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Leaching of Heavy and Radioactive Elements from Iraqi Phosphate Ore.

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Abstract

This research aims to study the influence of different parameters such as types of acid, acids concentration, reaction times, and liquid to solid ratio on the leaching process for phosphate ore. The hydrometallurgical method has been used for leaching phosphate ore after beneficiation with particle size 75-150 μm . The different acids HCl , H_2NO_3 , Acetic acid, and H_2SO_4 , were used with different concentration (0.5, 1, 1.5, 2 and 4 M), solid to liquid ratio (2/100, 4/100, 6/100, 10/100, 20/100), acid concentration ranged (0.5, 1, 1.5, 2 and 4 M), and the temperature range 25, 45, 65 and 85 $^\circ\text{C}$ at a stirring rate of 1200 rpm. XRD, and SEM used for phosphate ore inspection before leaching process. The best results represent by HCl when compared with the other acids, the hydrochloric acid concentration (4 M), solid to liquid ratio (2/100 g/l), temperature (85 $^\circ\text{C}$), stirring speed (1200 rpm) and contact time (120 min.).

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1. INTRODUCTION

Phosphate rock can be considered as a material that occurs in a natural way that could be commercially used, either directly or following processing. The majority of phosphate rock production in the world is used to manufacture phosphate fertilizers and the rest is used in medical and other chemical industries [1]. The phosphate resources in the world can be classified based on their type, within three groups: marine sedimentary, igneous and biogenetic deposits and diverse from country to country and from area to area. Sedimentary marine deposits are the source for the majority of phosphate rock reserves in the world [2]. Phosphate reserves are located in different regions at Al-Anbar Governorate in the western desert of Iraq. These phosphate rock deposits can rank Iraq as the 2nd nation in the world after Morocco with regard to the size of its reserves of

phosphate rock [3, 4]. The phosphate ores are mainly consisting of apatite minerals group in relation to the variable amounts of gangue minerals like clays, silicates, and carbonates (dolomite or calcite). Iraqi phosphate ores, however, are classified as calcareous ore [5]. Phosphate fertilizers contain traces of heavy and radioactive elements which could be accumulated in the soil with repeated application of phosphate fertilizer. Heavy and radioactive could result in unfavorable human and animal effects at elevated levels or at lesser levels in the case when the exposure happens over a prolonged period. For the purpose of preventing soil from element contamination of the phosphate fertilizer, removing such elements from phosphate ore is primordial. There are 2 suggested approaches for eliminating such elements from the phosphate ores: leaching and calcination, the leaching approach is cost-

effective, superior to the calculations, due to high power required [6].

The Rock phosphates consist of dangerous elements including heavy elements and radioactive elements, for example, Cd, Cr, Pb, Mn, Ni, Cu, Fe, U, Th as well as their daughter products, that are thought out to be toxic to the soil, animal and human health [13]. Heavy metals can be considered as major environmental pollutants, the toxicity they cause is an obstacle of growing importance for environmental, evolutionary, ecological and nutritional reasons. Yet, the chemical features regarding heavy elements are the major impacting factors in comparison to their density. Heavy metals consist of lead (Pb), cadmium (Cd), nickel (Ni), cobalt (Co), iron (Fe), zinc (Zn), chromium (Cr), arsenic (As), silver (Ag) and platinum group elements [14]. Heavy metals have the main availability in the soil and aquatic ecosystems and to a quite lesser amount in the atmosphere as vapors or particulate. The toxicity of heavy metals in plants differs with plant species, particular metal, pH, chemical form, concentration, and soil composition, as a lot of heavy metals are throughout to be important for the growth of plants [15]. Various stable and radioactive elements contained in natural phosphates which considered variances in activity concentrations measured for radionuclides with a half-life is high (>one year, Th-230, U-238, Th-232, and Ra-226), that was of environmental concern to the public [16]. In the phosphate rock, the natural Th and U decay-series have been in equilibrium. Throughout the industrial process, the equilibrium was disrupted, the radionuclides migrate to intermediate, final products and by-products, they were distributed between PG (U-series) and phosphoric acid, depending on chemical [17]. Most of the thorium and uranium content in rocks have been reported to redistribute itself into the acid phase, whereas the majority of rare earth, as well as radium, precipitate with phosphogypsum. After that, the phosphoric acid further converted to fertilizers and, in the procedure, transfer all the valuable elements into the product which is therefore lost through land

application. The valuable elements, if left in fertilizers, are considered as heavy metal contaminants and could cause negative environmental effects.

The aim of this study is to find methods to recover trace elements from the phosphate used in fertilizers production from Al-Qaim fertilizers complex in western Iraq. Thus extracting the heavy and radioactive elements from the ores to reduce their content. This allows for the evaluation of the movement of these elements and thus reducing their toxicity. However, recovery of heavy and radiation elements prior to fertilizer production, it presents a huge opportunity to utilizing these strategic metals in technological applications. Recovery of these valuable elements thus is not only a means of maximizing the mineral potential but also a means of environmental stewardship.

