



A COMPARATIVE STUDY BETWEEN CALCINATION AND LEACHING OF CALCAREOUS PHOSPHATE ORE

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ABSTRACT

Vast tonnages of low-grade calcareous phosphate rocks are found in several areas around the world. These rocks consist mainly of carbonate-apatite and free calcium carbonate with small amounts of silica. Commercial phosphate rock should not contain more than 8% carbonates (about 3.5% CO₂) in order to be economical for fertilizer industry.

In Egypt, the low calcareous sedimentary phosphate deposits are widely distributed in three main localities, namely, the Eastern desert, the Nile valley and the Western desert. Beneficiation of these ores by calcination, flotation and high intensity magnetic separation was tried by some investigators.

Consequently, the aim of this research is to make a comparison between calcination and leaching of calcareous phosphate ore. For this purpose, the major parameters which effect on calcination and leaching processes are studied. Among them are, particle size, heating temperature, duration time and quantity of ore treated with respect to the calcination process, and particle size, acid concentration, solid percent (W/V) and leaching time with respect to the leaching process. Loss on ignition is used for assessment the results in the two methods.

Calcination process is still used efficiently for removing free calcium carbonates. Therefore, this study was carried out on low-grade calcareous phosphate ore of Um-Hammad area (Red Sea phosphate) to reduce its calcium carbonates to an acceptable limit. To optimize the process, the influence of the most affecting parameters on the calcined samples was studied. A concentrate of 82.15% weight recovery and 2.98% L.O.I. was obtained under the optimum conditions.

Leaching using dilute acetic acid was applied on the same ore to increase its P₂O₅ content and to reduce its calcium carbonates content to the acceptable limit. To optimize the process, The influence of the most affecting parameters on the digested samples was studied. The selected optimum conditions of these parameters produced a phosphate concentrate having 5.01% L.O.I. with 65.6% weight recovery from a leached feed having 15.38% L.O.I.

From this comparative study it can be concluded that the calcination process is more reasonable than the leaching process for the beneficiation of the low calcareous phosphate ore of Um-Hammad area.

Key words: Beneficiation of phosphate ore - Leaching of calcareous phosphate ore –Calcination of phosphate ore.

1. Introduction

Phosphate plays a significant economic role in developing countries because of the increasing demand on phosphate rock for fertilizer production and its importance in animal feed stocks, as well as food-grade phosphates and other industrial uses [1]. It is estimated that approximately 60% of the phosphorus applied to crop land comes from this non-renewable resource, and around 90% of the phosphorus derived from phosphate rock is used in agriculture as a fertilizer or feed [2]. Several studies suggested that phosphate rock reserves could be depleted within 50-100 years [1-2].

There are five major types of phosphate resources in the world [3-4]. They are sedimentary marine phosphate deposit (75%), igneous phosphate deposits, metamorphic deposits and deposits as a result of weathering (15-20%) and biogenetic deposits (2-3%). The marketable phosphate grade usually contains 30% P_2O_5 or more. The run of mine ore can be classified according to its P_2O_5 content into high grade of 28-32%, medium grade of 22-27% and poor grade of 15-22% P_2O_5 [5-6].

Methods and techniques of varying sophistication are used in the processing of the ore, depending on its grade, its type and the quantity of gangue material associated with the phosphate minerals. The majority of techniques, however, depend on physical methods of separation. In some cases other methods of treatment, such as calcination are necessary [7-9]. Calcination is the process of heating the ore to a high temperature ranging between 800 °C and 1000 °C to decompose $CaCO_3$ to CaO and CO_2 . The formed CaO is then removed by washing and a higher grade phosphate product is obtained. This method is liable for ores contain calcite and/or dolomite as the major impurities with small amounts of silica. It is usually difficult to remove the carbonate minerals efficiently from such ores by conventional physical separation techniques. Separation by physical means becomes impossible also when the carbonate minerals are finely disseminated into the phosphate particles [2].

