



Enhancing Abu Tartur Phosphate Ore Beneficiation Using Novel Collectors



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M. M. Ahmed^{*1}; Mohamed M. A. Hassan¹; A. M. Saleh²; El-Sayed R. E. Hassan³

¹Faculty of Engineering, Mining & Petroleum Engineering Department, Al-Azhar University, Qena, Egypt

²Faculty of Engineering, Mining & Petroleum Engineering Department, Al-Azhar University, Cairo, Egypt

³Metallurgical Research & Development Institute (CMRDI), Helwan, Cairo, Egypt

Abstract

Phosphate ore requires processing to reduce the presence of gangue minerals like silicates, carbonates, and clays to meet the standards of phosphate industry. Enhancing phosphate beneficiation is essential to maximize resource utilization and support sustainable fertilizer production. This research explored the enhancement of Abu Tartur phosphate ore through beneficiation using several novel collectors including 2, 2-(Octadecyl azanediyl) di-ethanol, and 2-oxobutanol compared to oleic acid. Parameters affecting flotation, such as pH value, collector dose, and depressant dose, was studied. Also, measurements of zeta potential were conducted to follow up the interaction between the studied collectors and the phosphate sample. The results revealed that column flotation technology is a promising method for the beneficiation of Abu Tartur phosphate ore. Among the collectors tested, 2-(Octadecyl azanediyl) di-ethanol proved as the most effective, achieving a phosphate concentrate with 27.80% P_2O_5 with a high recovery rate of 88.50%, starting from a sample containing 20.60% P_2O_5 . Also, under optimum flotation conditions using 2, 2-(Octadecyl azanediyl) di-ethanol, the total content of rare earth elements (REEs) was enriched to 1135.35 ppm.

Keywords: Abu Tartur phosphate ore, mini-column flotation, novel collectors, total rare earth elements

1. Introduction

Phosphate ore is an important economic deposit in Egypt. Three main mine areas are found in Egypt, namely; the Western Desert between El-Kharga and El-Dakhla Oases (Abu Tartur area), the Nile valley near Idfu and along the Red Sea between Safaga and Quesir [1]. Abu Tartur phosphate deposit is one of the largest phosphates mining area (1000 million tons and 200 million tons proved) in the Middle East. This mining area is located in the Western Desert of Egypt, about 60 Km from El-Kharga City and 10 Km from the main road between El-Kharga and El-Dakhla Oases [2]. Phosphate ores in this area are of sedimentary origin and of the apatite group, of which the most commonly encountered, variants are; fluorapatite and francolite [3]. Recently, rare earth elements (REEs) play a critical role in civilization and human life, since they are widely utilized in most everyday applications because of their unique chemical and physical properties. These elements have many applications such as reduction of energy consumption, increasing energy efficiency and advanced lifestyle consumer and other medical applications [4-5].

Several pre-processing and processing methods are defined [6]. These are based on the ore type, associated gangue minerals and the amount of impurities, as well as factors such as the degree of liberation of apatite minerals, the cost of the beneficiation method [7]. The methods employed include gravity separation [A], magnetic separation [8], electrostatic separation [9], size reduction, screening [10], attrition, scrubbing, classification [11], heavy media separation [12], calcination [13], acidic leaching [14], direct flotation [15], reverse flotation [16] or the use of multiple methods. Sedimentary rocks have various chemical and mineralogical compositions in the gangue phase [17].

Fatty acids are the most commonly used collectors in direct flotation of the phosphate ores. The long chains of fatty acids and their salts are the most extensively used especially oleic acid or sodium oleate. The effect of oleic acid dosage on flotation performance of phosphate ore was evaluated [18].

Phosphate ore processing techniques depend on the specific phosphate minerals present as well as the nature of the associated gangue. To satisfy the stringent requirements of the fertilizer industry, the ore must be upgraded to approximately 30% P_2O_5 through efficient beneficiation processes [19]. For over fifty years, flotation has been the main method employed in the industry for enhancing phosphate quality. Globally, more than 60% of commercial phosphate is generated through this process [20]. The flotation process not only upgrading the P_2O_5 grade but also reducing the particle size to 100 mesh content more than 90% [20]. Current methods for floating sedimentary phosphate ore can be broadly categorized into three types: (i) direct-reverse flotation (or reverse-direct flotation), (ii) single-reverse flotation, and (iii) double-reverse flotation [21-22]. Earlier research on concentrating phosphate ores through flotation primarily employed oleic acid as the flotation collector [23-22]. This study investigates novel collectors, including 2,2-(Octadecyl azanediyl) di-ethanol and 2-oxobutanol, which would enhance phosphate beneficiation selectivity and efficiency. This study aims to optimize the beneficiation of Abu Tartur

*Corresponding author e-mail: elkady1500@gmail.com; (M. M. Ahmed).

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phosphate ore, containing 20.60% P_2O_5 with a feed size fraction of $-250 +45 \mu m$. Direct flotation experiments were conducted to evaluate these collectors, alongside oleic acid, under varying pH values, collector dosages, and depressant dosages. Furthermore, measurements of zeta potential were conducted to evaluate the interactions between the collectors and the phosphate and silica minerals across a broad pH spectrum. This innovative approach offers a promising alternative for efficient phosphate and rare earth element (REE) recovery.

2. Experimental

2.1 Sample Preparation

The phosphate sample underwent primary and secondary crushing leading to a product of 100% -6.3 mm . Sampling of the crushed product was carried out by a 'Denver' Jones riffle to about 15 kg batches. One of these batches was ground using rod mills to reach the liberation size, at 0.250 mm . The resulting ground material was deslimed at 0.045 mm screen. The fraction $-0.250 +0.045 \text{ mm}$ was utilized as input for the flotation separation process. The original and treated samples were finely ground to a particle size of $-75 \mu m$ for analysis using XRD, XRF, ICP, and chemical methods.

2.2. Chemical Analysis

Complete chemical analysis of the sample was conducted by X-ray fluorescence (XRF). For routine chemical analysis, acid insoluble residue (A.I.) was determined using standard method of opening and dissolution of the samples (using HCl and HNO_3). Silica was determined using the standard gravimetric method while the filtrate solution was used for determining P_2O_5 content using the standard spectrometric methods. The content of rare earth elements was measured using inductively coupled plasma atomic emission spectroscopy (ICP-AES).

2.3. X-ray Diffraction Analysis

Identification of the mineral composition of the considered sample was conducted by X-ray diffraction (XRD), using the data of the powder pattern of the ASTM on cobalt radiation target with Fe filter at 30 kV and 20 mA. A 'Philips' X-ray diffractometer (PW 1730) was used. The scanning was limited from $2\theta=1$ to $2\theta=80$ -degree range.

2.4. Zeta potential test

Measurements of Zeta potential for pure apatite and quartz samples were conducted utilizing a Brookhaven Zeta-Plus analyzer. The experiments were conducted under ambient conditions with a 1 mM KCl solution serving as the electrolyte. A sample of 0.5 g of $-45 \mu m$ mineral particles was dispersed in 80 mL of the 1 mM KCl solution. The suspension was stirred magnetically for 45 minutes at a constant speed of 250 RPM and then allowed to settle. The acidity or alkalinity (pH) of the mineral suspensions was adjusted to a variety of 2 to 12 using a NaOH solution. The prepared suspension was then transferred to the measurement of zeta potential cell, and the final pH of the supernatant liquid was recorded. Each measurement was repeated three times, and the average value was taken to ensure accuracy.