2. EXPERIMENTAL

2.1 Materials

The phosphate rock used in the present study was brought from Al-Qaim fertilizers complex for the purpose of this work. All the chemicals used have an analytical grade. Deionized water was used to prepare the solutions. HCl, citric acid, H₂SO₄, and H₂NO₃ have been used as leaching reagents.

2.2 Preparation of phosphate ore sample

The phosphate ore sample of one Kg was crushed to pass through 1 mm sieve opening, following ore homogenization, representative samples have been taken for size distribution analysis. To determine heavy and radioactive elements in phosphate ore, a mechanical separation process is done by taking 1 kg of phosphate sample. Sieving process is carried out by using auto sieve shaker (Impact, ISO 3310-1:2000, UK) with Sieve sizes of (1mm, 710, 500, 355, 250, 150, 106, 75 and 53 μm) opening. The material retained on each size opening fraction is they are weighed. The screening process time was 15 minutes and it is conducted in the laboratory of minerals

processing of the Department of Production Engineering and Metallurgy. X-ray fluorescence (XRF) has been utilized to perform chemical analysis on the samples as shown in table 1. It can be seen also from table (1) chemical composition of particles size (-150+75 μ m) has a higher concentration than other concentrations of heavy and radioactive elements. Therefore, particle size (-150+75 μ m) was selected in the tests.

2.3 Mineralogical Analysis

Qualitative mineralogical analysis regarding representative sample of phosphate ore taken from ore preparation step has been carried out in university of Baghdad/collage of geology/Iraqi-Germany laboratory, mineralogical analysis has been achieved through the use of X-ray diffraction (XRD) type (Shimadzu 7000). X-ray diffraction has been recorded at 40 kV and 30 mA for a Cu-target tube.

2.4 SEM Analysis

The inspection has been carried out at the University of Baghdad / Iraqi- German laboratory. Morphology regarding the phosphate powder has been examined through a scanning electron microscopy model (VEGA 3).

2.5 Atomic absorption spectroscopy

The atomic absorption spectroscopy type (nova AA 350). Was used to determine the dissolved some heavy and radioactive elements () in the solution of the leaching process, at the laboratories of Ibn-Sina state Company.

2.6. Leaching process

The leaching tests have been carried out in 250 mL spherical Pyrex reactor, that has been heated through a temperature-controlled water bath, and glass reflux condenser has been utilized for preventing the evaporation. Ground phosphate ore has been added to a 100 mL agent solution; the pulp has been agitated for a defined time through the use of magnetic stirrer. After the solution filtrated was liquid leaching was analyzed by AAS. The phosphate leaching percentage (L%) has been estimated through the

use of the following. The percentage regarding the elements removed from ore through batch reaction experiments. The % removal of the elements has been estimated depending on the following equation [18]:

$$L \% = [C_f \setminus C_i] \times 100$$

Where C_i can be defined as the initial concentration in mg/kg, and C_f is defined as the final concentration in mg/kg.

In leaching experiments, the role related to parameters like temperature, acid concentration, particle size, leaching time, and stirring speed in heavy and radioactive elements was investigated and the optimized conditions for maximizing heavy and radioactive elements leaching were determined.

3. RESULTS AND DISCUSSION

3.1 Mineralogical Properties

The mineralogical analyses for phosphate were performed using XRD spectra and their results are given in Figure 1. The Figure indicates that fluorapatite ($Ca_5(PO_4)_3F$) is the predominant phosphate mineral, whereas calcite $CaCO_3$ is the main carbonate gangue mineral associating the ore with some dolomite $Ca Mg (CO_3)_2$ mineral too. This could explain the high CaO/P_2O_5 value of the ore, and from same Fig., can be seen, the ratio of the components of the phosphate ore, where the percent of calcite and fluorine apatite have the largest proportion with a few percentages of dolomite and silica impurities.

3.2 Morphological Analysis SEM and EDX

Examination with scanning electron microscopy showed that the studied phosphate samples. The SEM-DEX analysis which was carried out on a phosphate ore sample is displayed in Figure (2). This figure reveals that the major components of phosphate ore are, Phosphate mineral (apatite) is fine grain size 0.1-0.2 mm, having a granular shape and gray color, Carbonate matrix, main calcite. There are also some carbonate grains present as inclusions in apatite mineral. This is

in good agreement with XRD results which showed the presence.

3.3 Effect of Acid Type

The impact of acid type (H_2NO_3 , H_2SO_4 , HCl and $C_6H_8O_7$) on total heavy and radioactive elements leaching from phosphate ore, was studied at parameters were fixed at a leaching time of 30 min., the temperature of 25 °C and solid/liquid (S/L) ratio of 2/100. And stirring speed 300 rpm. The obtained results are presented graphically as a relation between total leached (%L) and acid type in figure (3). For HCl , it is clear that has a bigger effect than others acids this due to, hydrochloric acid is a strong acid and a relatively small molecule, this could be likely to have quicker diffusion rate than certain other acids in leaching process[19]. Add to hydrochloric acid salts are soluble in water. Therefore, after the leaching process, they can be separated from the beneficiated phosphate rock by a simple filtration process. Some elements, such as (V and U), behave differently from other elements; these elements are affected by nitric acid rather than hydrochloric acid. The element (K) is very much affected by hydrochloric acid compared with other acidity. Whoever hydrochloric acid and nitric acid have the greatest effect on the leaching process. Therefore, hydrochloric acid was selected in subsequent tests as a leaching agent.