With sedimentary phosphate ore, the separation of phosphate from carbonate gangue using flotation is extremely complex due to their similarities in physico-chemical and surface chemistry properties [10].

However, calcination process is one of the best known processes, which is still used around the world until now; this process is accompanied by the high energy needed drawback [11].

Siliceous ores, those contain quartz and different forms of silica could be upgraded economically by such techniques as flotation or gravity separation methods [12-20]. The amenability of low-grade phosphate ore to flotation for separation of both calcareous siliceous ganged minerals by just PH control was investigated by Tawfik et al [13]. Phosphate concentrate assaying 30.54% P_2O_5 , 8.7% L.O.I. with P_2O_5 recovery of 64.34% was obtained [13].

Nowadays, the bio-flotation and bio-flocculation have been appeared as new techniques. These techniques change the physicochemical properties of the mineral surface due to its response to the bacterium presence [21-22].

Beneficiation of the Egyptian low grade calcareous sedimentary phosphate deposits was tried by many investigators [7-9, 12-19] using calcination, flotation and high intensity magnetic separation. Unfortunately literatures about leaching this type of ores with dilute organic acid are rare [23-28].

Therefore, the present investigation is devoted to study the amenability of upgrading the low grade calcareous phosphate ore of Um-Hammad area (Red Sea Phosphate) with dilute acetic acid as well as calcination process. Making a comparison between the results of the two methods is also intended.

2. Experimental work

2.1. Materials and methods

A sample of low grade calcareous phosphate ore from Um-Hammad area in the North East of Gabal Duwy mine (Red Sea Company) is used in this work. This area is not exploited until now, the chemical analysis of the head sample before beneficiation showed 15.38% L.O.I.

The sample was stage crushed using a laboratory jaw crusher with a set adjusted at 10 cm. the product was fed to a roll crusher with an opening of 5 cm. the roll crushed head sample (-5 cm) was thoroughly mixed, quartered and riffled to obtain samples of weight 50 gm each. Screen analysis of a representative head sample was carried out with vibrating shaker machine. The results of the screen analysis and the loss on ignition of each size fraction of the head sample are given in Table (1). From this table it is clear that the carbonates are slightly changed from one size fraction to another.

This result means that -5 cm or finer size can be used, but the desired commercially phosphate size should be in -2500 μm with 90% -1000 μm sizes.

Table 1.

Screen analysis and L.O.I. for the fractions of the head sample.

Particle size(μm)	wt.% retained	Cumulative wt.% retained	Cumulative wt.% passed	L.O.I. %
-5000+3150	38.98	38.98	61.02	14.78
-3150+2500	34.70	73.68	26.32	15.48
-2500+1600	10.90	84.58	15.42	15.71
-1600+1000	3.10	87.68	12.32	14.66
-1000+0	12.32	100	0	16.89
mean	100			15.38

2.2. Calcination procedures

To carry out the calcination process, batches of 50 gm weight each in porcelain crucibles of 150 cc capacity were put inside the muffle furnace at the identified temperature, time and rate of heating according to the value of these parameters listed in Table (2).

The studied parameters affecting the calcination process included:

- 1- Particle size of the raw material, μm ,
- 2- Temperature of calcination, $^{\circ}\text{C}$,
- 3- Duration time of calcination, minute,
- 4- Ore quantity (bed thickness).

