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DETERMINATION OF PHYSICO-CHEMICAL PARAMETERS OF DJEBEL ONK PHOSPHATE FLOTATION (ALGERIA)

Purpose. To define the optimal physical-chemical parameters for the flotation of Djebel Onk phosphate in Eastern Algeria, for obtaining a concentrate of high quality with minimal dispenses and environment protection and using micro-flotation tests to achieve maximum recovery of the latter.

Methodology. In order to achieve our purposes we used: the thermodynamic analysis method to characterize mineral surfaces and to define equilibrium pH value necessary for a complete phosphate recovery; micro-flotation tests to determine optimal quantity of sodium oleate and alkyl hydroxamic acid collector (AERO 6493) concentrations for a maximum concentrate extraction; Zeta-meter system to measure the Zeta potential of the phosphate electrokinetically to choose the suitable values insuring the best hydrophobicity of the particle surface.

Findings. pH equilibrium value for the “mineral-water” system makes 8.7 for $\text{Ca}_3(\text{PO}_4)_2$. The maximal mineral recovery is obtained, it is about 88.6 %, at Oleate concentration till 30.44 mg/l. However, in the case of AERO6493, it reaches 86 % provided pH value equals 8.7; Zeta potential (surface charge) values -20.45 and -10.87 mV, corresponding to concentration of Sodium Oleate collector 30.44 and 75 mg/l of alkyl hydroxamic acid collector, respectively, answering the minimal necessary concentrations for a maximum apatite flotation at equilibrium pH value of 8.7.

Originality. The originality of this research is in the application of flotation process for the first time on Algerian phosphate ore, performed in optimal conditions (pH, collector concentrations, Zeta potential). This leads to a complete recovery of phosphate concentrate with minimal reagents dispenses and environment protection.

Practical value. Application of the obtained optimal physical-chemical parameters of phosphate flotation permits a using phosphate ores rationally, obtaining a high quality concentrate with poor tailings. All this contributes to the improvement of technical-economical parameters of Djebel Onk plant and environment protection.

Keywords: *phosphate; flotation; pH; reagents; Djebel Onk deposit; thermodynamic analysis method*

Introduction. Phosphorus is a chemical element of atomic number 15 and symbol (P). It is a member of the group of Pnitrogens. The simple phosphorus body comes in many different colours: white-yellow, red and purple-black, transparent. It is a primary constituent of plants and animals' life, this element plays multiple roles in plant metabolism and is one of the essential nutrients required for plant growth and development; consequently, its deficiency restricts crop yield severely [1]. Phosphate rock minerals are the only significant global sources of phosphorus.

Since these nineteenth-century discoveries, phosphate has found widespread use in various fields, including the fertilizer industry and the manufacture of phosphoric acid, which accounts for 80–90 % of global phosphate consumption, and other sectors such as the paint industry, ceramics, cosmetics and pharmaceuticals, the food industry and others.

Algeria is among the world leaders in the production of phosphate. The main deposits are located at Djebel Onk region in the east near the Algerian-Tunisian borders. Currently, in the Jebel Onk complex, phosphate is concentrated by gravimetric classification, generating a large amount of sludge relatively rich in P_2O_5 , with the presence of different heavy metals such as Uranium, Cadmium, Zinc, Copper, and Arsenic which are considered environmentally harmful materials and polluting sources of the air, soil and both surface and groundwater [2].

The analysis of different research showed that the flotation is the most promising method which gives very satisfactory results. It occupies the first place in phosphate beneficiation; around 80 % of the world production is beneficiated by flotation [3].

Actually, flotation presents a kind of instability during separation process. The principal cause of the characteristics variation of minerals extraction at processing factories is the variable and not improved physical-chemical conditions of pulp while floating, due to ore

structure variation, liquid medium and quantities of chemical reagents injected in the pulp when the separation process is conducted [4].

Resolution of this problem can be made with the help of practical experimentation and thermodynamic analysis method. The main idea of this study is based on pH values, determination of the best reagent concentration and thermodynamic and zeta potentials [5].

By using the thermodynamic analysis method to assess the flotation medium we have considered the following equations:

- relationship between the equilibrium constant of reaction (K) and contributed in reactions substances concentrations ($[C]$);

- relationship between the standard free energy of reaction (F_0) and its equilibrium constant (K) or standard electrode potential (E_0);

- relationship between the reaction relationship between the reaction potential (E), its standard electrode potential (E_0) and equilibrium constant (K) [6].

