achorn, 2005) aligning with established industry standards for the manufacturing of phosphoric acid and various industrial applications.

The analytical results from XRF were corroborated by X-ray diffraction (XRD) analysis, as depicted in Fig. 1. The XRD analysis unveiled the presence of phosphate in the form of hydroxyapatite, while impurities were identified in the forms of calcite and silicate. This multimodal analytical approach provides a comprehensive understanding of the sample's chemical composition and mineralogical characteristics, affirming its low-grade nature and suitability for specific industrial applications (Ruan, He, & Chi, 2019)

Table 1. Complete chemical analysis of the origin sample

$P_2O_5\%$	$SiO_2\%$	MG O	Al_2O_3 %	CAO %	$SO_3\%$	Na_2 O	MNO	Fe_2O_3 %	CL	SR O	L.O.I.%
21.03	13.003	0.25	1.18	44.49	2.47	0.64	0.14	2.12	0.13	0.16	13.31

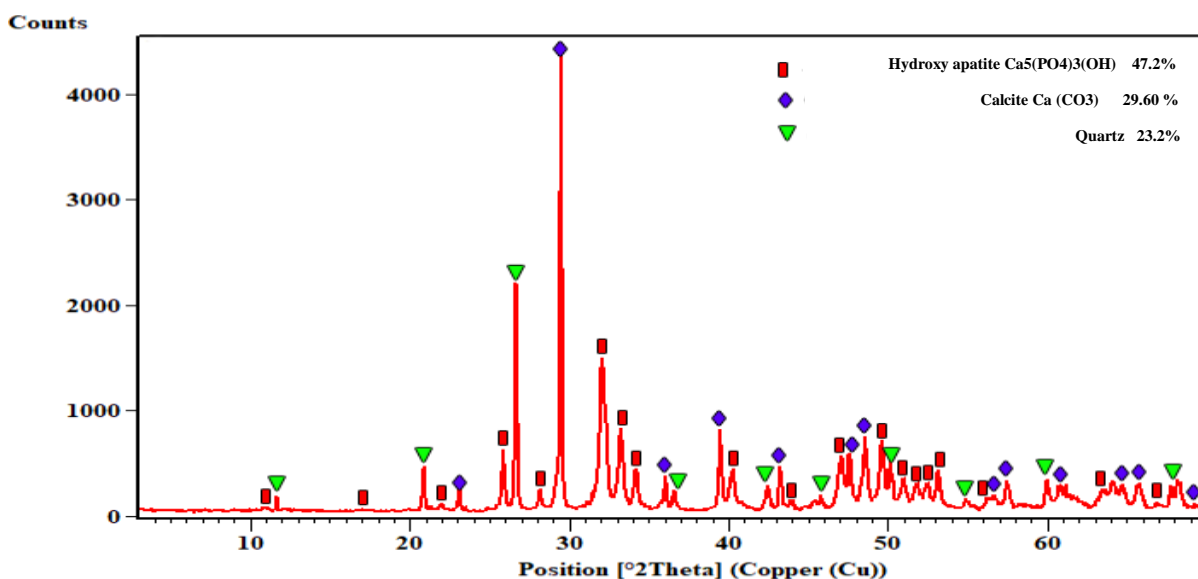


Fig. (1) XRD chart showing the main mineral phases in the origin phosphate sample and their semi qualitative percentage.

From (EDAX) measurements, both surface morphology and elemental structure of the phosphate sample were studied using Employing Energy Dispersive X-ray Spectroscopy. Figure (2) revealed an Fe-rich apatite grain alongside its corresponding apatite grain containing Cl, S, and F. The predominant phases consisted of Ca, P, and O, consistent with the expected composition of apatite (Ca₅(PO₄)₃). The presence of Ca, P, and Fe in the Energy Dispersive X-ray Spectroscopy (EDS) results signified the prevalence of Fe-rich hydroxyapatite. Additionally, the observation of Cl and F suggests the potential presence of fluorapatite and chlorine apatite in the sample (Ca₅(PO₄)₃(F, Cl)).