Table 2.
Sequence of Calcination tests

Test No.	Particle size (µm)	Calcination temp, c	Calcination time, (min.)	Weight of bed (W/V)%	L.O.I
1	-1000	900	42	50	3.78
2	-1600	900	42	50	3.95
3	-2500	900	42	50	3.50
4	-3150	900	42	50	4.50
5	-5000	900	42	50	4.73
6	-2500	700	42	50	12.73
7	-2500	750	42	50	10.02
8	-2500	800	42	50	6.39
9	-2500	850	42	50	5.06
10	-2500	900	42	50	3.99
11	-2500	900	30	50	4.18
12	-2500	900	36	50	3.67
13	-2500	900	42	50	3.40
14	-2500	900	60	50	3.27
15	-2500	900	78	50	2.76
16	-2500	900	60	30	2.75
17	-2500	900	60	40	2.79
18	-2500	900	60	50	2.98
19	-2500	900	60	60	3.21
20	-2500	900	60	70	3.67

The calcined ore is quenched in water to convert the formed oxides into hydroxides, until the liquid becomes clear. Extensive trials of calcination and washing processes were carried out to reach the optimum value of each parameter.

2.3. Leaching procedures

The dissolution tests were carried out in a glass reactor of 2000 ml capacity. The pulp was stirred with a variable speed mechanical stirrer fitted with a stainless steel impeller.

At the beginning of each run a known weight represents 10% solid by weight (50 gm) of the phosphate sample was added to a known volume and concentration of dilute acetic acid solution according to Table (3).

Dilute acetic acid has been found to be one of the most promising leaching agents. For this purpose the reaction between acetic acid and calcium carbonates can be written as follows [10, 27]:

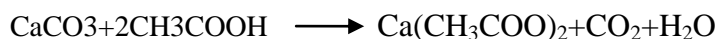


Table 3.
Sequence of leaching tests

Test No.	Acid conc. %	Particle size, μm	Duration time, min.	S% (W/V)	L.O.I. %
1	4	-2000	30	10	9.92
2	6	-2000	30	10	9.39
3	8	-2000	30	10	8.84
4	10	-2000	30	10	8.66
5	12	-2000	30	10	8.55
6	8	-2000	30	10	8.29
7	8	-1600	30	10	7.70
8	8	-1250	30	10	7.36
9	8	-1000	30	10	6.26
10	8	-500	30	10	6.60
11	8	-1000	10	10	7.96
12	8	-1000	20	10	7.50
13	8	-1000	30	10	6.34
14	8	-1000	40	10	6.50
15	8	-1000	50	10	6.55
16	8	-1000	30	5	5.01
17	8	-1000	30	10	7.08
18	8	-1000	30	15	7.91
19	8	-1000	30	20	8.24
20	8	-1000	30	25	8.84

The reaction has been conducted in a continuous stirred reactor vessel, for a specified time at room temperature. After the completion of each leaching run, the content of the reaction vessel was then filtered and washed many times with tap water to remove any soluble matter.

The solid leached residues were dried and subjected to chemical analysis to find its loss on ignition (LOI) percent. Loss on ignition is used as a quick assessment of leaching process which is considered as a good indicator of the amount of the remained carbonates in the sample.

The solid product from the leaching reactor has been first washed and then dried at about 105°C in the drying furnace to get rid of free moisture. The difference in weight between the original samples is mainly due to loss of carbonates from the sample as CO_2 gas and acetate salts.

Combined water and organic matter is usually represented by the loss in weight of the leaching sample after being dried from 150°C to 550°C for two hours. The amount of combined water and organic matter is included in loss on ignition.

The loss on ignition (LOI) is defined as the decrease in weight of the leached sample when it is ignited from 105°C to 1000°C . It refers to the amount of carbonates, organic matter and combined water that originally exists in the sample, or has not been eliminated during leaching.

3. Results and discussions

3.1. Calcination results

3.1.1. Effect of particle size on calcination process

The first series of tests was carried out to find out the best size of particle for calcination. Samples weighting 50 gm each were taken from each size fraction and

calcined at 900 °c for 30 minutes, then, quenched in 1.25 liter of tap water immediately after calcination. The results are listed in Table (4) and graphically represented in Fig (1).

Table 4.

Values of L.O.I. % & Loss in carbonates % at different calcined particle sizes.