2.5. Mini-Column Flotation Experiments

The experiments involving flotation of Abu Tartur phosphate ore sample were carried out utilizing a modified mini-column flotation cell. The mini-column flotation cell is made of Perspex glass with 30 cm height and 2.0 cm diameter. A sample of about 3 gm was conditioned at 400 rpm. Then a dose of sodium silicate as a depressant was added after 5 min. Sodium hydroxide or sodium carbonate was used as a modifier for the pH. Then the collector was added for 5 minutes, after which the speed was increased to 500 rpm and air flow rate was set to the desired level 25 mL/min. Both froth fraction and tailing were filtered dried and weighted before their analysis.

During this study, a direct flotation of phosphate ore sample is conducted using new collectors including, 2, 2-(Octadecyl-azanedyl) di-ethanol, and 2-Oxobutanol. Applying oleic acid collector to evaluate the outcomes of different collectors. The different parameters were studied at different levels to reach to the optimum conditions.

3. Results and Discussion

3.1. Characterization and Mineralogy of Phosphate Ore

Figure 1a and 1b represents the mineralogical study of Abu-Tartur phosphate ore which revealed that iron is found as pyrite, ankerite, clay minerals and iron oxide. Iron oxide occurs as cryptocrystalline aggregates of red to brown particles and is confined to the weathered outcrops.

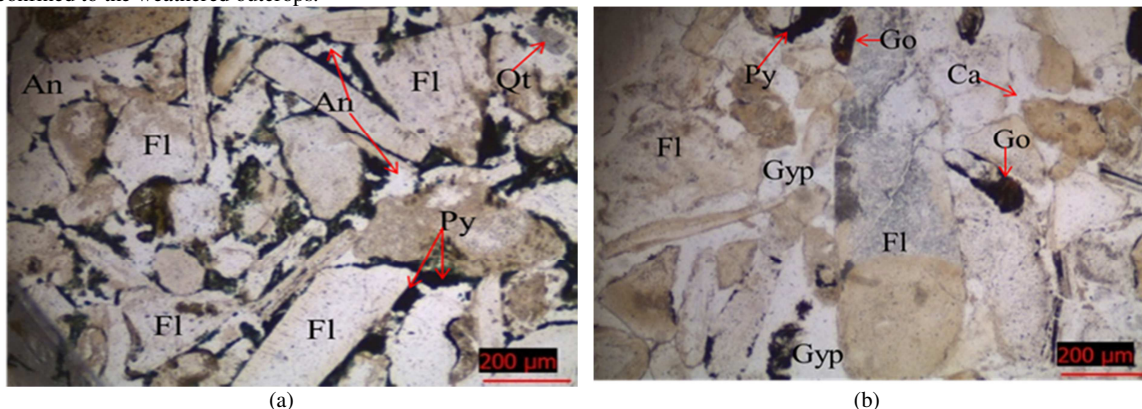


Figure 1(a,b): The mineral constituents of the Abu-Tartur phosphate ore.

The mineralogical investigation of the representative sample using XRD indicated that the sample consists mainly of the following minerals shown in Table 1 and Figure 2. As noticed in Figure 2, the phosphate sample is dominated by the main minerals in Abu Tartur phosphate sediments which include: fluorapatite, gypsum, quartz, and calcite. The phosphate-bearing minerals have been reported in all ore types as carbonate fluorapatite "francolite" and fluorapatite.

Table 1: Mineralogical composition of Abu Tartur phosphate ore

| Mineral Name | Chemical Formula |
|--------------|---|
| Gypsum | $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. |
| Quartz | SiO_2 . |
| Fluorapatite | $\text{Ca}_5(\text{PO}_4)_3\text{F}$. |
| Goethite | FeO_2H |
| Calcite | CaCO_3 |

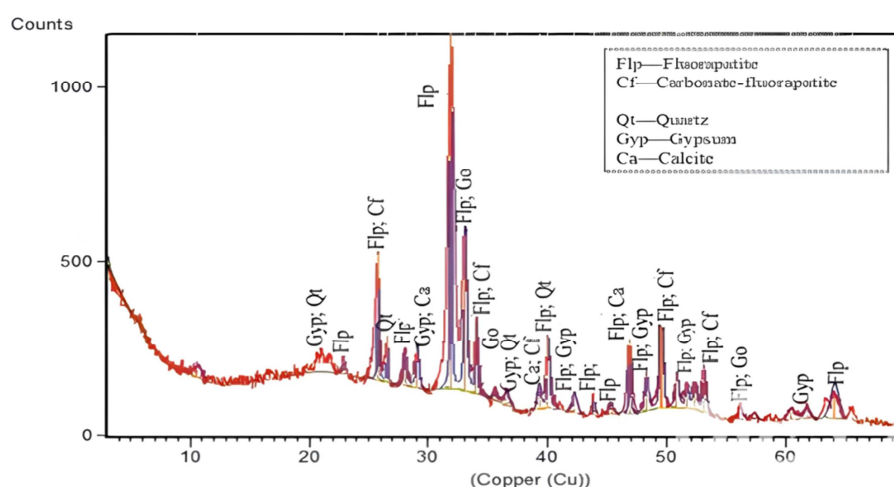


Figure 2: XRD pattern of phosphate ore sample.

The chemical analysis of the representative sample using XRF demonstrated that the sample composed of the following major elements shown in table 2. The sample contains a lower content of P_2O_5 (20.62%) and higher amount of CaO (34.51 %) suggesting the existence of additional phases.

Table 2: Chemical composition of Abu-Tartur ore sample

| Comp. | P_2O_5 | Al_2O_3 | MgO | Fe_2O_3 | CaO | K_2O | Na_2O | SiO_2 | SO_3 | F | CO_2 | L.O.I. | Σ REOs |
|--------|------------------------|-------------------------|------|-------------------------|-------|----------------------|-----------------------|----------------|---------------|------|---------------|--------|---------------|
| Wt., % | 20.62 | 2.25 | 1.52 | 6.76 | 34.51 | 0.14 | 0.69 | 8.59 | 4.79 | 2.09 | 6.51 | 11.53 | 0.08 |

3.2. Micro-Column Flotation of Abu Tartur Phosphate Ore Sample

3.2.1. Micro-Column Flotation Using Oleic Acid Collector

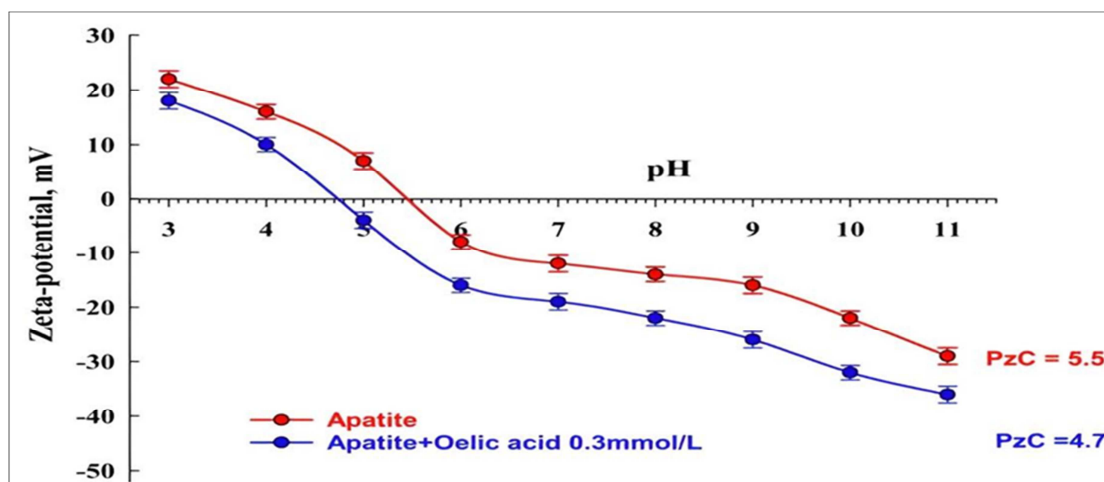
3.2.1.1. Zeta Potential Measurements

The zeta potential of the apatite was evaluated over a broad pH spectrum in the presence of oleic acid, as illustrated in (Figure 3a), the value and sign of the zeta potential are influenced by the pH of the medium. The zeta potential transitions from positive to negative as the pH increases. Additionally, the electronegativity of the zeta potential increases gradually with rising pH.