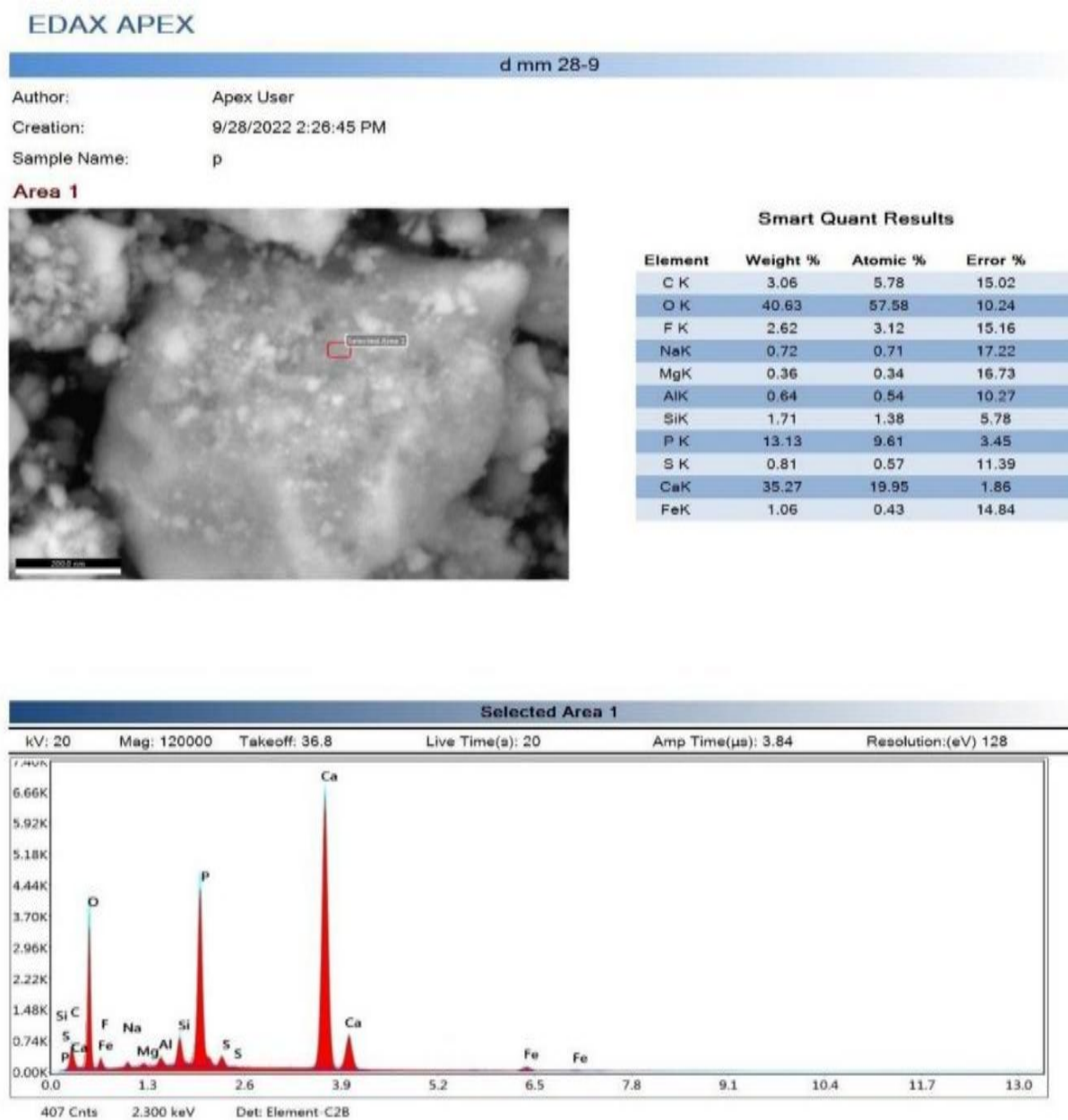


Fig. .2 EDAX analysis of the origin sample

Based on investigation and the particle size distribution provided in Table 2, it was observed that the liberation size between the phosphate and gangue minerals (carbonates and silicates) existed within the size range of (-250+45 μm). To enhance this liberation and facilitate further processing, the sample underwent grinding using a rod mill. The grinding process was conducted for a duration of 15 minutes, utilizing six rods, with the solid content maintained at 60%. This operational configuration was employed to prevent any loss of the sample and mitigate the adverse effects associated with the generation of slimes. The goal of this grinding procedure was to optimize the liberation of valuable phosphate minerals from associated gangue, thereby improving the efficiency of subsequent beneficiation processes.

Table 2. Chemical analysis and size distribution of the particles.