The aim of this modest work is optimization of physical-chemical properties of phosphate by using flotation process, for instance the pH values and necessary Na-Oleate and AERO 6493 concentration for a maximal phosphate recovery. According to the obtained results, the optimal pH value for a maximum extraction of phosphate is 8.7; however, the Na-Oleate collector concentration needed to have the best phosphate separation is 30.44 mg/l.

Materials and Methods. There are several beneficiation techniques for upgrading phosphate ores. The type of ore and the associated gangue minerals defines which and how many techniques are used, knowing that the most of sedimentary phosphate is valorized by flotation [7]. To optimize physical-chemical parameters of the latter, the thermodynamic analysis method and Micro-flotation tests were used .

Micro-flotation tests. The sample was ground in wet-milled to 38 μm . The size fraction $[-106 +38] \mu\text{m}$ was used as a float feed. The flotation tests were performed using alkyl hydroxamic Acid Collector (AERO6493) as a frother and Sodium Oleate ($\text{C}_{18}\text{H}_{33}\text{NaO}_2$) as a collector. As regulators, hydrochloric acid (HCl) and Sodium hydroxide (NaOH) were used.

The flotation tests were carried out in a micro-flotation cell with a volume of 100 ml related to a purified nitrogen gas for bubble formation (Fig. 1). For phosphate flotation we considered the following procedure:

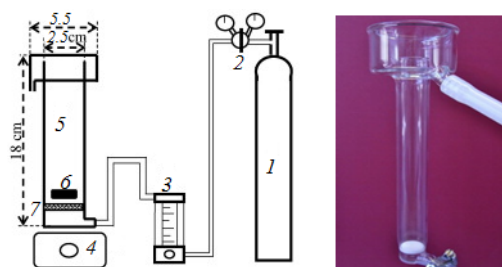


Fig. 1. Micro-flotation cell:

1 – Nitrogen Tank; 2 – Pressure Regulator; 3 – Air Flow-meter; 4 – Magnetic Stirrer; 5 – micro-flotation cell; 6 – Stirrer Bar ; 7 – porous Material

- 1 g of phosphate samples with range size $[-106 +38] \mu\text{m}$;

- condition time is 5 minutes in an agitator, it is sufficient time for the collector adsorption on the phosphate particles surfaces; concentrations of Sodium Oleate collector are [mg/l]: 0.034; 1.52; 3.04; 15.2; 30.44; 46.66; 76.11; 154.22; 228; 33; 258.33; 304.44;

- concentrations of alkyl hydroxamic Acid Collector (AERO 6493) are [mg/l]: 20; 50; 75; 100; 150; 250.

The flotation tests were performed through 4 applications.

First series: in the condition of pH value equal to 8.7 Na-Oleate as a collector with various concentrations from 0.30 to 304.44 mg/l, the aeration and flotation time was 1 minute.

Second series: in the condition of alkyl hydroxamic Acid (AERO 6493) as a collector and a frother at the same time, with varied concentrations from 20–250 mg/l, pH is maintained at 8.7;

Third series: in the condition of different pH values from 2 to 12, adjusted by adding the hydrochloric acid (HCl) and the Sodium hydroxide (NaOH), and the concentration of the Na-Oleate collector is fixed at 30.44 mg/l in all tests.

Forth series: by using different pH values from 2 to 12, adjusted by adding the hydrochloric acid (HCl), and the Sodium hydroxide (NaOH), and the concentration of the alkyl hydroxamic Acid Collector (AERO 6493) is fixed at 75 mg/l in all tests.

For concentrate recovery calculation we used the following equation

$$\text{Recovery; \%} = \frac{C_c}{F_f} \times 100, \quad (1)$$

where F is feed weight in g.; C is concentrate weight in g.; F is phosphate content in feed, %; C is phosphate content in concentrate, %.

Electrokinetic study of phosphate surface. The electrokinetic measurements of phosphate particles surfaces with Sodium Oleate and alkyl hydroxamic Acid Collector (AERO 6493) in different concentrations were conducted by using a Zeta-meter system. After pulp conditioning at pH value equal to 8.7 for 10 min, the latter was allowed to settle for 5 min. The suspended particles were transferred to the Zeta-meter cell where the zeta potential was measured.