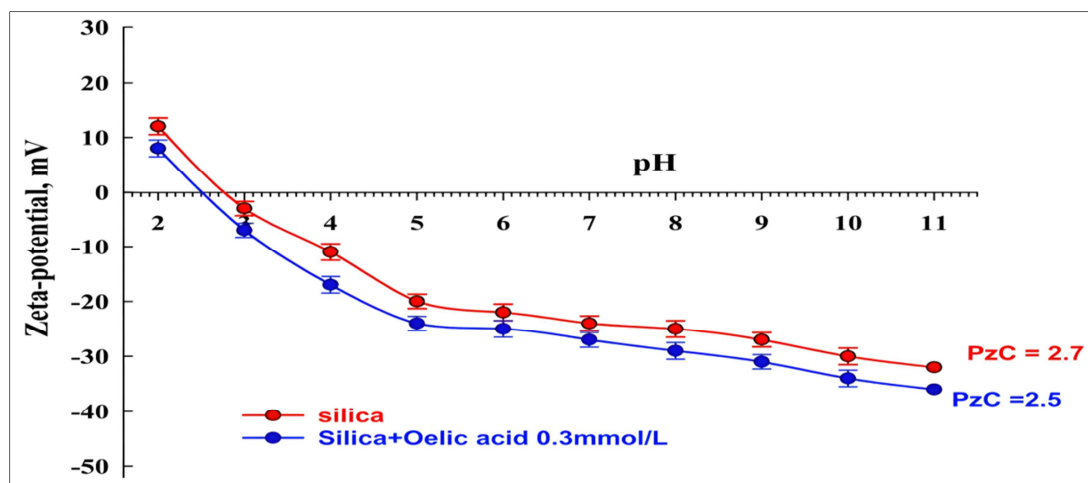
Zeta potential measurements of apatite and silica were conducted across different pH levels to explore the chemical interactions between the reagents and minerals, aiming to comprehend the adsorption behavior of the oleic acid collector. Apatite exhibits positive zeta potentials between pH 3 and 5.5, but the potentials become negative at pH levels above 5.5. The addition of the oleic acid collector results in a negative shift in the zeta potential of apatite within the pH range of 3 to 11. Additionally, it causes the point of zero charge to shift from 5.5 to 4.7, indicating a robust engagement between oleic acid and the apatite surface. The decrease in the zeta potential of quartz is lower when oleic acid is added in the pH range of 3–11,

indicating a weak interaction between oleic acid and the quartz surface (Figure 3 b). Also, the point of zero charge is slightly changed from 2.7 to 2.5.

At pH range from 8 to 10, the zeta potential of apatite in the presence of oleic acid has high negative alteration compared to quartz these results indicate a strong adsorption of apatite (phosphate mineral) with oleic acid compared to quartz. For phosphate mineral, adjusting the pH to levels above the ZPC, the surface charge of apatite mineral becomes more negative. At pH values above the ZPC, the oleic acid molecules are ionized. Ionization improves the ability of oleic acid to attach to the phosphate surface, enhancing the efficiency of the flotation process. Furthermore, the hydrolysis of oleic acid collector at these pH levels ensures that the collector molecules are in the right form to adsorb effectively onto the phosphate mineral.



(a)



(b)

Figure 3 (a,b): Zeta potential of a) the phosphate mineral apatite and b) quartz before and after treatment with the oleic acid collector.

3.2.1.2. Effect of pH Value

The pH value is a critical factor influencing phosphate flotation, affecting the optimal sequence of pH adjustment procedures, optimal points, and levels of additive usage. The pH of the solution dictates the ionization and hydrolysis of the collector, which in turn aids or hinders its adsorption at various ionized solid/liquid interfaces, impacting the selectivity of flotation [24]. Flotation tests are conducted by varying the pH from 2 to 10 to assess its impact on collector performance in phosphate flotation. The results, detailed in Table 3 demonstrate that increasing the pH from 8 onwards enhances flotation efficiency. The best concentrated grade with P_2O_5 24.50% and a recovery of 81.50% was achieved at pH 9.5, beyond which the grade declined. This aligns with previous studies investigated by Kou et al., [25].

Table 3: Effect of pH value on phosphate flotation using different collectors

| pH | Oleic acid | | pH | 2, 2-(Octadecyl-azanediyl) di-ethanol | | pH | 2-Oxobutanol | |
|----|-----------------------------------|-------------|----|---------------------------------------|-------------|----|-----------------------------------|-------------|
| | P ₂ O ₅ , % | Recovery, % | | P ₂ O ₅ , % | Recovery, % | | P ₂ O ₅ , % | Recovery, % |
| 2 | 22.51 | 81.22 | 2 | 24.88 | 84.36 | 2 | 23.56 | 87.15 |
| 4 | 24.13 | 81.35 | 4 | 25.78 | 85.42 | 4 | 23.71 | 87.21 |
| 6 | 24.35 | 81.49 | 6 | 25.82 | 85.56 | 6 | 23.82 | 87.28 |
| 8 | 24.50 | 81.50 | 8 | 26.88 | 86.42 | 8 | 23.95 | 87.52 |
| 10 | 24.49 | 81.50 | 10 | 26.88 | 86.41 | 10 | 23.94 | 87.53 |

3.2.1.3. Effect of Sodium Silicate Dosage

The impact of sodium silicate dosage on phosphate flotation was examined by evaluating different dosages, ranging from 1 kg/ton to 3 kg/ton, while maintaining a constant pH of 9.5, Table 4. The study discovered 2.50 kg/ton was sufficient to attain the best P₂O₅ grade and recovery. Further addition of sodium silicate beyond 2.50 kg/ton did not improve the phosphate flotation, indicating optimal conditions at this dosage. Sodium silicate is known to enhance flotation efficiency when used because of oleic acid's ability to depress silica, thereby increasing collector selectivity and improving separation efficiency [26]. The study's findings, detailed in (Table 4) show that sodium silicate solution exhibits a greater depressing effect as it can cover a larger surface area of the silica mineral.

Table 4: Effect of Sodium Silicate Dose on phosphate flotation using different collectors

| Sodium Silicate Dose, | Oleic acid | | Sodium Silicate Dose, | 2, 2-(Octadecyl-azanediyl) di-ethanol | | Sodium Silicate Dose, | 2-Oxobutanol | |
|-----------------------|-----------------------------------|-------------|-----------------------|---------------------------------------|-------------|-----------------------|-----------------------------------|------------|
| | P ₂ O ₅ , % | Recovery, % | | P ₂ O ₅ , % | Recovery, % | | P ₂ O ₅ , % | Recovery % |
| 1 | 24.85 | 89.71 | 1 | 25.75 | 87.82 | 1 | 24.15 | 88.20 |
| 1.5 | 25.15 | 89.84 | 1.5 | 25.80 | 87.84 | 1.5 | 24.23 | 88.23 |
| 2 | 25.35 | 90.10 | 2 | 25.85 | 87.90 | 2 | 24.35 | 88.28 |
| 3 | 25.34 | 90.12 | 3 | 25.86 | 87.91 | 3 | 24.34 | 88.27 |

3.2.1.4. Effect of Oleic Acid Dosage

Fatty acids, particularly oleic acid and its salts, are the most commonly used collectors in the direct flotation of phosphate ores. This study assesses how varying the dosage of oleic acid influences the flotation performance of phosphate ore. The collector used is a 1:1 mixture of oleic acid and kerosene, which has shown promising results in phosphate beneficiation [27-28]. To comprehend how the collector influences the performance of micro-column flotation, various dosages ranging from 1.0 to 3.0 kg/ton, were tested, as illustrated in Figure 4.