4.3 Effect of HCl Concentration

The impact of HCl concentration on leaching effectiveness of heavy and radioactive elements from phosphate ore has been implemented at constant values of particle size 150-75 μm , 30 minutes contact time, 2/100 S/L ratio, stirring speed 300 rpm at room temperature. Leaching of heavy and radioactive elements increases using acid concentration ranged (0.5, 1, 1.5, 2 and 4 M). Increasing the acid concentration increases the acid attack on the structure which that results in more broken bonds and forming, and thus more heavy and radioactive elements are leached from the phosphate ore as shown in Figure(4).

The best leaching at 4 M hydrochloric acid concentration. This is possible because of the elevation in viscosity with elevation in the concentration of leached radioactive and heavy elements [20]. or specific elevation in sulfuric acid concentration tends to the accelerator in the leaching process due to more hydrogen ions[21]. Thus, 4 M acid concentration is suggested as ideal for later experiments.

3.5 Effect of solid to liquid ratio

The best ratio for solid/liquid is (2/100 g/ml) from many experiments (2/100, 4/100, 6/100, 10/100, 20/100). The time, temperature, concentration, and stirring rate are constant at 4 M hydrochloric acid of 25°C, 300 rpm, and 30 minutes consequently. Figure (5) shows the association of liquid to solid ratio and leaching of heavy and radioactive elements from phosphate ore. The leaching effectiveness of ore sample increased with the decreasing solid to liquid ratio [22]. The increase in leaching effectiveness is due to diffusion of ore particles within the solution when a liquid to solid ratio was increased. This process helps to prevent aggregations and the ease of mixing the solution with the ore particles, where high solid percentage that caused a condition of a high viscosity which hinders stirring of reactants, thus increasing the reaction rate. Also, increasing the ratio of liquid to steel means increasing the amount of acid, which leads to increased hydrogen ions [21]. Thus, 2/100 g/ml solid to liquid ratio is proposed as optimum for latter experiments.

4.6 Effect of stirring speed

Stirring speed impact on phosphate rocks dissolution through hydrochloric acid has been examined for various stirring speed: 300, 600, 900, 1200 and 1500 rpm. The other parameters have been fixed at 4 M hydrochloric acid concentration, particle size 150-75 μm and reaction time 30 minutes at room temperature. The experimental results specified in figure (6), indicated that leaching of heavy and radioactive elements increases graded at speeds between 300 to 1200 rpm. But, when increasing the speed for

over 1200 rpm, the leaching efficiency starts to decrease. During the leaching process, a heterogeneous reaction that happens at the interface between solid and liquid phase, at the boundary between the 2 phases a diffusion layer is created. When the solid in aqueous phase this layer contains stationary aqueous layer. Diffusion layer could be thinned via vigorous stirring but certainly not be totally removed. Therefore, the efficiency of the leaching is increased with a higher stirring speed. While lower the efficiency of the leaching with higher the stirring speed may be due to the aggregation of the granules due to the collision due to the high speed, which leads to a decrease in the interaction between granules and solution. Thus, stirring speed of 1200 rpm is proposed as best for later experiments.

4.7 Effect of Reaction Time

Within the conditions of hydrochloric acid 4M, liquid/solid ratio 2:100 g/l, reaction temperature 25°C, ore sample particle size 150–75 μm a series of experiments was implemented for examining the impact of reaction time on leaching efficiency of heavy and radioactive elements. The results of the impact of leaching efficiency time on heavy and radioactive elements leaching are represented in figure (7). According to figure (7), it is clear that the increase of leaching time leads to an increase in leaching efficiency [23]. Nature and type regarding phosphate rock feed have a direct impact on the speed of attack of carbonates through leaching acid. Hard phosphate rocks have a tendency to be extra rigid and less porous than soft phosphates. Therefore, additional time is going to be required the acid to penetrate through hard phosphate particles for reaching the inside carbonates [24]. The reason for increasing the leaching efficiency with the increase in leaching time is due to that the increase in time increases the solubility of the solution by contacting with the solids, and giving sufficient time for the diffusion of the solution in the solids, thus increasing the reaction rate.

4.8 Effect of Temperature

The impact of temperature on leaching effectiveness of radioactive and heavy metals has been implemented in the temperature range 25, 45, 65 and 85 °C at a stirring rate of 1200 rpm, for 120 min, 4M concentration acid and 2:100 solid to liquid ratio. The resulting leaching efficiencies are displayed in figure (8) and specify that temperature is considered to be of high importance in leaching of radioactive and heavy elements. Where the leaching efficiency of heavy and radioactive elements increases with increasing temperature in the temperature range studied and the best leaching efficiency at 85 °C. Temperature is of high importance on dissolution kinetics and extraction because of the leaching of radioactive and heavy elements are a process dependent on diffusion. In the case of increasing the reaction temperature, the entropy will be increased, this results in increased ΔG as it is shown in the presented study [25].

This result agrees with [26]. who mentioned that the increased temperature leads to increase the leaching efficiency, where it increased leaching efficiency gradually for REE from phosphate rock with temperatures increasing.