Thermodynamic analysis method. In this work, the thermodynamic analysis method is used to compute and characterize the phosphate flotation system. Nowadays, this method is largely applied in the mineral processing domain, especially in the separation by flotation. It also permits us to define the salts compounding composition representing the minerals surfaces, to conduct physico-chemical patterns for the mineral surface interaction processes with the reagents, taking into account the laws of the absorptive collector layer formation, as well as to carry out entire chemical state of liquid phase for ionic components of floating pulp.

The thermodynamic analysis method is based on these equations:

- the equilibrium constant equation

$$K = \frac{[D]^d [C]^c}{[A]^a [B]^b}; \quad (2)$$

- relationship between both free standard energy of reaction and the equilibrium constant

$$\Delta F^0 = \sum \Delta F_{init}^0 - \Delta F_{final}^0 = RT \ln K. \quad (3)$$

Or at 25° C

$$\Delta F_r^0 = -1.364 \log K;$$

- relationship between both free standard energy of reaction and the potential of standard electrode

$$E^0 = \frac{\Delta F^0}{nF} = \frac{\Delta F^0}{23.03n}; \quad (4)$$

- relationship between measured potential, the standard potential and equilibrium constant of reactions

$$E = E^0 + \frac{RT}{nF} \log \frac{[D]^d [C]^c}{[A]^a [B]^b}. \quad (5)$$

In the case of 25° C

$$E = E^0 + \frac{0.059}{n} \log \frac{[D]^d [C]^c}{[A]^a [B]^b}. \quad (6)$$

Different authors [5–8] confirmed the possibility to estimate the surface quality of salt in minerals, relying on



Fig. 2. Zetameter System

thermodynamic calculation. The essence of this method is creating a system of basic reactions of dissociation and hydrolysis of minerals in the joint solution, and resolving the realized equations obtained on the basis of these reactions.

Besides, the elaborated system must be completed by the equilibrium and electro-neutrality equations for their conformity and unknown quantities. Gibbs energies' species and compounds were used in Kcal/mol once calculating the standard free formation energies: According to [8] $Ca_3(PO_4)_2 - 927.70$; $Ca^{2+} - 132.18$; $Ca(OH)_2 - 207.38$; $CaOH^+ - 171.55$; $HCO_3^- - 140.31$; $CO_3^{2-} - 126.22$; $H_2PO_4 - 271.3$; PO_4^{3-} ; $HPO_4^{2-} - 261.5$; $CaHCO_3^+ - 273.673$; $OH^- - 37.595$; $H_2O - 56.69$.

Results and Discussion. Estimation of electrochemical characteristics state of apatite surface. In order to estimate the electrochemical characteristics state of apatite surface, we used the following reactions and equations (Table 1): reaction of dissociation (1–13), the equations of balance and electro-neutrality (I.1, I.2).

Table 1

Reactions and equations of thermodynamic system (K , F^0 , E^0)