Test no.	Particle size	L.o.I. %	Loss in carbonates %
1	-1000	3.78	75.43*
2	-1600	3.95	74.32
3	-2500	4.1	73.34
4	-3150	4.5	70.74
5	-5000	4.73	69.25
Head sample		15.38	

$$*\frac{15.38-3.78}{15.38} \times 100 = 75.43$$

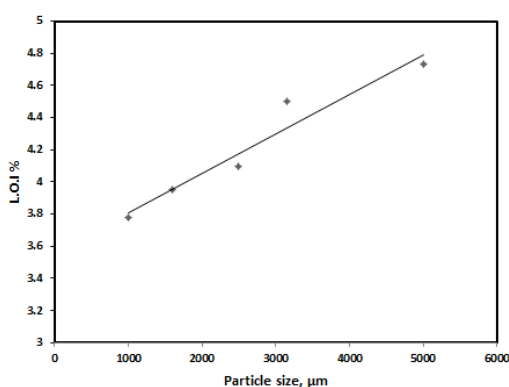


Fig. 1. Effect of particle size on calcination process.

From the results of Table (4) and Fig. (1) it is clear that the calcinations of the different studied sizes has a little effect on the loss on ignition percentage, where increasing the particle size from 1000 μm to 5000 μm increased the L.O.I. from 3.78 to 4.73%. Therefore, it is not necessary to grind the ore to finer sizes to decrease the grindability costs. Consequently, -2500 μm size is used at studying the other parameters.

3.1.2. Effect of calcination temperature

A series of tests was done at varied temperatures ranged from 700°C to 900°C with an interval of 50°C to achieve the optimum temperature of calcination.

Results are listed in Table (5) and represented graphically in Fig.(2). These results indicate that 900°C is more convenient for changing most of the carbonates into oxides, thus giving approximately the best grade of the calcined phosphate.

Table 5.

Values of L.O.I. % & loss in carbonates % at different temp.

Test no.	Temp. °c	L.O.I %	Loss in carbonates %
6	700	12.73	17.23
7	750	10.02	34.85
8	800	6.39	58.45
9	850	5.06	67.10
10	900	3.99	74.06
Head sample		15.38	

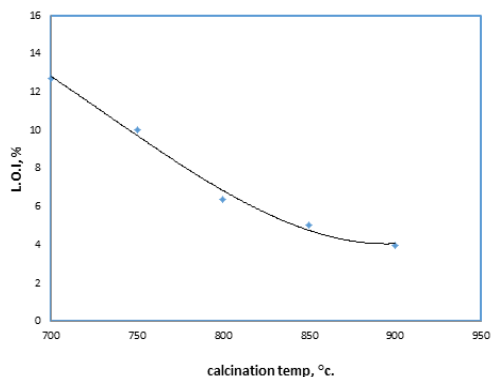


Fig. 2. Effect of temperature on calcination process

3.1.3. Effect of duration time

A series of tests was carried out to find the optimum time for calcination, results of these experiments are listed in Table (6) and represented graphically in Fig.(3). From these results it is clear that the optimum time of calcination is 60 minutes, where this time achieves a suitable percentage of L.O.I. accompanied with an acceptable loss in carbonates. Longer than this time consumes more heating energy.

Table 6.

Values of L.O.I. % & Loss in carbonates % at different calcination times.

Test no.	Time, min.	L.O.I %	Loss in carbonates %
11	30	4.18	72.82
12	36	3.70	79.94
13	42	3.42	77.76
14	60	3.20	79.19
15	78	2.80	81.79
Head sample		15.38	

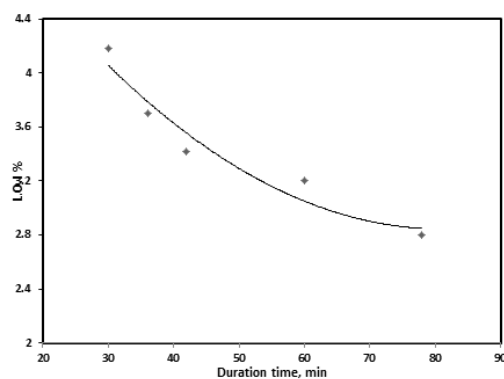


Fig. 3. Effect of duration time on calcination process.