5. CONCLUSION

The HCl acid was the most efficient for removing heavy and radioactive elements from phosphate ore. Factors affect the leaching efficiency as acid concentration, solid to liquid ratio, contact time, string speed and temperature, were investigated at a constant particles size of phosphate powder (75-150 μm). The results presented that on a great ratio of heavy and radioactive elements could be leached at the optimum conditions.

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Table 1 chemical composition of particles size of phosphate sample

symbol	Concentration %					
	As received	75-53 μm	150-75 μm	355-150 μm	710-355 μm	2mm-710 μm
MgO	1.777	1.553	1.932	1.766	1.694	1.585
Al ₂ O ₃	0.586	0.5894	0.4379	0.6469	0.6299	0.5143
SiO ₂	5.097	4.88	4.498	5.018	5.228	4.798
P ₂ O ₃	11.55	9.613	15.02	13.33	11.12	11.2
SO ₃	1.044	0.9132	1.384	1.259	0.9983	0.9138

Cl	0.05721	0.05281	0.06391	0.06471	0.05858	0.05579
K ₂ O	0.0565	0.0609	0.0258	0.0293	0.0685	0.0442
CaO	50.36	49.23	48.93	47.25	50.34	50.13
TiO ₂	0.0971	0.0976	0.0583	0.0791	0.1061	0.0864
V ₂ O ₅	0.059	0.058	0.0489	0.0507	0.0659	0.617
Cr ₂ O ₃	0.0633	0.06363	0.05382	0.05631	0.06493	0.06858
MnO	0.0034	0.00458	0.00272	0.00348	0.00443	0.00354
Fe ₂ O ₃	0.526	0.6152	0.3814	0.4578	0.6105	0.5554
CoO	<0.00039	0.00039	0.00039	0.00039	0.00039	0.00039
NiO	0.01059	0.01205	0.00811	0.00976	0.01273	0.0125
CuO	0.0042	0.00825	0.00479	0.00547	0.00619	0.00523
ZnO	0.138	0.1459	0.1118	0.1425	0.1389	0.1561
Ga	0.00101	0.00076	0.00046	0.00062	0.00069	0.00076
Ge	<0.00005	0.00007	0.0005	0.00005	0.00011	0.00005
As ₂ O ₃	0.00069	0.00071	0.00069	0.00072	0.0007	0.00071
Se	0.00007	0.00009	0.0006	0.00008	0.00008	0.0001
Br	0.00068	0.00056	0.00085	0.00077	0.00071	0.00067
Rb ₂ O	0.00057	0.00067	0.00047	0.00056	0.00059	0.00059
SrO	0.1469	0.1117	0.1916	0.1693	0.1384	0.1376
Y	0.00547	0.00475	0.00729	0.00636	0.00524	0.00529
ZrO ₂	0.00232	0.00392	0.00254	0.00223	0.00298	0.00308
Nb ₂ O ₅	<0.00014	0.00014	0.00014	0.00014	0.00005	0.00014
Mo	0.00175	0.00244	0.00269	0.00205	0.00196	0.00189
Ag	<0.0002	0.0002	0.0002	0.0002	0.00022	0.00023
Cd	0.00863	0.01095	0.00584	0.00663	0.00902	0.00941
SnO ₂	0.00158	0.00121	0.00173	0.00214	0.00156	0.00201
Sb ₂ O ₅	0.00261	0.00195	0.00282	0.00228	0.00185	0.00226
Te	0.00144	0.001	0.00665	0.00146	0.001	0.00094
I	0.00416	0.00264	0.00439	0.00432	0.00385	0.00349
Cs	<0.00040	0.0004	0.0004	0.0004	0.0004	0.0004
Ba	0.00309	0.00544	0.00776	0.00718	0.00747	0.00765
La	<0.00020	0.0002	0.012	0.0002	0.0002	0.0134
Ce	<0.00020	0.0002	0.0101	0.0002	0.0002	0.0002
Er	<0.00051	0.00051	0.00051	0.00051	0.00051	0.00051
Yb	<0.00020	0.0002	0.0002	0.0002	0.0002	0.0002
Hf	<0.00010	0.0001	0.0001	0.0001	0.0001	0.0001
Ta ₂ O ₅	0.00928	0.00828	0.0211	0.00958	0.00951	0.00924

WO3	0.00013	0.00013	0.00013	0.00013	0.00013	0.00013
Hg	<0.00010	0.0001	0.0001	0.0001	0.0001	0.0001
TiO2	<0.00002	0.0001	0.0001	0.0001	0.00002	0.0001
Pbo	0.00061	0.00066	0.00063	0.00054	0.00068	0.00057
Bi	<0.00010	0.0001	0.0001	0.0001	0.0001	0.0001
Th	0.0002	0.00021	0.00026	0.00015	0.00017	0.00018
U	0.00536	0.0046	0.0065	0.0059	0.00517	0.00525