N° React & Eq.	Reactions and equations	K	ΔF^0 reaction, Kcal.	E^0 , V
1	$CaCO_3 \leftrightarrow Ca^{2+} + CO_3^{2-}$	$K_1 = 10^{-8.27}$	11.28	
2	$CaOH^+ \leftrightarrow Ca^{2+} + CO_3^{2-}$	$K_2 = 10^{-1.3}$	1.77	
3	$Ca(OH)_2 \leftrightarrow CaOH^+ + OH^-$	$K_3 = 10^{1.29}$	-1.765	
4	$HCO_3^- \leftrightarrow CO_3^{2-} + H^+$	$K_4 = 10^{-10.33}$	14.09	
5	$H_2CO_3 \leftrightarrow HCO_3^- + H^+$	$K_5 = 10^{-6.37}$	8.69	
6	$H_2O \leftrightarrow H^+ + OH^-$	$K_w = 10^{-14}$	19.035	
7	$CaCO_{3aqua} \leftrightarrow Ca^{2+} + CO_3^{2-}$	$K_7 = 10^{-3.29}$	4.50	
8	$CaHCO_3^+ \leftrightarrow 3Ca^{2+} + HCO_3^-$	$K_8 = 10^{-0.865}$	1.18	
9	$Ca_3(PO_4)_{2cryst} \leftrightarrow 3Ca^{2+} + 2PO_4^{3-}$	$K_9 = 10^{-30.02}$	40.96	
10	$Ca_3(PO_4)_{2aqua} \leftrightarrow 3Ca^{2+} + 2PO_4^{3-}$	$K_{10} = 10^{-33.17}$	45.26	
11	$HPO_4^{2-} \leftrightarrow H^+ + 2PO_4^{3-}$	$K_{11} = 10^{-12.02}$	16.4	
12	$H_2PO_4^- \leftrightarrow H^+ + HPO_4^{2-}$	$K_{12} = 10^{-7.18}$	9.8	
13	$H_3PO_4 \leftrightarrow H_2PO_4^{2-} + H^+$	$K_{13} = 10^{-2.13}$	2.9	
I.1	$Ca^{2+} + CaOH^+ + Ca(OH)_2 + 3Ca_3(PO_4)_{2aqua} = CO_3^{2-} + HCO_3^- + H_2CO_3$			
I.2	$2Ca^{2+} + CaOH^+ + H^+ = CO_3^{2-} + 3PO_4^{3-} + 2HPO_4^{2-} + H_2PO_4^-$			

As a result of full chemical computation, pH equilibrium values for apatite mineral were defined (Fig. 3), as well as the regularities of potential determining ions concentrations at various pH values corresponding to them.

The pH equilibrium values for the “mineral-water” system make 8.7 for $\text{Ca}_3(\text{PO}_4)_2$. Thus, the equilibrium condition of the system corresponds to the minimum value of its free energy, so the obtained pH equilibrium value of system can be considered as value of potential of minimal charge of apatite oxidized surface [9].

Evaluation of Sodium Oleate and alkyl hydroxamic Acid collector concentrations and pH value necessary for a complete apatite recovery by using micro-flotation.

Effect of Sodium Oleate collector concentration. Experimental results of apatite micro-flotation in condition of pH value equal to 8.7, Na-Oleate being a collector with various concentrations from 0.304 to 304.44 mg/l are presented in Fig. 4. They show a gradual increase in the particles floatability by increasing the Oleate concentration till 30.44 mg/l, where a maximal mineral recovery is obtained, which is about 85 %. With this concentration, it is observed that the recovery gradually decreases till a minimum value of 40.1 % corresponding to 304.44 mg/l of Sodium Oleate concentration. This decrease in phosphate recovery even provided the increased concentration of the collector, is explained by the creation of collector multilayers which increase the particle

density. Thus, it will be too difficult to transport the latter to the pulp.

Effect of alkyl hydroxamic Acid Collector. The micro-flotation tests are conducted in the condition of alkyl hydroxamic Acid (AERO 6493) being a collector and a frother at the same time, with varied concentrations from 20–250 mg/l while the pH value is maintained at 8.7, which was adjusted by using HCl. Experimental results are illustrated in Fig. 5. They indicate that the phosphate recovery is proportional to the AERO6493 concentration; the particles were mostly floated at 250 mg/l of collector concentration where the recovery reaches 74.14 %.

Influence of pH values on apatite recovery in the presence of Sodium Oleate. During these tests, pH values are varied from 2 to 12 by using HCl and NaOH, and the concentration of the Na-Oleate collector is maintained at 30.44 mg/l in all tests. Results of pH values influence on apatite recovery in those conditions are recapitulated in Fig. 6. They proved that phosphate particles floated completely up to pH 8.7. The recovery increases simultaneously with the increase in pH values up to an optimum value of 8.7; at this value the recovery becomes almost stable – it is about 88.60 %. The complete floatability at pH values equal to 8.7 confirms the important role of the free Ca^+ ions for the phosphate micro-flotation. The free Ca^+ ions interact with Oleate, or both and enhance the micro-flotation. After pH 8.7, the free Ca^+