The dosage of oleic acid significantly affects the efficiency and selectivity of phosphate flotation. Oleic acid selectively coats phosphate minerals, aiding their separation from gangue minerals. Optimal dosage is crucial for high phosphate recovery while minimizing unwanted mineral entrainment. Low dosages may lead to poor recovery, while excessive dosages can decrease selectivity by causing indiscriminate attachment of oleic acid to both phosphate and gangue minerals. Thus, precise control and optimization of oleic acid dosage are vital for effective phosphate flotation. Figure 4 shows that increasing the collector dosage from 1.0 to 2.5 kg/ton significantly improves P₂O₅ quality and recovery up to 26.40% and 90.30% respectively. At collector dosage of 2.50 kg/ton, the selectivity for phosphate separation is improved, resulting in higher P₂O₅ purity in the float fraction. However, oleic acid dose higher than 2.5 kg/ton resulted in lower phosphate flotation efficiency.

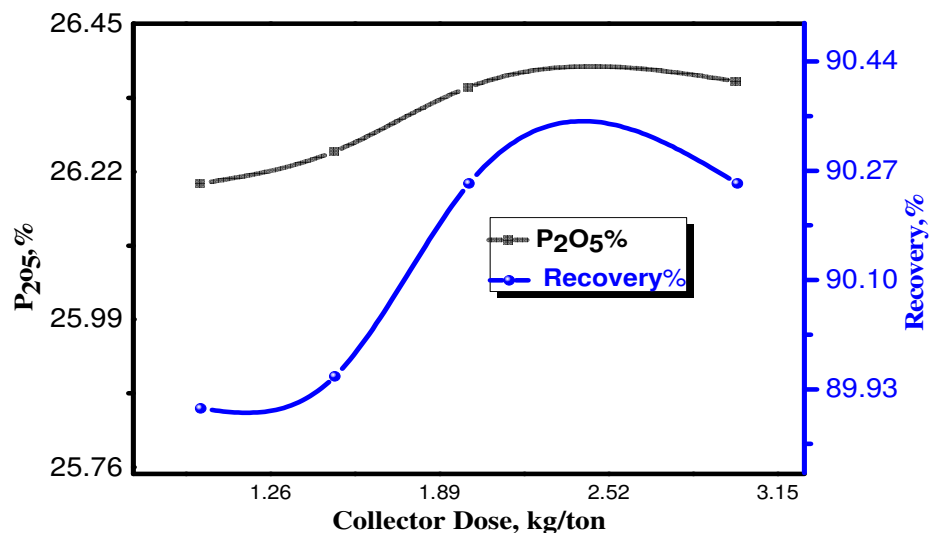


Figure 4: Effect of collector dosage on phosphate flotation

[Test condition comprised: pH 9.5, agitation speed 1200 rpm, sodium silicate 2.5 kg/ t, air flow rate 25 mL/ min and conditioning for 10 min, flotation time 5 min]

3.2.2. Micro-Column Flotation Using 2, 2-(Octadecyl-Azanediyl) di-Ethanol Collector

3.2.2.1. Zeta Potential Measurements

Measurements of zeta potential of phosphate with 2, 2-(Octadecyl-Azanediyl) di-Ethanol collector provide valuable insights into the surface charge properties of phosphate. These measurements are crucial for understanding the behavior of minerals in various pH environments and their interaction with flotation reagents, which directly impacts the efficiency of mineral separation processes as illustrated in Figure 5. The experiments showed that the zeta potential of the phosphate ore varied significantly across the pH range from 3 to 11. As expected, the zeta potential exhibited a transition from positive to negative values with the increase in pH. This behavior indicates that both H and OH ions play critical roles as potential-determining ions for these minerals. The point of zero charge (ZPC) for phosphate mineral can be identified where the zeta potential crosses zero, which is a crucial parameter to optimize flotation conditions.

Apatite exhibits positive zeta potentials in the pH range of 3 to 5.5, but these potentials turn negative when the pH exceeds 5.5. The addition of 2, 2-(Octadecyl azanediyl) di-ethanol causes a negative shift in the zeta potential of apatite across the pH range of 3-11, as well as a significant shift in the point of zero charge from 5.5 to 4.4, indicating a strong interaction between 2, 2-(Octadecyl azanediyl) di-ethanol and the apatite surface.

On the other hand, the alteration in the zeta potential of quartz is negligible when exposed to 2, 2-(Octadecyl azanediyl) di-ethanol across the pH range of 3-11, with a very slight decrease in the point of zero charge from 2.7 to 2.6, indicating a weak interaction between 2, 2-(Octadecyl azanediyl) di-ethanol and the silica surface (Figure 5 a ,b). Over a pH range to 6, the zeta potential of apatite in the existence of 2, 2-(Octadecyl azanediyl) di-ethanol shows a high negative alteration compared to quartz, indicating strong adsorption to apatite. At pH levels exceeding the ZPC, the 2, 2-(Octadecyl azanediyl) di-ethanol molecules are ionized, enhancing their ability to attach to the surface of apatite and thereby improving flotation efficiency. The zero point of charge (ZPC) for phosphate apatite minerals shifts from pH 5.5 to 4.4 in the presence of 2, 2-(Octadecyl azanediyl) di-ethanol. This shift exceeds that of caused by oleic acid (which shifts to pH 4.7), suggesting that 2, 2-(Octadecyl azanediyl) di-ethanol may result in better phosphate flotation efficiency compared to oleic acid.