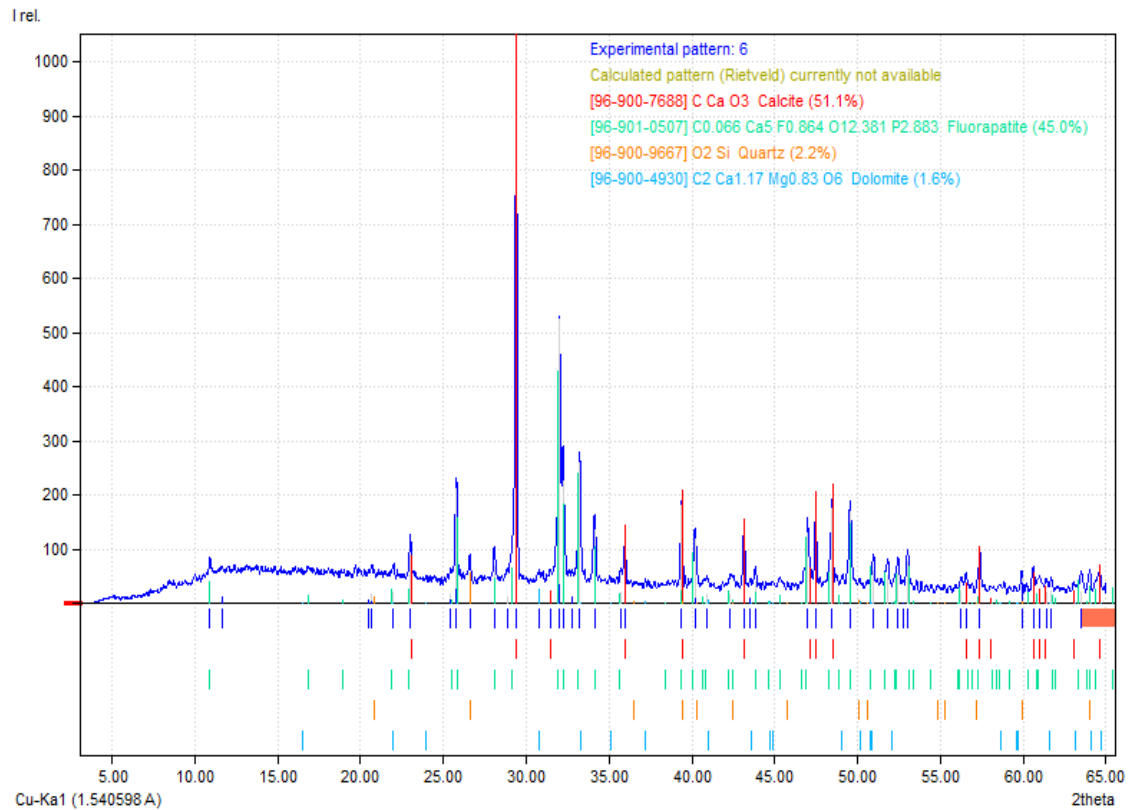


Figure (1): XRD pattern of phosphate ore

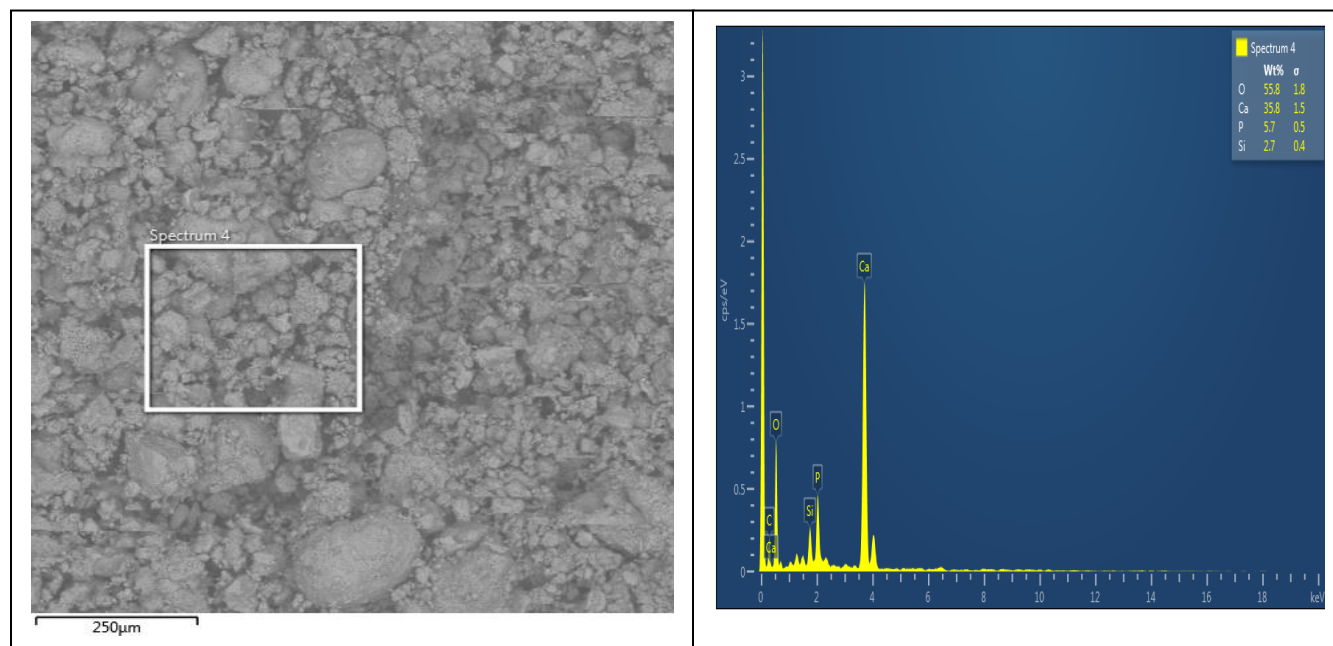


Figure (2): SEM and EDX pattern of phosphate ore

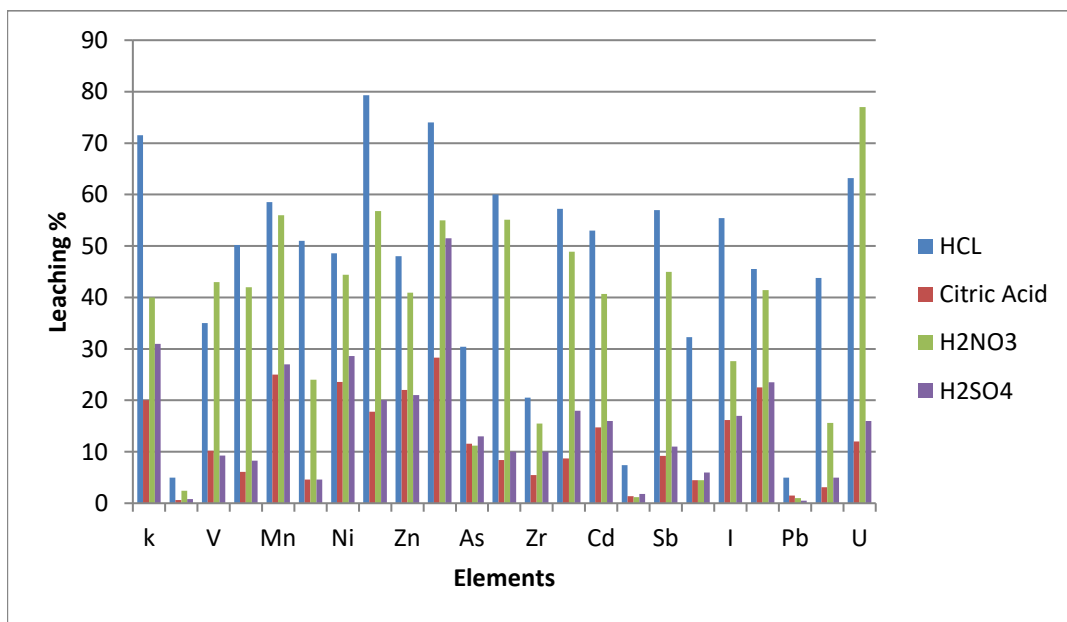


Figure (3): heavy and radiation elements leaching efficiency of different leaching agents (leaching time of 