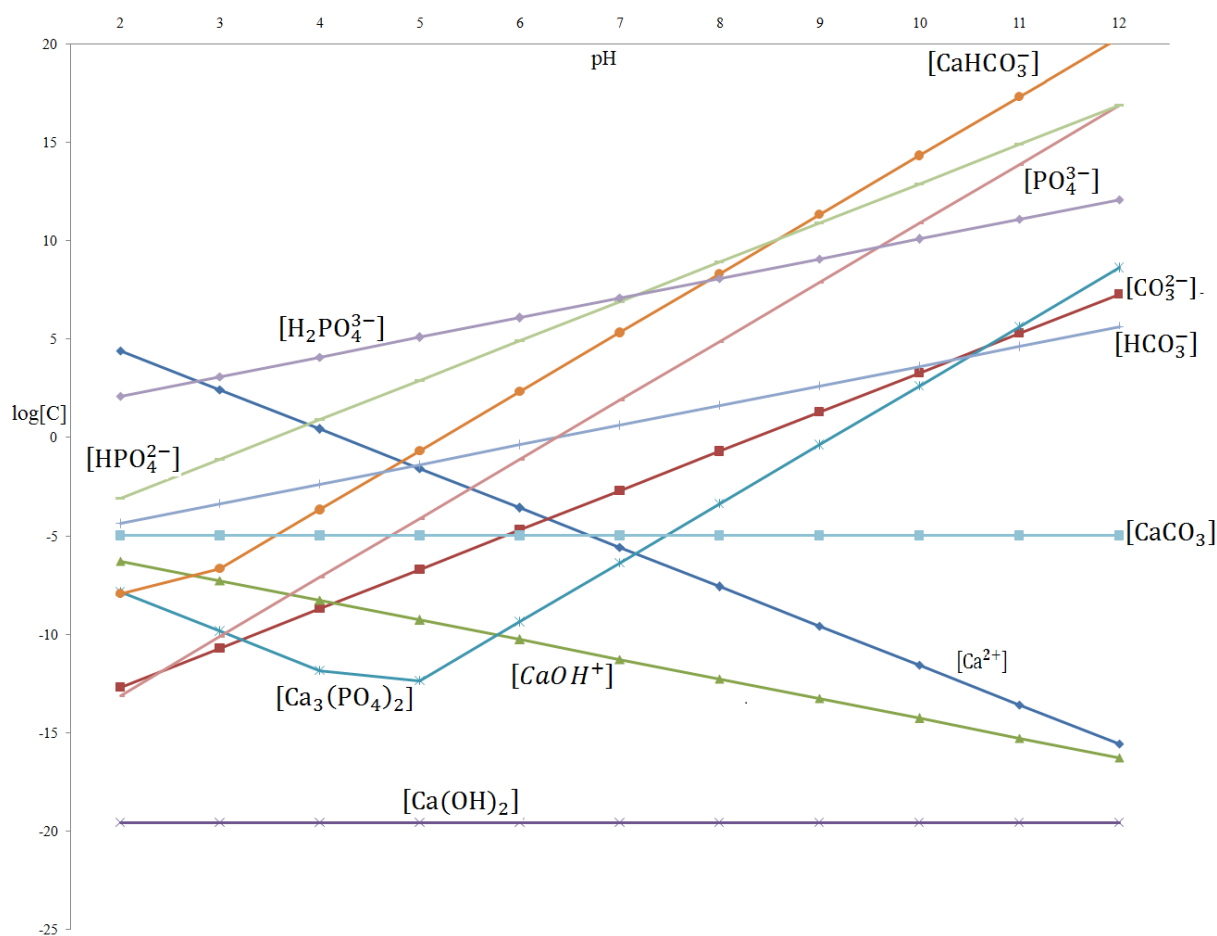


Fig. 3. Effect of pH on the ions concentrations, and equilibrium pH determination in the case of different phosphate oxidation products