SIZE, MM	WT %	P ₂ O ₅ %	I.R.%	L.O.I.%
5	0	0	0	0
-5+4	6.77	10.11	24.77	14.94
-4+3.3	6.06	13.61	20.49	18.65
-3+2	17.00	18.42	19.16	15.72
-2+1	20.37	21.06	13.05	13.99
-1+0.83	5.07	20.09	10.61	13.06
-0.83+0.58	7.88	20.43	10.59	11.99
0.58+0.4	6.53	25.76	9.06	11.03
-0.4+0.25	10.31	26.31	8.52	10.35
-0.25+0.125	12.22	27.62	6.44	10
-0.125+0.075	3.20	28.21	8.25	18.82
-0.075+0.045	2.18	22.49	10.44	11.46
-0.045	2.42	13.18	23.68	15.12
Feed %	100.00	21.04	13.53	13.49
Assay%	100.00	21.13	13.13	13.31

Effect of oleate concentration on phosphate flotation

The flotation of the sample sized (-250+45 μm) was conducted for a duration of 5 minutes at room temperature, and the results are illustrated in Figure 3. It was observed that the weight percentage (% wt.) of the float fraction (concentrate) increased proportionally

with the collector dose. Additionally, both the grade and recovery of phosphate increased notably at a collector dose of 2.5 kg/t.

Specifically, at this dosage, the obtained concentrate exhibited a phosphorus pentoxide (P₂O₅) content of 29.37%, with a corresponding recovery ratio of 78.48%. The recovery ratio was calculated using the formula: (Yianatos, Moys, Contreras, & Villanueva, 2008)

$$\text{Recovery} = \frac{\text{WT\% float fraction} \times \text{grad\%}(P_2O_5)}{P_2O_5\% \text{ in feed}}$$

This indicated that the flotation process, particularly at the specified collector dosage, was effective in concentrating phosphate minerals, resulting in a higher-grade concentrate with a significant recovery of phosphorus content from the feed material.

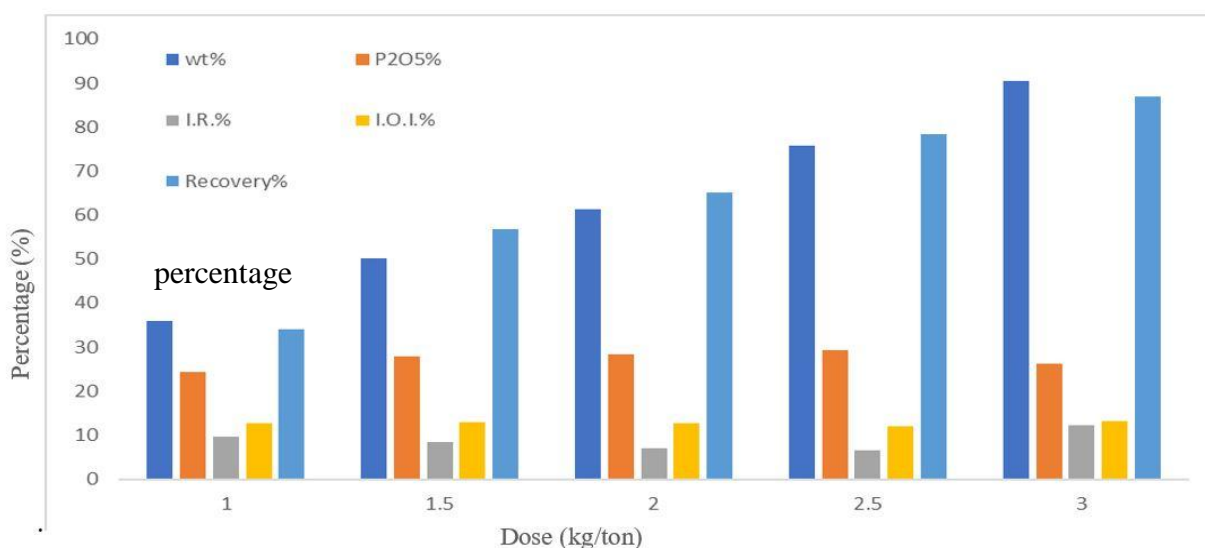


Fig3. Effect of collector dose on flotation behavior of phosphate flotation.

Simultaneously, the insoluble residue (I.R.) content decreased to 6.55% with an increase in the collector dose to 3 kg/t, representing approximately 90% by weight of the floated sample. However, a reduction in selectivity towards phosphate was observed at higher

collector doses, indicative of an overdose effect. This observation aligns with findings by Yehia, *et al.*, (2019) under pH 10 conditions.

This phenomenon can be elucidated by the molecular orientation of oleate on the mineral surface. At lower dosages, oleate molecules tend to align horizontally, resulting in suboptimal flotation outcomes. With an escalating concentration of oleate, the collector molecules reorient themselves obliquely, eventually adopting a vertical orientation at higher concentrations. This geometric transformation induces the formation of hydrophobic clusters on the mineral surface, enhancing its floatability, as illustrated in Figure (4a). Paradoxically, this leads to increased hydrophobicity and a subsequent decrease in flotation recovery. A schematic diagram, depicted in Figure (4 b, c), illustrates the progressive geometric orientation of oleate species with increasing concentration, as proposed by Yehia, *et al.*, (2017).