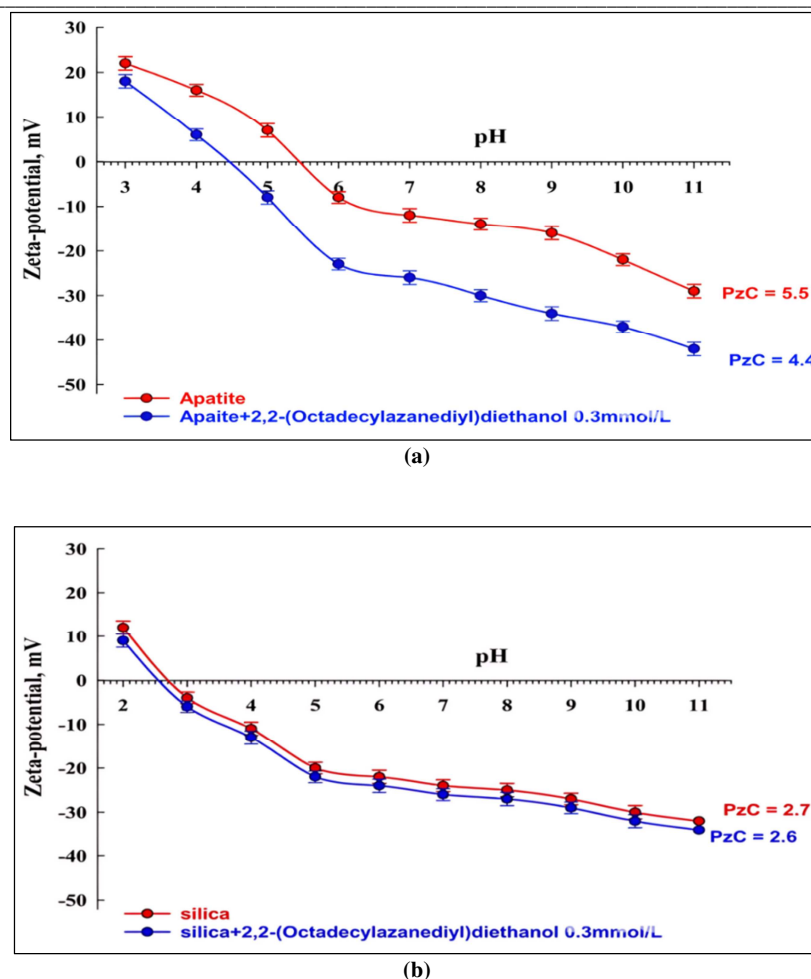


Figure 5 (a,b): Zeta potential of a) the phosphate mineral apatite and b) silica before and after treatment with the 2, 2-(Octadecyl azanediyl) di-ethanol collector

3.2.2.2. Effect of pH Value

The effect of pH on phosphate flotation in the presence of 2, 2-(Octadecyl azanediyl) di-ethanol as a novel collector was investigated, with results presented in (Table 3) pH is recognized as a crucial factor influencing phosphate flotation, as it determines the ionization and hydrolysis of the collector, thereby affecting its adsorption at solid/liquid interfaces and the selectivity of flotation [29]. Therefore, the effect of pH on the flotation performance of Abu Tartur phosphate ore sample was studied. Several experiments were conducted, varying the pH values from 2 to 10, while keeping other flotation conditions constant (particle size of 250 - 45 μm , collector dosage of 1.5 kg/ton, solid-to-liquid ratio of 1:5, flotation time of 5 minutes, agitation speed of 1200 rpm) at 25°C.

Sodium hydroxide was used as the pH regulator. The results indicate that flotation efficiency increased with rising pH values, particularly from pH 8 onwards. As expected by zeta potential measurements, pH values between 8 and 10 are optimal for the phosphate flotation. Notably, the use of 2, 2-(Octadecyl azanediyl) di-ethanol as a collector yielded superior results, especially at pH value of 9, where it achieved high-grade values of 27.0%, with recovery of 86.50%.

3.2.2.3. Effect of Sodium Silicate Dosage

The effect of sodium silicate dosage on phosphate flotation using 2, 2-(Octadecyl azanediyl) di-ethanol collector was examined by testing various dosages ranging from 0.5 kg/ton to 3.0 kg/ton, at pH value of 9.0, as shown in (Table 4). Sodium silicate is known to improve phosphate flotation efficiency by depressing silica, which increases collector selectivity and enhances separation efficiency [30]. The study revealed that an amount of 2.0 kg/ton was optimal for achieving the highest P_2O_5 grade and recovery. Increasing the sodium silicate dosage beyond 2.0 kg/ton did not further enhance the phosphate flotation.

3.2.2.4. Effect of 2, 2-(Octadecyl-Azanediyl) di-Ethanol Dosage

Figure 6 demonstrates the effect of altering the dosage of 2,2-(Octadecyl azanediyl) di-ethanol as a collector in the flotation of phosphate minerals (P_2O_5). No previous research has shown the use 2, 2-(Octadecyl azanediyl) di-ethanol as a collector for phosphate flotation using mini-column flotation cell. Therefore, various levels of collector dosages were examined: from 1.0 to 3.0 kg/ton. It is displayed that increasing the collector dosage up to 2.5 kg/ton enhanced the phosphate flotation. At this dose a product of 27.80% P_2O_5 with recovery of 88.50%. Increasing the collector dose beyond 2.5 kg/ton did not significantly improve phosphate flotation. These findings suggest that optimizing the dosage of 2, 2-(Octadecyl azanediyl) di-ethanol as a novel promising collector can enhance the retrieval and quality of phosphate minerals in flotation processes.

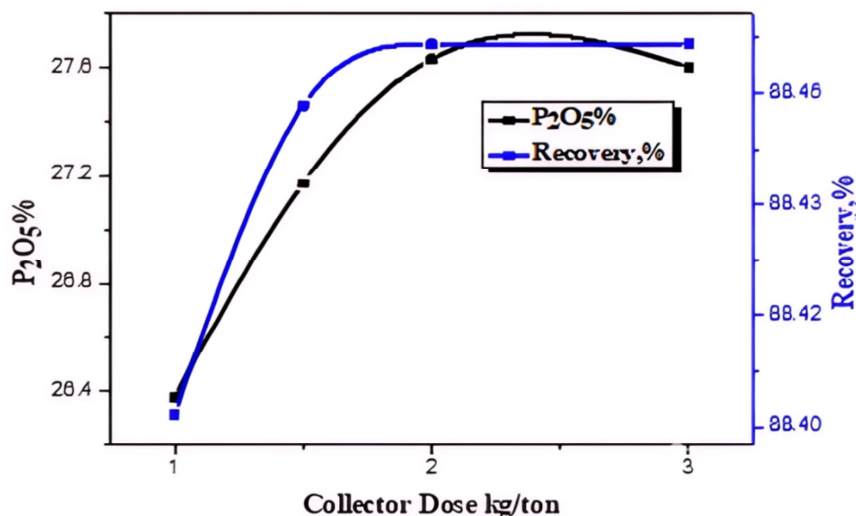


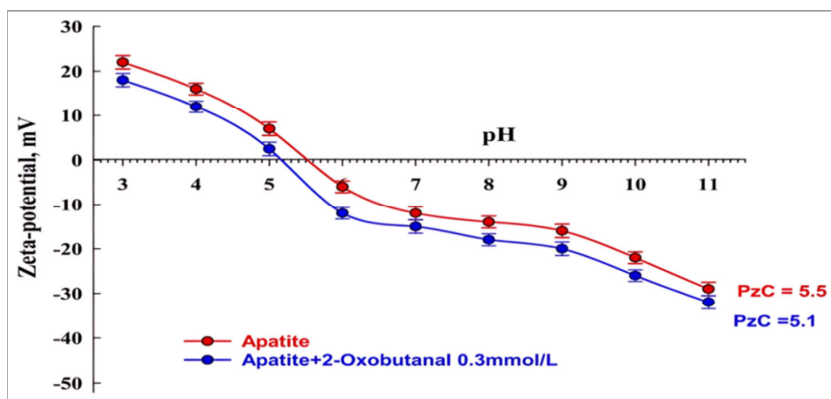
Figure 6: Effect of 2, 2-(Octadecyl azanediyl) di-ethanol dosage on phosphate flotation.