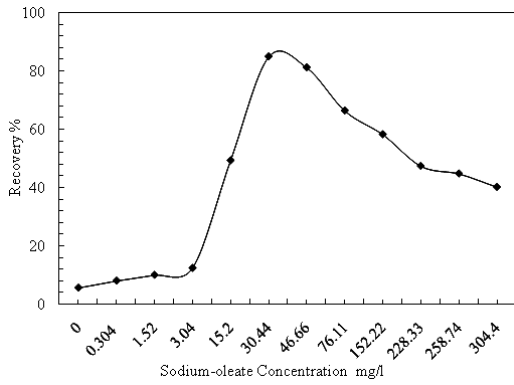


Fig. 4. Effect of Sodium Oleate concentration on flotation recovery

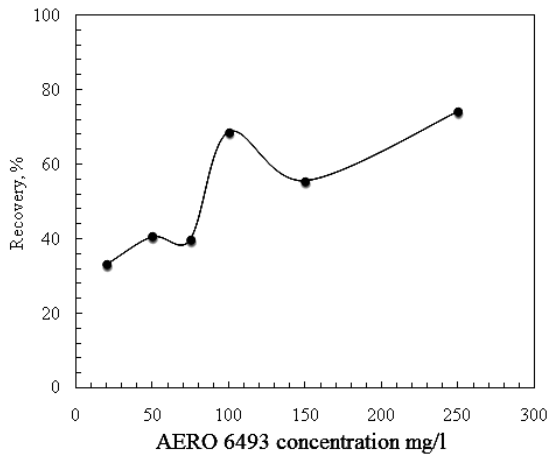


Fig. 5. Effect of AERO 6493 concentration on flotation Recovery

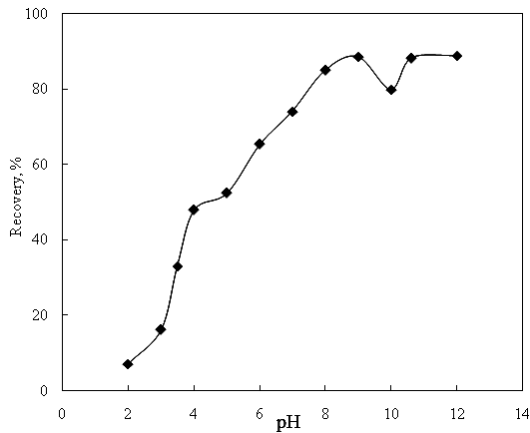


Fig. 6. Effect of pH on phosphate flotation with Sodium Oleate at 30.44 mg/l

ions decrease to the minimum as well as the formation of CaOH^+ and $\text{Ca}(\text{OH})_2$ which increases gradually by increasing the pH values, which explains the lower recoveries at this pH range [10].

Influence of pH values on phosphate recovery in the presence of alkyl Hydroxamic Acid Collector (AERO6493). In order to investigate the influence of pH values and phosphate flotation behavior in the presence of AERO 6493, the pH values were adjusted by adding HCl and

NaOH. Fig. 7 presents experimental results; they show the variation of phosphate particles recovery in the dependence of pH values. It is clear that maximal flotation of mineral particles reaches 86.00 % at pH of about 8.7, which is confirmed by the method of thermodynamic analysis (see chapter 3.1.) and in different bibliographical references [5, 7, 8].

Influence of collectors on Zeta potential values of phosphate surface. The kinetic Zeta potential has an important influence on mineral surface hydrophobicity. So, in order to investigate the effect of this parameter and its behavior on phosphate flotation in the presence of different collectors the zeta potential was measured. Results of Zeta potential measurements are demonstrated in Fig. 8.

They show that the zeta potential (surface charge) value -20.45 mV corresponding to a concentration of Sodium Oleate collector of 30.44 mg/l, meets the requirement of the minimal concentration for a maximum apatite flotation at equilibrium pH value of 8.7. However, the obtained results, conducted with alkyl hydroxamic Acid collector with different concentrations, are presented in Fig. 9.

They justified that surface energy or Zeta potential value corresponding to optimal collector concentration 75 mg/l, insuring a maximal phosphate recovery (Fig. 4) at equilibrium pH value of 8.7, is about -10.87 mV. This value of zeta potential implied that chemical adsorption