3.1.4. Effect of bed weight (bed thickness)

A series of tests were carried out to find the optimum quantity of ore for calcination at a temperature of 900°C for a duration time of 60 minutes. The calcined samples were immediately quenched with 1.25 L of water. The results are shown in Table (7) and represented graphically in Fig. (4).

Table 7.
Effect of bed weight on calcination process.

Test no.	(w/v), %.	L.O.I %	Loss in carbonates %
16	30	2.75	82.12
17	40	2.79	81.86
18	50	2.98	80.63
19	60	3.21	79.13
20	70	3.67	76.14
Head sample		15.38	

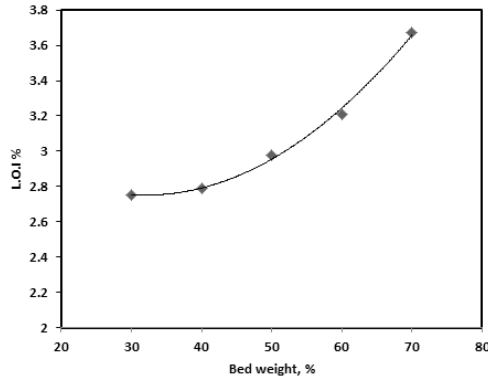


Fig. 4. Effect of bed weight on calcination process.

From the above mentioned results, it is clear that L.O.I increases as the quantity of the ore increases. From Fig. (4) it is clear that the best value of the ore quantity is 50% (w/v).

From the aforesaid calcination results, the optimum values of the studied parameters which achieved the best values of L.O.I % can be summarized in Table (8).

Table 8.
Optimum values of the studied parameters of the calcination process.

Parameter	Optimum value
Particle size	-2.5 mm
Temperature	900°C
Duration time	60 mins
Bed weight	50% (w/v)
L.O.I approximately	2.98%
Wt. recovery	82.15 %

Figures. (5) and (6) show the x-ray diffraction analyses for the head sample and calcined calcareous phosphate of UM-Hammad ore. From these figures, it can be seen that calcite is the highest peak in the head sample, while phosphate minerals represent the high peaks in the calcined ore.

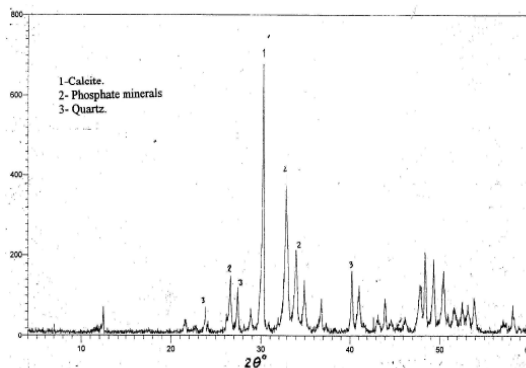


Fig 5. x-ray diffraction of calcareous phosphate head sample.

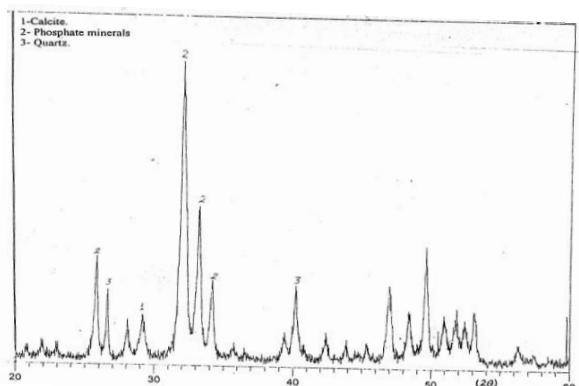


Fig. 6. x-ray diffraction of calcareous phosphate final product after calcination.