[Test condition comprised: pH 9.0, sodium silicate 2.0 kg/ton, agitation speed 1200 rpm, air flow rate 25 mL/ min, conditioning for 10 min, flotation time 5min]

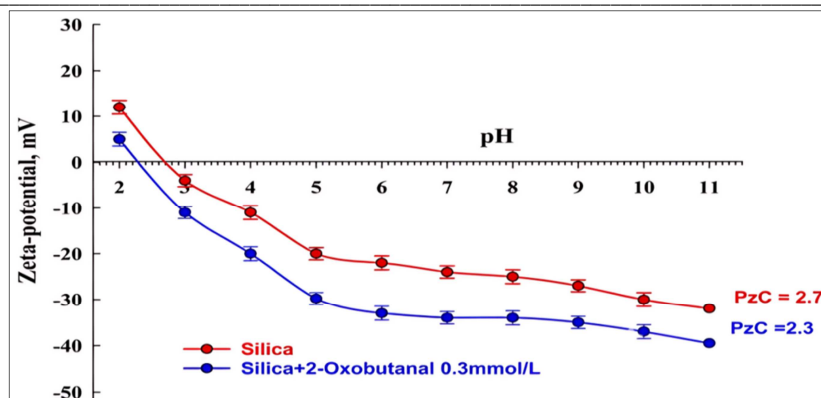
3.2.3. Micro-Column Flotation Using 2-Oxobutanol Collector

3.2.3.1. Zeta Potential Measurements

Figure 7 (a,b) presents the zeta potential measurements for both apatite and quartz when using the 2-oxobutanol collector over a broad range of pH values. The zeta potential values of both apatite and quartz showed a transition from positive to negative values as the pH increased. The zero point of charge (ZPC) for apatite is shifted from pH 5.5 to pH 5.1 in the presence of 2-oxobutanol. This shift is smaller compared to other collectors studied, indicating less adsorption on the apatite surface. While the (ZPC) for silica is shifted from pH 2.7 to pH 2.3 in the presence of 2-oxobutanol. This shift is higher than that of other studied collectors suggesting higher adsorption on silica surface. These investigations suggest lower selectivity of apatite over quartz and consequently lower phosphate flotation efficiency using 2-oxobutanol compared to other studied collectors.



(a)



(b)

Figure 7 (a,b): Zeta potential of a) the phosphate mineral apatite and b) silica before and after treatment with the 2-oxobutanol collector.

3.2.3.2. Effect of pH Value

The impact of pH on phosphate flotation using the 2-oxobutanol collector was examined, and the findings are displayed in (Table 3). The research involved adjusting pH levels from 2 to 10, as shown in (Table 3). Test conditions included 2-oxobutanol dosage of 1.5 kg/t, sodium silicate dosage of 0.5 kg/t, agitation speed of 1200 rpm, air flow rate of 10 mL/min and conditioning for 10 minutes. The results indicate, as usual for phosphate flotation, that efficiency increased with rising pH values, particularly from pH 8 onwards. The use of 2-oxobutanol as a novel collector yielded moderate results at pH value of 8.5, where it achieved a product with P_2O_5 grade of 24.00% and recovery of 87.60%. Higher pH values do not improve the phosphate flotation.

3.2.3.3. Effect of Sodium Silicate Dosage

The effect of sodium silicate dosage on phosphate flotation using 2-oxobutanol as a collector was examined by testing various dosages ranging from 1.5 kg/ton to 3.0 kg/ton, while maintaining a constant 2-oxobutanol collector dosage of 2.70 kg/ton, as shown in (Table 4). The study revealed that a dosage of 2.60 kg/ton was optimal for achieving the highest P_2O_5 grade and recovery. Increasing the sodium silicate dosage beyond 2.60 kg/ton did not further improve phosphate flotation.

3.2.3.4. Effect of 2-Oxobutanol Dosage

Figure 8 illustrates the effect of altering the dosage of 2-oxobutanol as a collector in phosphate flotation. It was noted that employing 2-oxobutanol as a collector yielded moderate results and elevated recovery rates at pH of 8.5, with dosages ranging from 1.0 to 3.0 kg/ton. The optimal results were achieved at 2-oxobutanol dosage of 2.70 kg/ton, with a P_2O_5 grade of 25.75% and recovery of 90.25%.

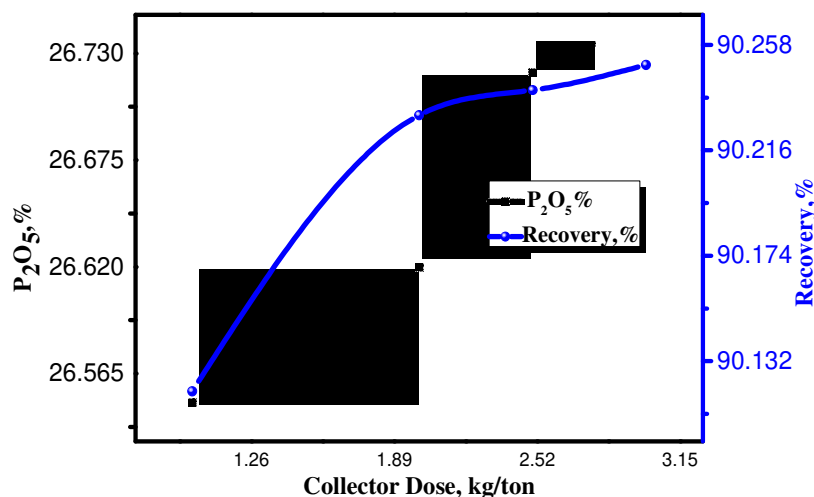


Figure 8: Effect 2-oxobutanol dosage on phosphate flotation.

[Test conditions: pH 8.5, sodium silicate 2.60 kg/ton, agitation speed 1200 rpm, air flow rate 25 mL/ min and conditioning for 10 min, flotation time 5min]

3.3. Comparison between Performances of Studied Collectors

The zeta potential of apatite (phosphate mineral) and quartz, before and after the treatment with the studied collectors, was measured over a wide pH range. It was observed that the zeta potential shifts from positive to negative with an increase in pH. The zeta potential of the apatite mineral remains positive between pH 3 and 5.5 but turns negative at pH levels above 5.5. Introducing collectors results in a negative shift in the zeta potential of apatite across the pH range of 3 to 11 and also alters the point of zero charge. This shift is much higher with 2, 2-(Octadecyl azanediyl) di-ethanol (from pH 5.5 to 4.4), indicating a robust interaction with the surface of apatite. 2-oxobutanol causes the lowest shift of the studied collectors (to pH 5.1) suggesting lower adsorption on apatite surface. This trend is reversed with the shift of (ZPC) for silica with the studied collectors. These investigations suggest a descending order of the apatite selectivity over silica and consequently in phosphate flotation efficiency as follows: 2, 2-(Octadecyl azanediyl) di-ethanol > Oleic acid > 2-Oxobutanol.

In phosphate flotation, adjusting the pH to levels above the ZPC, the electrical charge on the surface of phosphate minerals becomes more negative, improving the adsorption of reagents and thus enhancing flotation efficiency. For all the collectors examined, the optimal concentrate grade and recovery were obtained at a pH range of 8.5 to 9.5. pH values between 8 and 10 are optimal for the ionization and hydrolysis of the collector because these pH levels create conditions that enhance the performance of collectors. At these pH values, the chemical environment favors the ionization of the collector molecules, which is crucial for their interaction with the phosphate particles. Ionization increases the ability of collectors to attach to the phosphate surface, enhancing the efficiency of the flotation process.

Furthermore, the hydrolysis of the collector at these pH levels (8-10) ensures that the collector molecules are in the right form to adsorb effectively onto the phosphate minerals. This adsorption is critical for separating the phosphate from impurities, as it allows for targeted flotation where phosphate particles are preferentially floated, while silicate and other gangue minerals are left behind. So, maintaining the pH within this optimal range of 8.5 to 9.5 for the studied collectors improves the selectivity and efficiency of the flotation process, resulting in higher phosphate recovery rates and better grade. The obtained results with different collectors are summarized in Table 5.