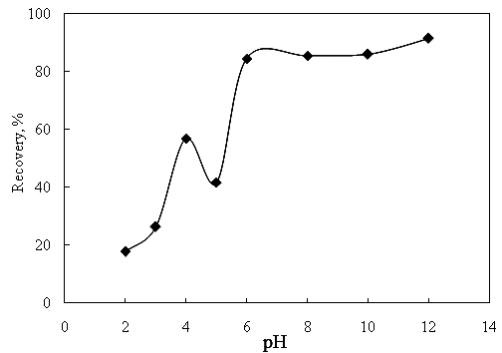


Fig. 7. Effect of pH on phosphate flotation with AERO 6493 at 75 mg/l

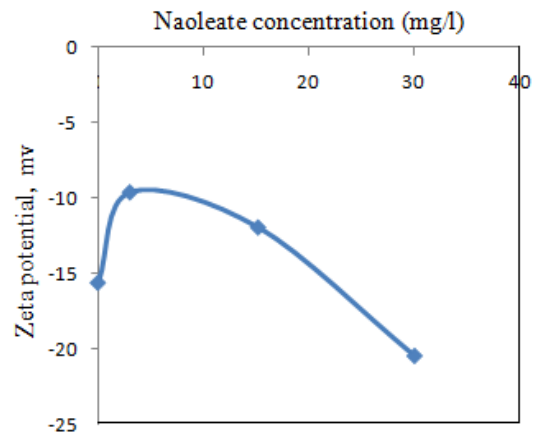


Fig. 8. Results of Zeta potential measurements according to Sodium Oleate concentration

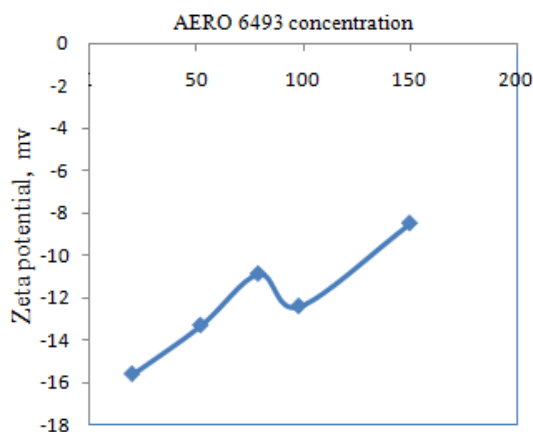


Fig. 9. Results of Zeta potential measurements according to alkyl hydroxamic acid collector concentration

was achieved on apatite surface. Thus, the obtained results testify that at potentials, corresponding to minimal charge on apatite surface, we found optimum structure of the collector sorption layer, consisting of both kinds of sorption; chemically fixed Oleate ions (Ol^-) and physically adsorbed Oleate molecules (Ol_2) [11]. The necessary hydrophobicity of mineral surface and the thermodynamic possibility of its fastening on bubbles are insured by the chemically fixed collector.

The physically adsorbed collector plays the role of kinetic functions at flotation as providing effective destruction of hydrolayers between the bubble and particle, and hardening the bubble-particle contact. As a result, effective flotation could be provided [12].

Conclusions.

The results obtained from full chemical computing of apatite surface by using the thermodynamic analysis method, and those of phosphate micro-flotation tests at different Sodium Oleate and alkyl hydroxamic acid concentrations, allow us to conclude the following:

- pH equilibrium value for the “mineral-water” system makes 8.7 for $Ca_3(PO_4)_2$. Thus, the equilibrium condition of the system corresponds to the minimum value of its free energy, so the obtained pH equilibrium value of the system can be considered as the value of potential of minimal charge of apatite oxidized surface;

- gradual increase in the particle floatability by increasing the Oleate concentration till 30.44 mg/l where a maximal mineral recovery is obtained, is about 85 %. However, in the case of AERO6493, the particles were mostly floated at 250 mg/l of collector concentration where the recovery reaches 74.14 %;

- in the case of Na-Oleate and AERO6493 concentrations of respectively 30.44 and 75mg/l, maximal apatite recovery was found being respectively 88.6 and 86.0 %, at the same value of equilibrium pH 8.7, which was confirmed by thermodynamic analysis method;

- zeta potential (surface charge) values -20.45 mV and -10.87 mV, corresponding respectively to concentration of Sodium Oleate collector of 30.44 and 75 mg/l of alkyl hydroxamic acid collector, corresponded to the minimal necessary concentrations for a maximum apatite flotation at equilibrium pH value of 8.7.

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Визначення фізико-хімічних параметрів флотації фосфатів родовища Джебел Онк (Алжир)

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Мета. Визначити оптимальні фізико-хімічні параметри флотації фосфатів родовища Джебел Онк на сході Алжиру з метою отримання високоякісного концентрату з найменшими втратами та шкодою для навколишнього середовища, використовуючи мікрофлотаційне тестування для його максимального вилучення.