4. Leaching results

4.1. Effect of acid concentration on leaching process

The influence of acid concentration on the dissolution of carbonates was investigated for acid concentration of 4,6,8,10 and 12%. The results are tabulated in Table (9) and represented graphically in Fig.(7). According to these results the dissolution of carbonates increases as the acid concentration increases up to 8%, above this value the increase in acidity of the leaching solution has slightly effect on L.O.I%. Therefore on the intermediate acid concentration of 8% which gave 8.84 L.O.I.% at weight recovery of 76.92 % was adopted for the subsequent experiments.

Table 9.

Effect of acid conc. on leaching process.

Test no.	Acid conc.%	L.O.I %	Loss in carbonates %
1	4	9.92	35.5
2	6	9.39	38.95
3	8	8.84	42.52
4	10	8.66	43.70
5	12	8.55	44.41
Head sample		15.38	

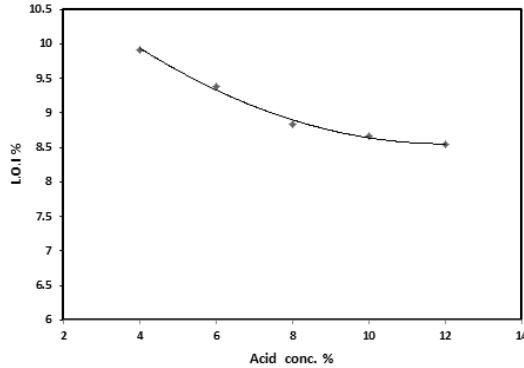


Fig. 7. Effect of acid conc. on leaching process.

4.2. Effect of particle size

The effect of particle size on leaching process was investigated using five representative samples of the ore crushed and ground to ϕ -2, -1.6, -1.25, -1 and -0.5mm. From the results given in Table (10) and Fig.(8), it is clear that the L.O.I% decreases with decreasing the leached particle size, because the smaller the size the greater is the interfacial area between the solid and acid. The sample of -1 mm size which has 6.62 % L.O.I. % was found to be sufficient for performing the all subsequent experiments, as the fine grinding of phosphate are tends to cause problems related to handling, filtration, marketing, and lastly to environmental pollution.

Table 10.

Effect of particle size on leaching process.

Test no.	Particle size, μm	L.O.I. %	Loss in carbonates %
6	-2000	8.29	46.09
7	-1600	7.70	49.93
8	-1250	7.1	52.15
9	-1000	6.62	56.96
10	-500	6.60	57.08
Head sample		15.38	

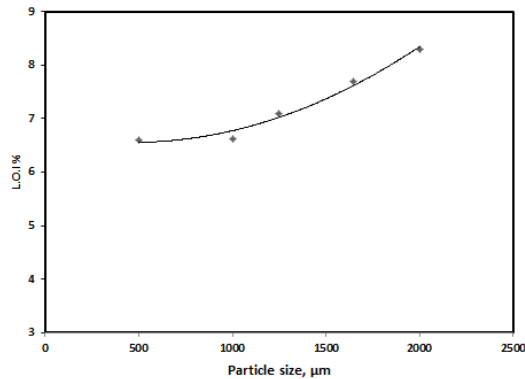


Fig. 8. Effect of particle size on leaching process.

4.3. Effect of leaching time

The effect of leaching time on the leaching process was investigated for duration times of 10,20,30,40 and 50 minutes. The results are given in Table (11) and represented in Fig.

(9). These results show that the best leaching result is obtained at 30 minutes, which gave a product of 6.34 L.O.I at 71.46 % weight recovery.

Table 11.

Effect of time on leaching process.