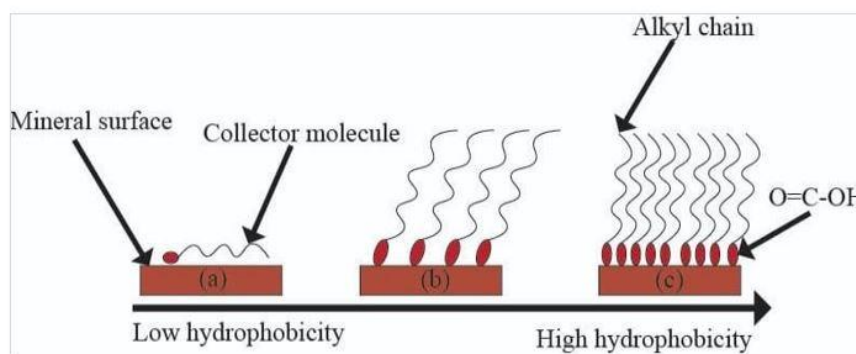


Fig. 4. Schematic representation of the orientation of collector molecules on the mineral.

After (Yehia, *et al.*, 2017)

pH Effect on phosphate flotation

The pH value is widely acknowledged as a critical variable in the flotation process. Variations in the relative acidity and alkalinity exert a profound influence on the behavior of other parameters, particularly owing to the hydrolysis of fatty acids in aqueous solution. The varied species formed are contingent upon the pH, and the surface charge is directly affected by the medium's pH (Lazo *et al.*, 2017).

Figure (5) illustrated the recovery of phosphate ore as a function of pH, employing a 2.5 kg/t oleic acid/kerosene mixture at pH 10. Under these conditions, a concentrated product was achieved, assaying 29.37% P₂O₅, with an exceptional recovery rate of 92.93%. This underscores the significant impact of pH on the flotation performance, emphasizing the pivotal role it plays in achieving optimal recovery and concentrate grade in the beneficiation of phosphate ore.

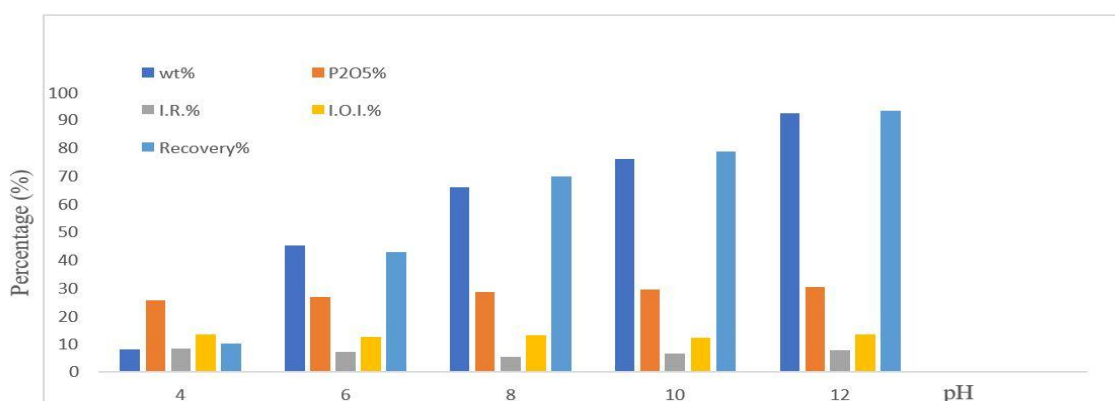


Fig. 5. pH effect on the flotation of phosphate ore as a function of pH using 2.5 kg/t oleic acid/kerosene mixture.

The observed phenomena depicted in Figure (6) can be elucidated through the understanding of oleic acid behavior in aqueous solution, particularly its role as a weak acid collector. Oleic acid undergoes hydrolysis, leading to the formation of complex species with markedly distinct solubilities and surface-active characteristics. This is explained by the following chemical equilibria. In the acidic region of the oleate/water equilibrium diagram, oleic acid primarily exists in its molecular form, particularly below pH 5 (Lega, 1982). In this state, oleic acid is physically adsorbed on the mineral surface, resulting in low flotation recovery.