30 min., temperature of 25 °C and liquid/solid(S/L) ratio of 2/100g/l and stirring speed 300 rpm.)

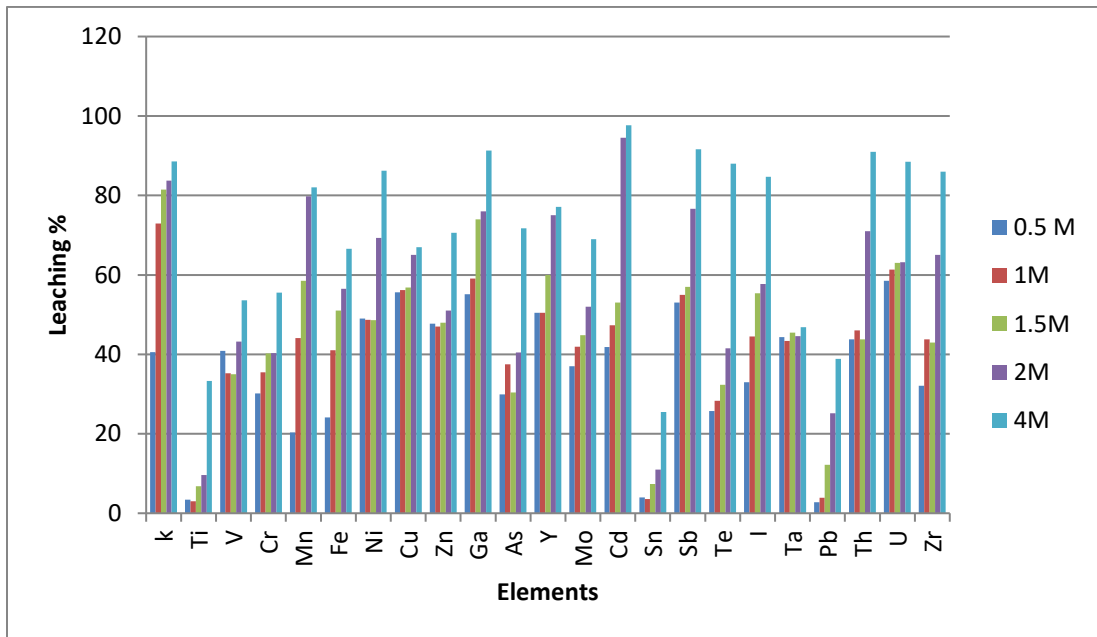


Figure (4):heavy and radiation elements leaching efficiency of different HCL concentration (leaching time of 30 min., temperature of 25 °C and liquid/solid(S/L) ratio of 2/100g/l.and