Test no.	Time, min.	L.O.I %	Loss in carbonates %
11	10	7.96	48.24
12	20	7.5	51.24
13	30	6.34	58.78
14	40	6.40	57.74
15	50	6.30	57.41
Head sample		15.38	

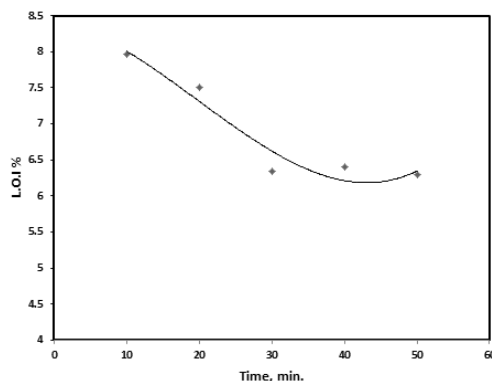


Fig. 9. Effect of leaching time on leaching process.

4.4. Effect of solids percent

The effect of solid percent by weight on the dissolution of carbonates from calcareous phosphate ore was studied at percents of 5,10,15,20 and 25 % (w/v).The L.O.I. contents of the samples after digestion are shown in Table (12) and Fig.(10). From these results it is clear that the percent of 5% is sufficient to give phosphate product of 5.01 % L.O.I. with 65.6 % weight recovery.

Table 12.

Effect of solid percent on leaching process.

Test no.	Solid %	Wt. rec. %	L.O.I %	Loss in carbonates %
16	5	65.6	5.01	67.43
17	10	72.6	7.08	53.97
18	15	75.27	7.91	48.67
19	20	76.30	8.24	46.16
20	25	76.92	8.84	42.53
Head sample			15.38	

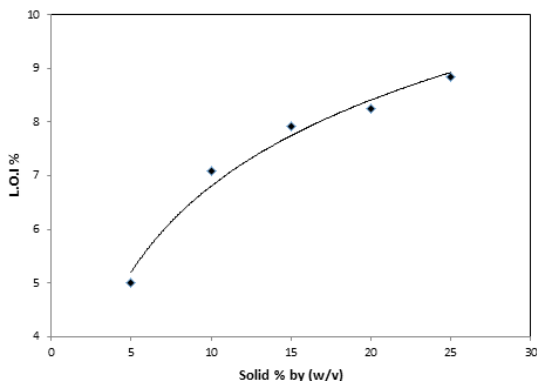


Fig. 10. Effect of solid % on leaching process.

From the previous results, the optimum values of the studied parameters which achieved the best value of L.O.I. % are given in Table (13).

Table 13.

Optimum values of the studied parameters of the leaching process.

Parameter	Optimum value
Particle size	-1.0 mm
Acid concentration	8%
Duration time	30 min
Solid % (w/v)	5 %
L.O.I.	5.01 %
Wt. recovery	65.60 %

Fig. (11) shows the X-ray diffraction analysis for the leached calcareous phosphate of Um- Hammad area. Comparing Figs.(6) and (11) of X-ray diffraction for the calcined and leached calcareous phosphate ores respectively clears that calcination process is more reasonable than leaching process.

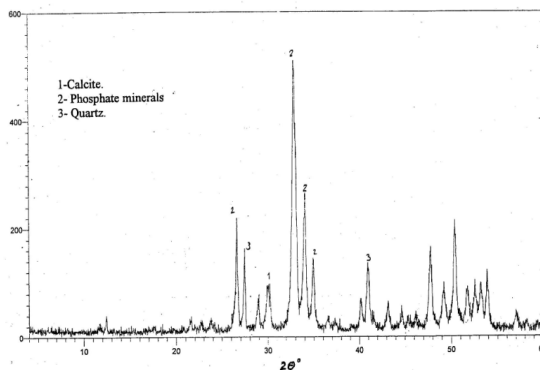


Fig. 10. Effect of solid % on leaching process.

5. Conclusions

From the obtained results and their discussions the following conclusions can be drawn:

- Calcination process gives satisfactory results, and hence it is recommended for the beneficiation of calcareous phosphate ore of Um-Hammad area. On the other hand the results show a good tendency to leaching process using dilute acetic acid.