In the alkaline region, competition arises between OH⁻ ions and RCOO⁻ species for the mineral surface. Additionally, at high pH values, the formation of micelles is favored. Consequently, soap derived from oleic acid is completely dissociated, attributed to micelle formation. Micelles, due to their structure, tend to be hydrophilic. The cessation of flotation

at high pH values, where the soap is entirely dissociated, can be attributed to the formation of micelles (Somasundaran and Kulkarni, 1980). This intricate interplay between pH and the speciation of oleic acid highlights the significance of pH control in optimizing the performance of oleic acid as a collector in the flotation process.

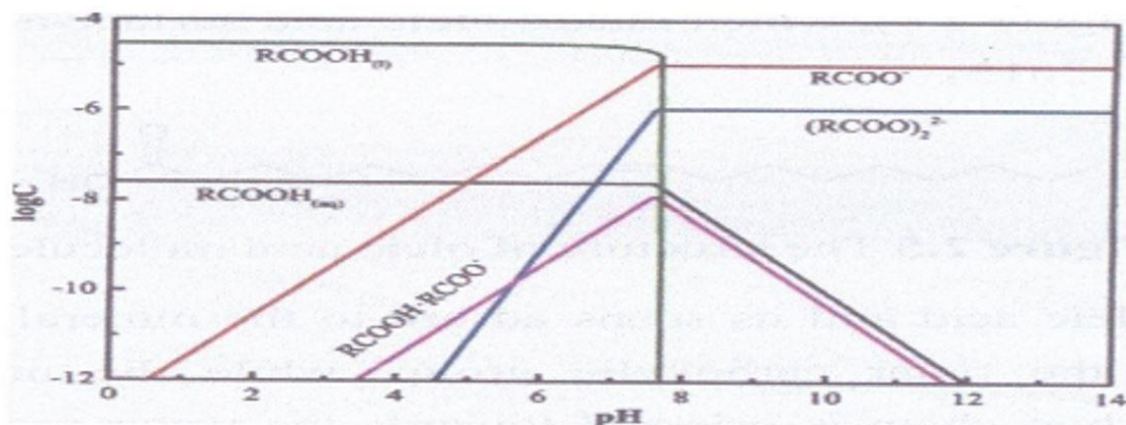


Fig 6. Species distribution diagram of oleate as a function of pH at 25°C for oleate concentration of 3×10^{-3} mol/l. After (Somasundaran and Kulkarni, 1980).

The surface tension (γ) - pH graph of oleic acid in Figure (7) reveals that the maximum reduction in surface tension occurs in the pH range of 8-10. This behavior can be ascribed to variations in the extent of dissociation of oleic acid with changing pH. The lower surface tension is typically achieved within the pH range where dissociation occurs (Purell and Sun, 1963). The observed reduction in surface tension is attributed to the co-adsorption of surfactant species at the air/water interface, further influencing the interfacial properties of oleic acid.

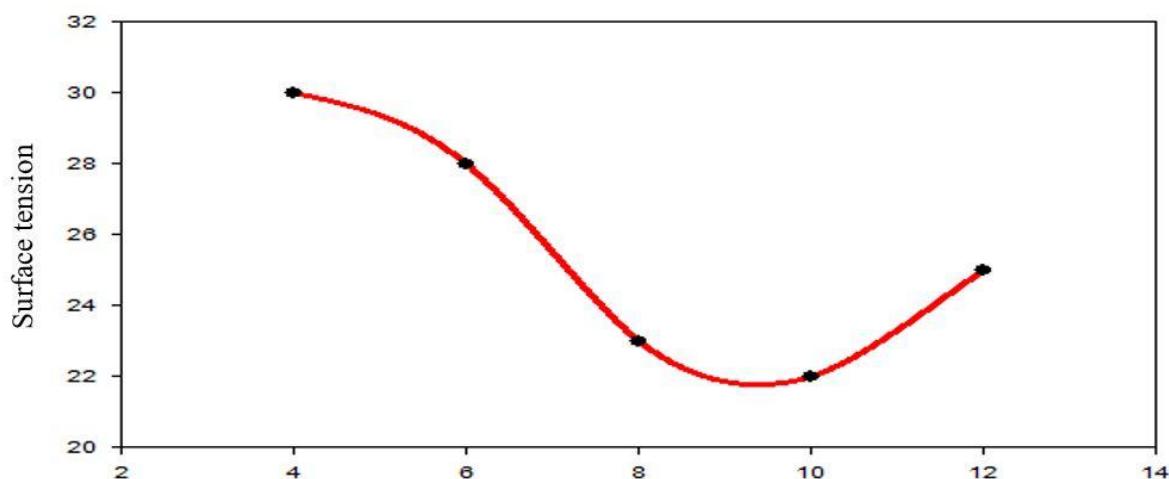


Fig. 7. Surface tension of oleic acid at different pH, and concentration of 10^{-5} M.