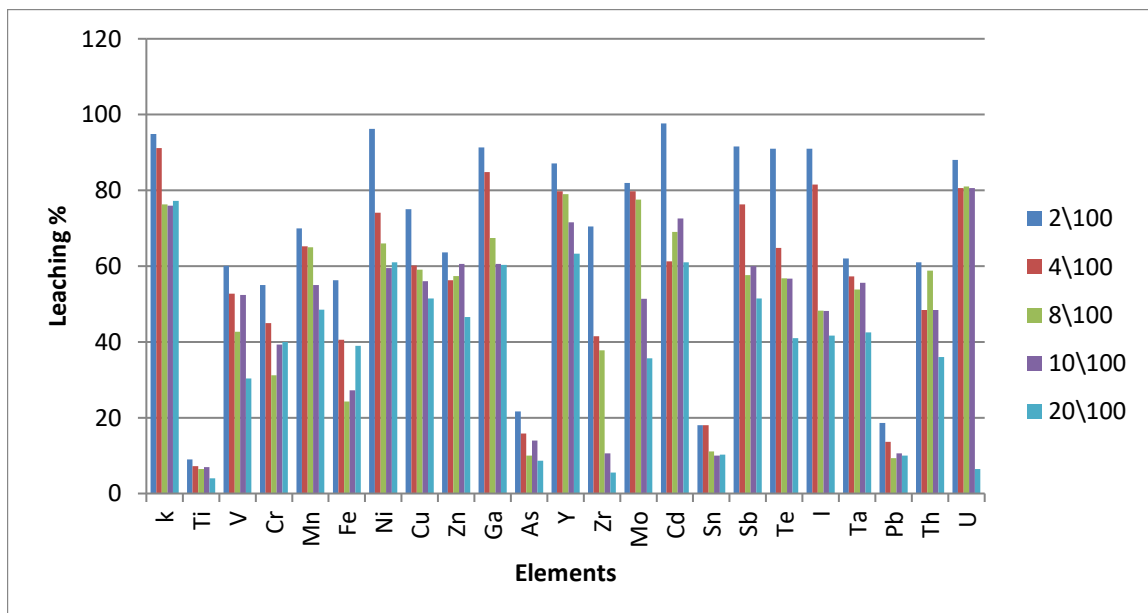


Figure (5):heavy and radiation elements leaching efficiency of sold to liquid ratio (leaching time of 30 min., temperature of 25 °C and liquid/solid(S/L) ratio of

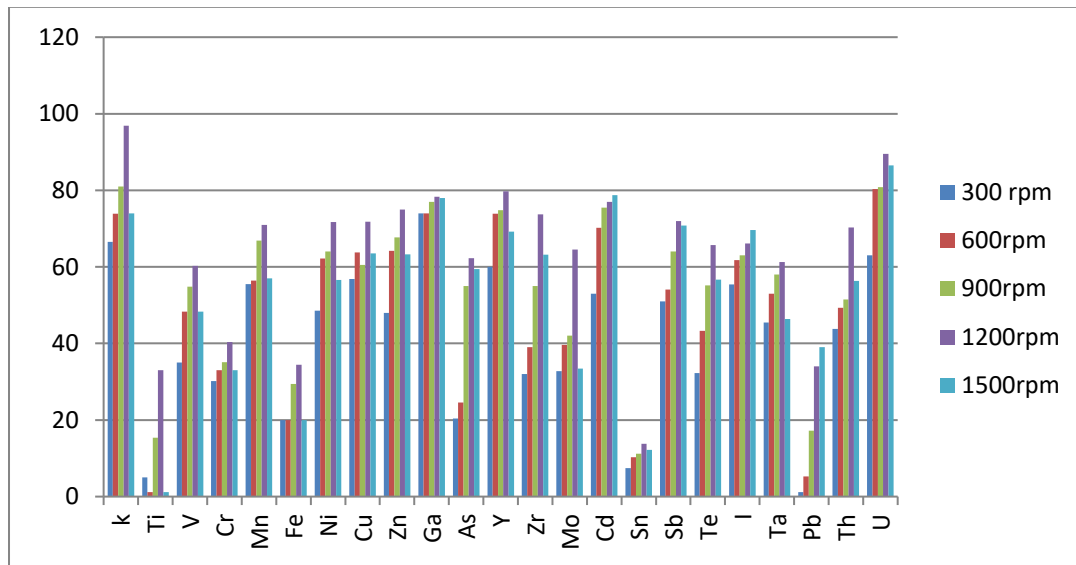


Figure (6):heavy and radiation elements leaching efficiency of sold to liquid ratio (leaching time of 30 min., temperature of 25 °C and liquid/solid(S/L) ratio of 2/100g/l.and