Методика. Для досягнення мети були використані: метод термодинамічного аналізу для охарактеризування мінеральних поверхонь і визначення показника балансу рН, необхідного для повного відновлення фосфатів; мікрофлотаційне тестування для визначення оптимальної кількості необхідної концентрації колектора олеїновокислого натрію та алкіл-гідроксамової кислоти (АЕРО 6493) для максимального вилучення концентрату; система вимірювання Зета-потенціалу фосфату для електрокінетичного вибору потрібного показника, що відповідає гідрофобності поверхні частинок.

Результати. Показник балансу рН для системи „мінерал – вода“ становить 8,7 для $\text{Ca}_3(\text{PO}_4)_2$. Досягається максимальне вилучення мінералів, що становить близько 88,6 %, при концентрації олеата до 30,44 мг/л. Однак у разі застосування АЕРО6493 досягається 86 % за умови показника рН рівного 8,7; Зета-потенціал (поверхневий заряд) становить $-20,45$ і $-10,87$ мВ, відповідаючи концентрації колектора олеїновокислого натрію 30,44 і 75 мг/л для колектора алкіл-гідроксамової кислоти відповідно, що відповідає мінімально необхідній концентрації для максимальної флотації апатитів за показника балансу рН 8,7.

Наукова новизна. Оригінальність даного дослідження полягає в тому, що вперше застосовано процес флотації на фосфатній руді Алжиру в оптимальних умовах (рН, концентрації колекторів, Зета-потенціал). Це призводить до повного вилучення концентрату фосфату з мінімальними втратами та шкодою для навколишнього середовища.

Практична значимість. Застосування отриманих оптимальних фізико-хімічних параметрів флотації фосфату дозволяє раціонально використовувати фосфатні руди, отримати високоякісний концентрат з низькою кількістю відходів. Усе це сприяє покращенню техніко-економічних параметрів заводу Джебел Онк і захисту навколишнього середовища.

Ключові слова: фосфат, флотація, рН, реагент, родовище Джебел Онк, метод термодинамічного аналізу

Определение физико-химических параметров флотации фосфатов месторождения Джебел Онк (Алжир)

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Цель. Определить оптимальные физико-химические параметры флотации фосфатов месторождения Джебел Онк на востоке Алжира с целью получения высококачественного концентрата с наименьшими потерями и вредом для окружающей среды, используя микрофлотационное тестирование для его максимального извлечения.

Методика. Для достижения цели были использованы: метод термодинамического анализа для охарактеризования минеральных поверхностей и определения показателя баланса рН, необходимого для полного восстановления фосфатов; микрофлотационное тестирование для определения оптимального количества необходимой концентрации колектора олеиновокислого натрия и алкил-гидроксамовой кислоты (АЕРО 6493) для максимального извлечения концентрата; система измерения Зета-потенциала фосфата для электрокинетического выбора подходящего показателя, соответствующего гидрофобности поверхности частиц.

Результаты. Показатель балансу рН для системы „минерал – вода“ составляет 8,7 для $\text{Ca}_3(\text{PO}_4)_2$. Достигается максимальное извлечение минералов, составляющее около 88,6 %, при концентрации олеата до 30,44 мг/л. Однако в случае применения АЕРО6493 достигается 86 % при условии показателя рН равного 8,7; Зета-потенциал (поверхностный заряд) составляет $-20,45$ и $-10,87$ мВ, соответствующая концентрации колектора олеиновокислого натрия 30,44 и 75 мг/л для колектора алкил-гидроксамовой кислоты соответственно, что соответствует минимально необходимой концентрации для максимальной флотации апатитов при показателе балансу рН 8,7.

Научная новизна. Оригинальность данного исследования состоит в том, что впервые применен процесс флотации на фосфатной руде Алжира в оптимальных условиях (рН, концентрации колекторов, Зета-потенциал). Это приводит к полному извлечению концентрата фосфата с минимальными потерями и вредом для окружающей среды.

Практическая значимость. Применение полученных оптимальных физико-химических параметров флотации фосфата позволяет рационально использовать фосфатные руды, получить высококачественный концентрат с низким количеством отходов. Все это способствует улучшению технико-экономических параметров завода Джебел Онк и защите окружающей среды.

Ключевые слова: фосфат, флотація, рН, реагент, месторождение Джебел Онк, метод термодинамического анализа

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