7. Effect of solution temperature on the flotation of phosphate

The flotation process is intricately controlled by the interaction of three phases: the mineral to be floated, the aqueous solution, and the gaseous phase. Temperature played a crucial role in governing the various phenomena associated with these phases, particularly as it is believed to induce oxidation of the double bond present in the hydrocarbon chain of fatty acids (Hung *et al.*, 2005).

In the context of phosphate flotation, the impact of temperature was illustrated in Figure (8) using 1 kg/t of the collector. The phosphate is conditioned with an oleate/kerosene mixture at pH 9 for five minutes at different temperatures (30, 40, 50, 60 °C). Notably, there is limited flotation observed at 30°C. As the temperature increased from 30°C to 40°C, the flotation grade rises, reaching 32.15% P_2O_5 with an 80.01% P_2O_5 recovery ratio.

However, further increasing the temperature to 60 °C leads to a decrease in both insoluble residue (I.R.) percentage (4.61%) and loss of ignition (L.O.I.) percentage (10.1%). The recovery also declines. This was attributed to the denaturation of the fatty hydrocarbon chain, resulting in a loss of selectivity towards phosphate. The denaturation effect at elevated temperatures leads to a decrease in both grade and recovery, underscoring the sensitivity of the flotation process to temperature variations. This aligns with findings by

Yehia *et al.* (2019), emphasizing the significant role of temperature in phosphate fatty acid flotation.

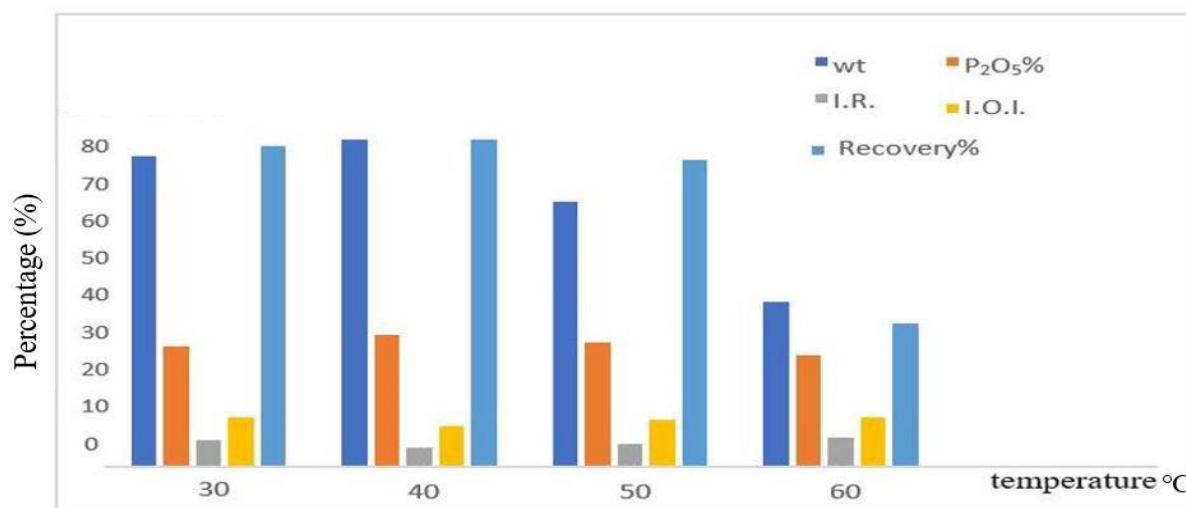


Fig. 8 The effect of different temperature degrees on phosphate flotation

Zeta Potential of Pure apatite & silicate Different pH

The surface charge characteristics of minerals can be determined at an ionic strength of 10^{-2} M NaCl across a wide pH range, as illustrated in Figure 9. The negative charge increases with rising pH, and the zero point of charge (ZPC) was measured at pH 4.5 for apatite and 1.8 for quartz. These values are consistent with findings reported by other authors, such as Mishra, (1978) and Merma et al, (2013). The ZPC is a crucial parameter in understanding the electrical neutrality of the mineral surface under varying pH conditions.