- The optimum values of the most affecting studied parameters are obtained for calcination and leaching processes as listed in the following table:

Studied parameters	Calcination	Leaching
Acid concentration (%)	—	8
Particle size (mm)	-2.5	-1.00
Duration time (min.)	60	30
S% (W/V)	50	5
Temperature (c°)	900	Room temp.
Quenching media	Tap water	Water is used

- The above mentioned optimum values of the studied parameters gave calcined and leached phosphate with the following characteristics:

Product characteristics	Calcination	Leaching	Feed
L.O.I. %	2.98	5.01	15.38
Wt.% recovery	82.15	65.6	100

- Calcination process results are more reasonable than those of leaching ones; on the other hand it is costly from the energy consumption point of view.
- It is recommended to use a cheap source of energy such as natural gas to be cost efficiently, as it is the better way to remove free calcium carbonates from calcareous phosphate ores.

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دراسة مقارنة بين كلسنة واذابة خام الفوسفات الكالسي

الملخص العربي:

توجد كميات هائلة من صخور الفوسفات الكالسي منخفضة الرتبة في أماكن عديدة من العالم. تتكون هذه الصخور أساساً من كربونات الأباتيت وكربونات الكالسيوم الحر، ولكي يتم تسويقه يجب ألا يحتوي على أكثر من 8% كربونات كالسيوم حتى يكون صالحاً لصناعة السماد.

تتوزع خامات الفوسفات الرسوبي الكالسي في مصر عبر الحزام الفوسفاتي الممتد من الصحراء الشرقية (ساحل البحر الأحمر) مروراً بوادي النيل (ادفو والسباعية) إلى الصحراء الغربية (أبو طرطور).

وقد أجريت محاولات عديدة لمعالجة هذه الخامات لرفع رتبته باستخدام تقنيات مختلفة منها التعويم والفصل المغناطيسي عالي الشدة. وما زالت عملية الكلسنة تستخدم حتى الآن في أنحاء متفرقة من العالم كطريقة فعالة لإزالة كربونات الكالسيوم من الخام. وعليه، أجرى هذا البحث بهدف عمل دراسة مقارنة بين نتائج تطبيق عمليتي الكلسنة والإذابة بحمض الخليك المخفف على عينات من الفوسفات الكالسي منخفضة الرتبة من منطقة أم حماد على ساحل البحر الأحمر التي لم تستغل حتى الآن. وقد استخدم فاقد الحرق (L.O.I) الذي يعبر عن نسبة الكربونات المتبقية في العينة كتحليل سريع لتقييم كل من عمليتي الكلسنة والإذابة.

ل للوصول إلى الناتج الأمثل لعملية الكلسنة تم دراسة أهم العوامل المؤثرة عليها، والتي شملت حجم خام التغذية ودرجة حرارة العملية وزمن مكوث العينة بالفرن وكمية الخام. وقد تم الحصول على ناتج أمثل مكلسناً يحتوي على 2,98% كربونات بعائد وزني 82,15% مقارنة بالعينة الأصلية والتي تحتوي على 15,38% كربونات. وبتطبيق عملية الإذابة بحمض الخليك المخفف وللوصول إلى الناتج الأمثل تم دراسة حجم الخام وتركيز الحامض ونسبة الصلب في المحلول وزمن الإذابة. وقد تم التوصل للحالة المثلى لعملية الإذابة على مقياس معملّي والتي أنتجت خام يحتوي على 5,01% كربونات كالسيوم بعائد وزني 65,6% لنفس العينة الأصلية.

من هذه النتائج تعتبر عملية الكلسنة أكفاً من عملية الإذابة في التخلص من كربونات الكالسيوم في خامات الفوسفات الكالسي منخفضة الرتبة. وعليه يوصى باستخدام أي مصدر رخيص للطاقة في عملية الكلسنة حيث أنها الأكفاً.