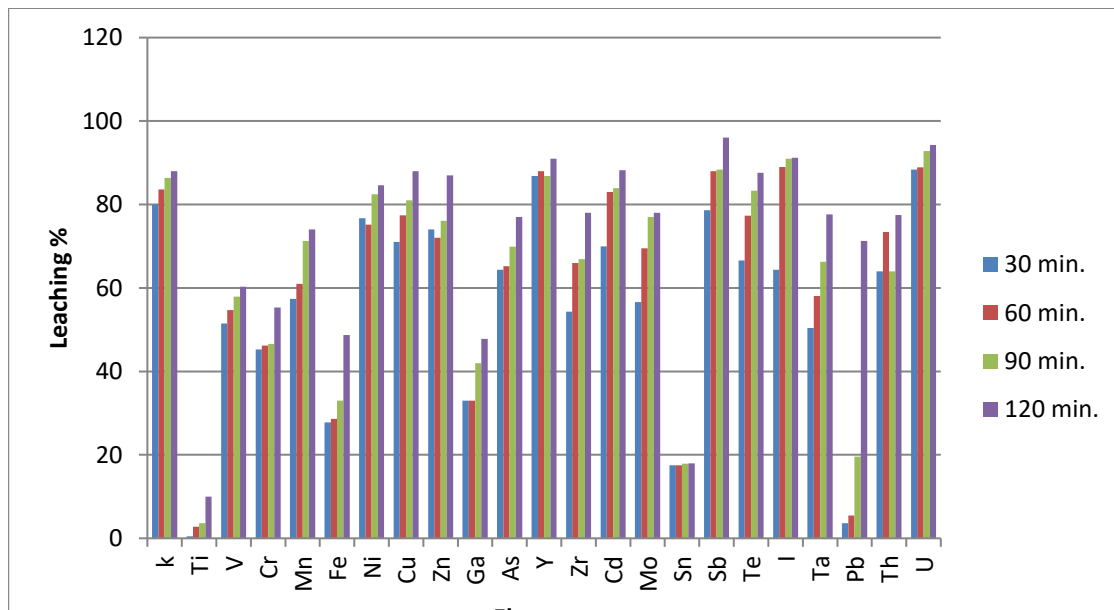


Figure (7):heavy and radiation elements leaching efficiency of time contact (stirring speed 1200 rpm, temperature of 25 °C , liquid/solid(S/L) ratio of 2/100 g/l and concentration acid

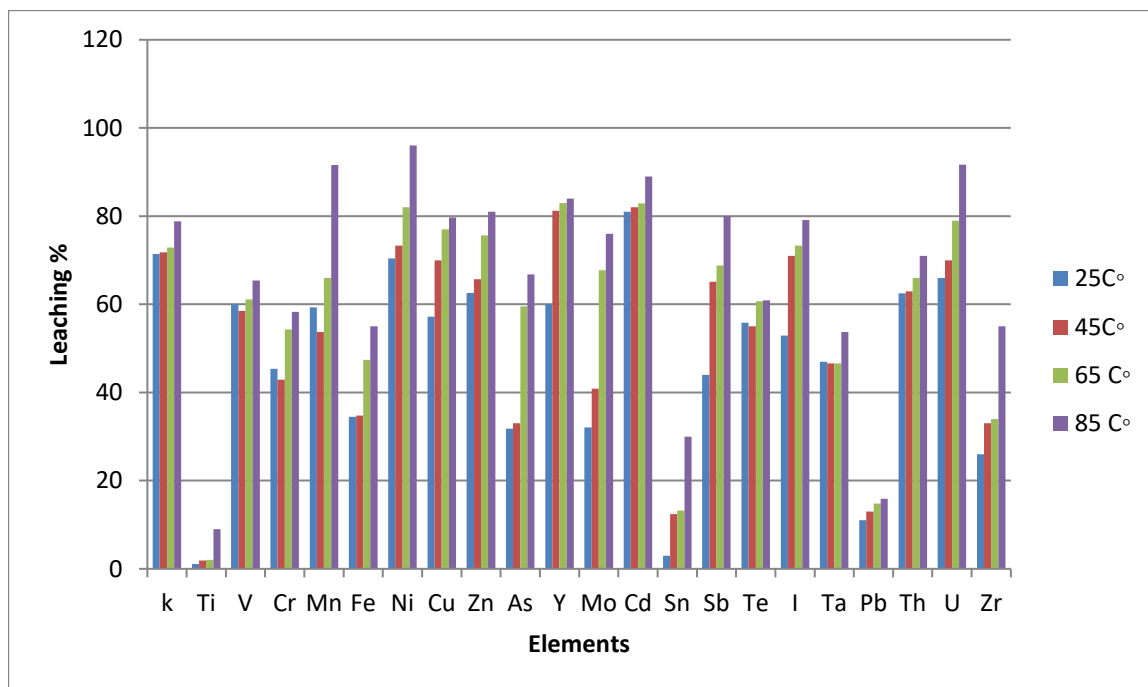


Figure (8):heavy and radiation elements leaching efficiency of temperature (stirring speed 1200 rpm. time contact 120 min . liquid/solid(S\L) ratio of 2/100 a/l and concentration acid