Also, this study evaluates the effect of collector dosage on the flotation performance of phosphate ore. The dosage of collector significantly affects the efficiency and selectivity of phosphate flotation. The collector coats phosphate minerals, aiding their separation from quartz and other gangue minerals. Optimal dosage is essential for high phosphate recovery while minimizing unwanted mineral entrainment. Low dosages may lead to poor recovery, while excessive dosages can decrease selectivity by causing indiscriminate attachment of collector to both phosphate and gangue minerals. Thus, precise control and optimization of collector dosage are vital for effective phosphate flotation. At collector dosage between 2.50 and 3.00 kg/ton, the selectivity for phosphate separation is improved for all studied collectors, resulting in higher P_2O_5 grade and recovery in the float.

The effect of sodium silicate dosage as depressant for silica in the flotation of phosphate was investigated by testing various dosages. Sodium silicate is known to enhance phosphate flotation efficiency due to its ability to depress quartz, thereby increasing collector selectivity and improving separation efficiency. The results of the study show that sodium silicate solution exhibits a greater depressing effect as it can cover a larger surface area of the silica mineral. It was found that sodium silicate values from 2.00 to 2.60 kg/ton was sufficient to achieve the best P_2O_5 grade and recovery.

This study underscores that among the three collectors investigated for mini-column flotation of Abu Tartur phosphate ore sample, 2, 2-(Octadecyl-azanediyl) di-ethanol emerged as the most effective, yielding the highest quality product with a P_2O_5 grade of 27.80% and recovery of 88.50%, Table 6. The experimental findings suggest that Abu Tartur phosphate ore sample can be effectively beneficiated via mini-column flotation. This will result in a product enriched with rare earth elements, Table 7.

Figure 9 shows the X-ray diffraction of the flotation phosphate concentrate using 2,2-(Octadecyl-azanediyl) di-ethanol. It is obvious that the principal mineral included in the concentrate is apatite and gypsum, a higher concentration due to local accumulation increased crystallinity of gypsum during curing better orientation or grain growth, making it more detectable by XRD. The SEM-EDX images, figure 10 (a,b), evidence the effective separation of silicates from phosphate as well as enrichment of the total rare earth elements.

Table 5: Comparison between micro-column flotation products with different collectors

| Phosphate concentrates of micro-column flotation using different collectors | | | |
|---|--|-------------------|----------------------|
| Type of collector | Optimum conditions | Assay, P_2O_5 % | Recovery, P_2O_5 % |
| Oleic acid | pH 9.5, collector dose 2.50 kg /t and sodium silicate 2.50 kg /t | 26.40 | 90.30 |
| 2,2-(Octadecyl-azanediyl) di-ethanol | pH 9.0, collector dose 2.50 kg /t and sodium silicate 2.00 kg /t | 27.80 | 88.50 |
| 2-Oxobutanol | pH 8.5, collector dose 2.70 kg /t and sodium silicate 2.60 kg /t | 25.75 | 90.25 |

Table 6: The chemical analysis of phosphate concentrates of micro-column flotation using different collectors at optimum conditions.

| Component | Head sample | phosphate concentrates using different collectors | | |
|--------------------------------|-------------|---|--------------------------------------|--------------|
| | | Oleic acid | 2,2-(Octadecyl-azanediyl) di-ethanol | 2-Oxobutanol |
| P ₂ O ₅ | 20.62 | 26.40 | 27.80 | 25.75 |
| Al ₂ O ₃ | 2.25 | 1.35 | 1.25 | 1.62 |
| MgO | 1.52 | 0.87 | 0.81 | 0.98 |
| Fe ₂ O ₃ | 6.56 | 5.65 | 5.50 | 5.75 |
| CaO | 34.51 | 39.55 | 40.20 | 38.95 |
| K ₂ O | 0.14 | 0.12 | 0.11 | 0.15 |
| Na ₂ O | 0.69 | 0.64 | 0.59 | 0.68 |
| SiO ₂ | 8.59 | 3.79 | 2.85 | 4.82 |
| SO ₃ | 4.79 | 3.50 | 3.11 | 3.62 |
| F | 2.09 | 2.42 | 2.54 | 2.36 |
| CO ₂ | 6.51 | 5.75 | 5.55 | 5.85 |
| L.O.I. | 11.53 | 9.82 | 9.53 | 9.94 |
| Tot. REOs | 0.0790 | 0.13 | 0.14 | 0.12 |

Table 7: Σ REEs/REOs of phosphate concentrates of micro-column flotation using considered collectors.

| Component | REEs ppm | Σ REEs of phosphate concentrates using different collectors | | | |
|--------------|---------------------------------|---|------------|---------------------------------------|--------------|
| | | Head Sample | Oleic acid | 2, 2-(Octadecyl-azanediyl) di-ethanol | 2-Oxobutanol |
| Scandium | Sc | 17 | 23.50 | 26.00 | 19.45 |
| | Sc ₂ O ₃ | | 36.04 | 39.88 | 29.83 |
| Yttrium | Y | 149 | 214.85 | 232.55 | 197.20 |
| | Y ₂ O ₃ | | 272.85 | 295.32 | 250.43 |
| Lanthanum | La | 99.80 | 143.58 | 151.65 | 134.10 |
| | La ₂ O ₃ | | 168.39 | 177.85 | 157.27 |
| Cerium | Ce | 185.20 | 282.50 | 304.15 | 254.00 |
| | CeO ₂ | | 347.03 | 373.62 | 312.02 |
| Praseodymium | Pr | 57 | 86.40 | 91.20 | 74.50 |
| | Pr ₆ O ₁₁ | | 104.39 | 110.19 | 90.01 |
| Neodymium | Nd | 94.10 | 138.25 | 153.35 | 127.40 |
| | Nd ₂ O ₃ | | 161.26 | 178.87 | 148.60 |
| Promethium | Pm | 22 | 0.00 | 0.00 | 0.00 |
| | Pm ₂ O ₃ | | 0.00 | 0.00 | 0.00 |
| Samarium | Sm | 21.20 | 27.75 | 29.20 | 27.80 |
| | Sm ₂ O ₃ | | 32.18 | 33.86 | 32.24 |
| Europium | Eu | 12 | 7.30 | 8.10 | 6.85 |
| | Eu ₂ O ₃ | | 8.45 | 9.38 | 7.93 |
| Gadolinium | Gd | 30 | 41.50 | 42.45 | 38.80 |
| | Gd ₂ O ₃ | | 45.72 | 46.77 | 42.75 |
| Terbium | Tb | 15 | 5.55 | 5.90 | 6.50 |
| | Tb ₄ O ₇ | | 6.53 | 6.94 | 7.65 |
| Dysprosium | Dy | 16,20 | 22.05 | 23.75 | 22.25 |
| | Dy ₂ O ₃ | | 25.31 | 27.26 | 25.54 |
| Holmium | Ho | 10 | 9.95 | 12.95 | 9.35 |

| | | | | | |
|----------------------|--------------------------------|--------|---------|---------|---------|
| | Ho ₂ O ₃ | | 11.40 | 14.83 | 10.71 |
| Erbium | Er | 12 | 16.45 | 17.80 | 16.50 |
| | Er ₂ O ₃ | | 18.81 | 20.35 | 18.87 |
| Thulium | Tm | 17 | 0.00 | 3.20 | 0.00 |
| | Tm ₂ O ₃ | | 0.00 | 3.65 | 0.00 |
| Ytterbium | Yb | 14.10 | 22.25 | 26.60 | 19.75 |
| | Yb ₂ O ₃ | | 25.34 | 30.29 | 22.49 |
| Lutetium | Lu | 19 | 0.00 | 6.50 | 0.00 |
| | Lu ₂ O ₃ | | 0.00 | 7.39 | 0.00 |
| Σ Total (ppm) | Σ REEs | 790.50 | 1041.88 | 1135.35 | 954.45 |
| | Σ REOs | | 1263.68 | 1376.46 | 1156.33 |
| Thorium | Th | | 47.08 | 50.69 | 42.33 |
| | ThO ₂ | | 53.58 | 57.68 | 48.17 |
| Uranium | U | | 3.53 | 3.80 | 3.18 |
| | U ₃ O ₇ | | 4.16 | 4.48 | 3.74 |