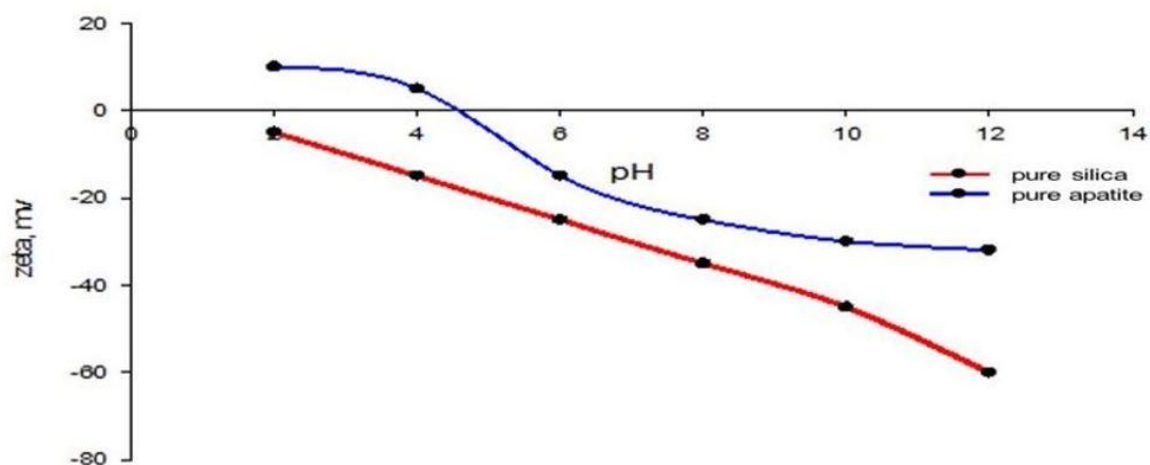


Fig. 9. Zeta potential of pure apatite and pure quartz in different pH.

Furthermore, the addition of oleate onto pure minerals has a significant impact on the ZPC of apatite, leading to a displacement of the zero-point charge. In contrast, there is a comparatively minor effect on the quartz surface. This observation in Figure 10 suggested that oleate adsorption on the phosphate surface is more pronounced than on the silica surface. Such insights into surface interactions are essential for comprehending the mechanisms involved in flotation processes, providing valuable information for optimizing mineral separation techniques.

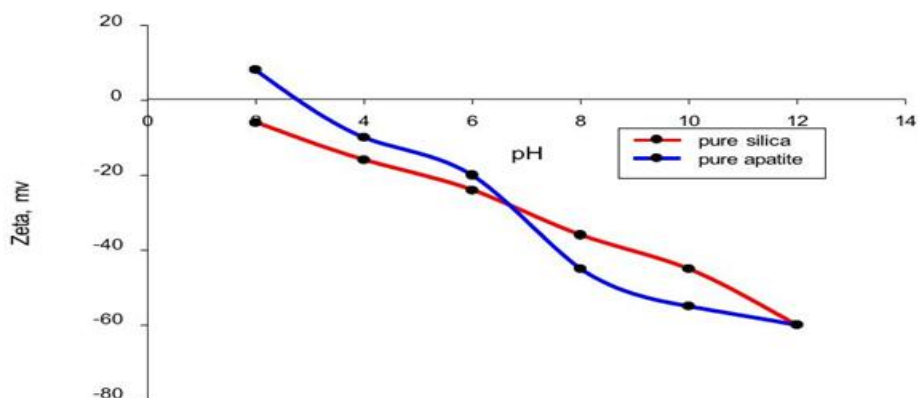


Fig. 10. Effect of solution pH on zeta potential of pure minerals (10^{-4} M oleat)

Adsorption Measurements

The adsorption density of the collector on the two surfaces of pure minerals was examined across the entire pH range, revealing the differential effect of oleate on the apatite and quartz surfaces, as described by (Kosmalski, 2020). The adsorption density of oleate (5×10^{-4} M) on both apatite and silica surfaces at pH 8-9 is depicted in Figure 11. Notably, the adsorption density is higher on the apatite surface compared to the silica surface. This observation aligns with the earlier determination of the zero point of charge (ZPC), where the apatite surface was found to be more hydrophobic and, consequently, more floatable than the silica surface. These findings collectively emphasized the preferential adsorption of oleate on the apatite surface, further highlighting the selective nature of the flotation process based on surface properties.