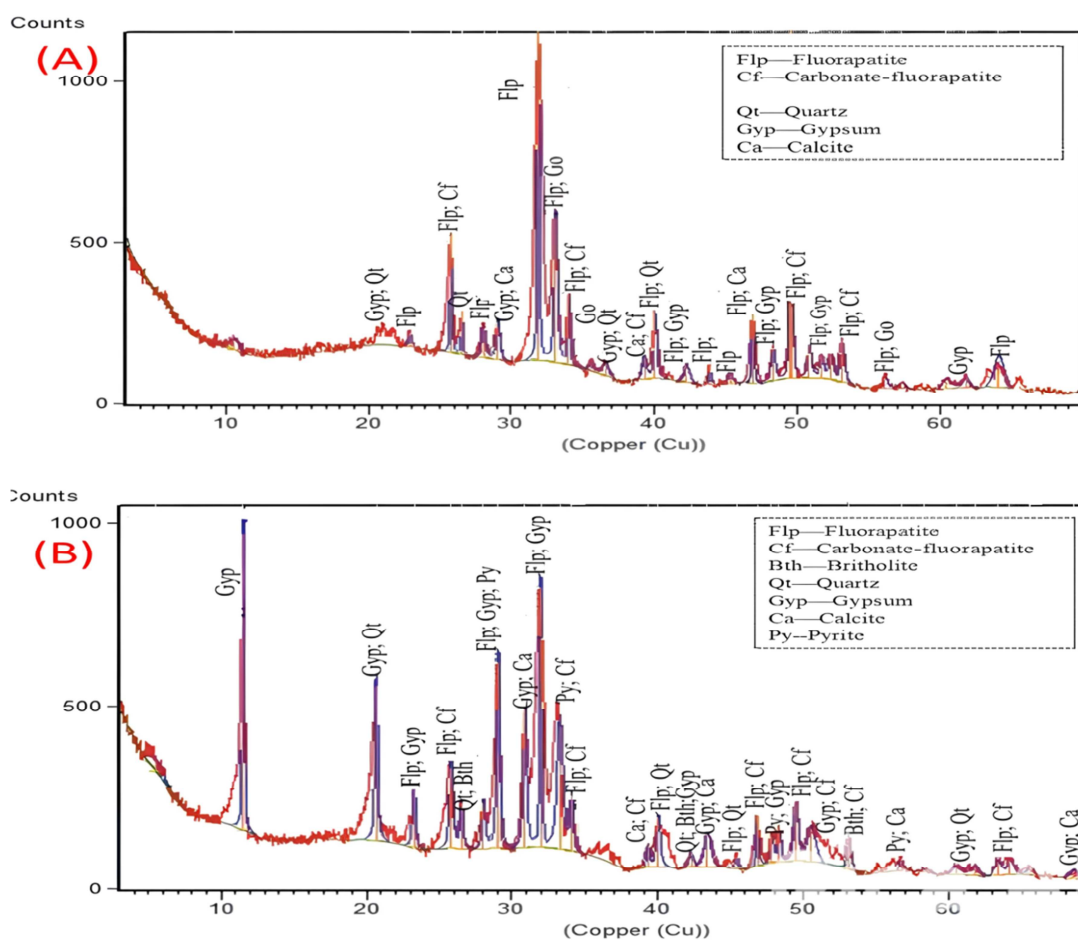


Figure 9: XRD pattern of (a) phosphate ore sample and (b) phosphate concentrate using 2,2-(Octadecyl-azanediyl) di-ethanol.

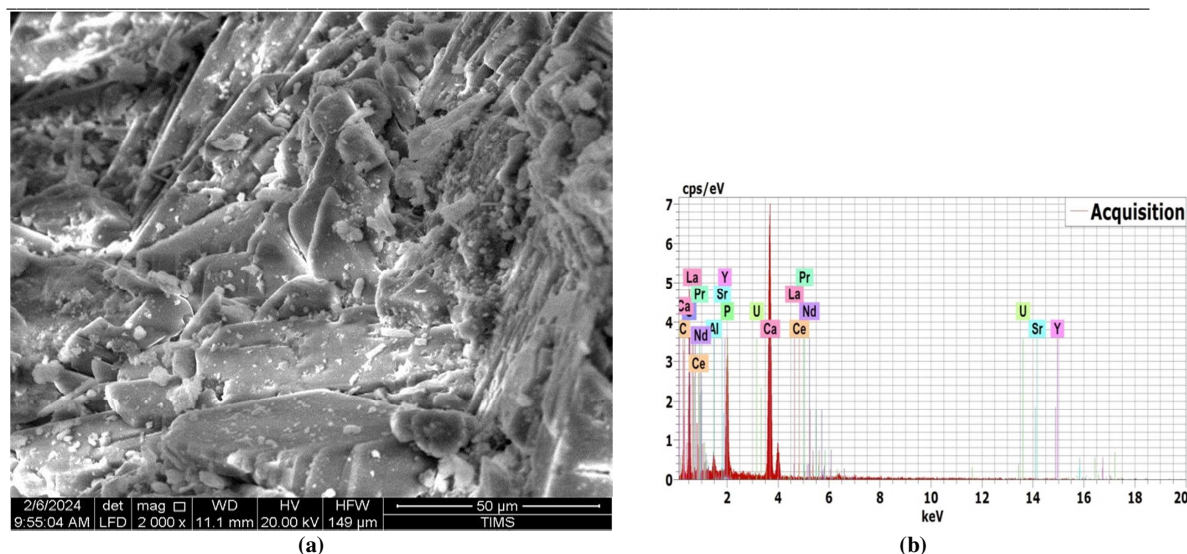


Figure 10 (a,b): SEM-EDX of the phosphate concentrate using 2,2-(Octadecyl-azanediy) di-ethanol.

4. Conclusions

This study sought to enhance the upgrading process of the Abu Tartur phosphate ore sample, of 20.60% P_2O_5 and size fraction -250 +45 μ m. During this study, a direct method for floating phosphate ore sample is conducted using novel collectors including 2, 2-(Octadecyl azanediy) di-ethanol and 2-Oxobutanol compared to oleic acid. Also, this study evaluated the effect of collector and depressant dosages at collector dosage between 2.50 and 3.00 kg/ton, the selectivity for phosphate separation is improved for all studied collectors, resulting in higher P_2O_5 grade and recovery in the float fraction. The sodium silicate values from 2.00 to 2.60 kg/ton were adequate as a depressant to accomplish the best P_2O_5 grade and recovery. Among the studied collectors investigated for mini-column flotation of Abu Tartur phosphate ore sample, 2, 2-(Octadecyl azanediy) di-ethanol emerged as the most effective, yielding the highest quality product with a P_2O_5 grade of 27.80% and recovery of 88.50% from a feed containing 20.60% P_2O_5 . Additionally, the product was enriched with approximately 1135.35 ppm of total rare earth elements (Σ REEs).

5. Conflicts of interest

The authors declare no conflicts of interest regarding this article.

6. Formatting of funding sources

None declared.

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