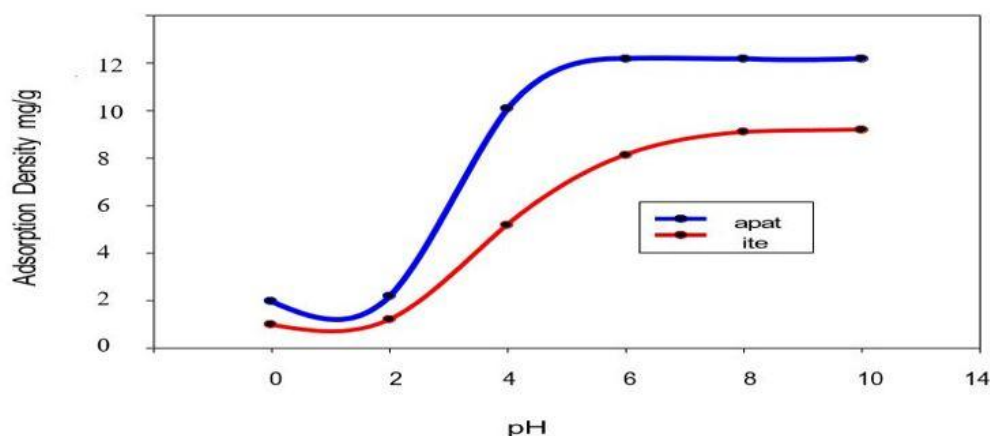


Fig. 11. Adsorption density of oleate at phosphate surface for 5 min. and conc, 4×10^{-5} M at different pH

CONCLUSION

Fatty acids and their soaps serve as common collectors in the flotation of oxide and sparingly soluble minerals. However, their utility is hindered by sensitivity to slimes and ions, elevated temperature requirements, susceptibility to pH variations, and relatively high consumption with increasing prices. To mitigate these challenges, an oily collector, such as

fuel oil, is often employed in conjunction with fatty acids. This not only reduces consumption but also minimizes excess foaming associated with fatty acids.

The effectiveness or selectivity of phosphate ore flotation is significantly influenced by the mineralogical structure, flotation reagents, and the medium. Highly crystallized and coarse-grained phosphate ores exhibit favorable processibility, with flotation being the dominant method in their beneficiation. Optimal conditions, including pH 10 where the collector (oleic acid) is present in hydrolyzed forms, result in obtaining a concentrate with the highest grade. Following a thorough examination of factors affecting oleate activity and under optimum conditions of dosage, pH, and temperature, a concentration is achieved containing 32.15% P₂O₅, with an 80.01% P₂O₅ recovery. The concentrate, with an insoluble residue (I.R.) content of 4.61% and a loss of ignition (L.O.I.) of 10.1%, holds promise for various industrial applications, particularly in the production of phosphoric acid and fertilizers.

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List of Abbreviations

P ₂ O ₅	phosphorus Penta oxide
L. O.I	loss of Ignition
I.R.	Insoluble Residue
γ	surface Tension
M	mole\liter
Recovery	Distribution % of valuable minerals in the concentrate
Zeta, mv	the number of charges (-ve or +ve) on the mineral surface
Adsorption density	the amount of collector absorbed on the mineral particles per mg/g.
XRD	X-Ray Diffraction analysis
XRF	X-Ray fluorescence

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إثراء خام الفوسفات المصري منخفض الجودة باستخدام تكنولوجيا التعويم

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المستخلص

تعتبر عمليات رفع جودة خام الفوسفات منخفض الجودة أمراً ضرورياً بالنسبة للتنمية المستدامة. وبمراجعة العديد من الدراسات العلمية والصناعية حول كيفية استغلال وإثراء الخامات منخفضة الجودة وجد أن عمليات التعويم الأيوني تعتبر من أكثر الطرق المناسبة لرفع جودة خامات الفوسفات لدرجة تسمح بان يكون صالحاً لإنتاج حامض الفوسفوريك. حيث تم استخدام عينة منخفضة الجودة من خام الفوسفات الذي يحتوي على 21% من خامس أكسيد الفوسفور وشوائب الكالسيوم والسليكات. تم طحن العينة بطاحونة القطبان لتصل إلى حجم (-250+45 ميكرون) ودراسة العوامل المؤثرة على عمليات التعويم من كمية المجمع والأس الهيدروجيني ودرجة الحرارة باستخدام خليط من حمض الأوليك والكيروسين (كمجمع) لخام الفوسفات وعند الظروف المثالية للتعويم تم الحصول على ركاز مناسب اقتصادياً وبيئياً يحتوي على 32.23% من خامس أكسيد الفوسفور، 4.67% (مواد غير ذائبة) تمثل السليكا و 10.10% فاقد الحرق (تمثل الكربونات) مع نسبة استرداد 80.01% من اصل يحتوي على 21.50% خامس أكسيد الفوسفور و 13.03% مواد غير ذائبة و 13.13% فاقد الحرق.