



Upgrading of Phosphate Ore Wastes of El-Nasr Mining Company, Egypt

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Abstract: Phosphates are vital nonrenewable resources. The treatment of phosphate ore wastes of El-Nasr Mining Company, Sepaeya, Egypt aims to upgrade P₂O₅ percent, adds a new economic value, development of the Egyptian national income, and aids to clean the mine district. The main aim of this paper is to compare different processing alternatives methods to concentrate and upgrade of phosphate ore tailings assayed 18.37% of El-Nasr Mining Company, Sepaeya, Egypt. The concentration of wastes not only add an economic value but also safe the human, animals, and plants. Two options of sizes obtained from grinding and sieving were separated to obtain the highest grade of P₂O₅ with the greatest recovery. The first option (i.e. sizes below 100 μm) gave P₂O₅ percent of 23.88%, P₂O₅ recovery of 42.37%, and weight recovery equals to 33.68%. The second option (i.e. sizes below 160 μm) gave P₂O₅ percent of 22.52%, P₂O₅ recovery of 53.25%, and weight recovery equals to 44.88%. The calcination process raised P₂O₅ to 22.4 with weight recovery 83.75%, and P₂O₅ recovery of 99.10%. Leaching process using acetic acid gave the best results at the following conditions: a) solid to liquid ratio 15% where the percent of P₂O₅ raised from 18% to 22.23% with weight recovery about 86%, b) Agitation time 15 minutes where the percent of P₂O₅ raised from 18% to 22.35% with weight recovery about 86%, c) Agitation speed 1200 rpm where the percent of P₂O₅ increased from 18% to 22.23% with weight recovery about 86%, d) Acid to ore ratio 16.7% where the percent of P₂O₅ increased from 18% to 21.44% with weight recovery about 88.7%. The leaching process on size -100 μm raised P₂O₅% from 23.88% to 28.44% with weight recovery 84%. The weight recovery and P₂O₅ recovery with respect to the original feed were 28.29% and 42.39%, respectively.

Keywords: Phosphate, Wastes, Crushing, Grinding, Sieving, Calcination, Leaching

1. Introduction

The annual consumption of phosphates approached 150 million tons. Phosphates are vital nonrenewable resources [1]. They are essential components in agricultural fertilizers and phosphorous-based chemicals [2]. They are also very important constituents in animal feed stocks. About 95% of the phosphate produced around the world is consumed in the fertilizer industry [3-4].

Phosphate ores in terms of quality and P₂O₅ grade can be divided into three groups: low grade ores (12–16% P₂O₅), intermediate-grade ores (17–25% P₂O₅), and high-grade ores (26–35% P₂O₅) [5].

Physical as well as thermal treatment techniques are used for concentrating the run-of-mine phosphates. The processing techniques of phosphate ores depend mostly on the type of

associated gangue minerals present in the mined rock [6]. The most common gangue minerals in phosphate ores are quartz, chert, clay, feldspar, mica, calcite, and dolomite [7].

In some cases, simple, inexpensive techniques are enough to produce the required grade. For example, crushing and screening is used to get rid of the coarse hard siliceous material, and attrition scrubbing and desliming is used to remove the clayey fine fraction.

If silica is the main gangue material, single-stage or double-stage flotation is the conventional mineral processing technique used in this case. The calcareous types of phosphate ores are extremely difficult to concentrate via flotation due to the similar physicochemical surfaces characteristics of the main constituents, carbonates and phosphates [6, 8-9].

In Egypt phosphate reserves are present in three localities namely; the eastern desert, Nile valley, and the new valley in western desert. Currently, phosphate ores are being mined from Nile valley areas. The mining companies export the fractions of high grade (over 30% P₂O₅) while the fractions of low grade is being rejected. This is because such fractions contain different gangue minerals such as calcite and silica.

This paper aimed to compare different processing alternatives methods to concentrate and upgrade of phosphate ore tailings assayed 18.37% of El-Nasr Mining Company, Sepaeya, Egypt. The concentration of wastes not only add an economic value but also safe the human, animals, and plants.

2. Petrographical and Mineralogical Study of the Samples

Thin and polished sections of phosphate ore wastes of Sepaeya mines were prepared for petrographical and mineralogical investigation. The study revealed that these wastes are presented by phosphatic rock (phosphorite).

Microscopically, the phosphate ore wastes of Sepaeya mines are moderately hard, compact and of creamy color with pale yellow and brown spots.

Microscopically, in thin section, it is shown that the rock is fine to coarse grained and composed mainly of cellophane & carbonate minerals as essential constituents associated with minor amounts of iron oxides and quartz. Cellophane (represent about 40% of total minerals constituents) occurs as fine to coarse-grained in the form of rounded to sub-rounded brownish pellets, fish teeth, scales, bones, fossil shells and fragments. Cellophane embedded in very fine-grained matrix of carbonate minerals and sometimes stained and surrounded by iron oxides. The matrix of the sample is composed of very

fine-grained calcite (micrite) and/or dolomite and sometimes stained by traces of iron oxides in some parts. Some fossil shells and fragments are present in the sample as elongated and curved filaments scattered in the carbonate matrix. Quartz presents as a very fine to fine-grained, surrounded to sub-angular in shape and scattered in the very fine-grained matrix of carbonates. Iron oxides are present in minor amounts in the rock.

The mineralogical study of the polished section revealed that the opaque minerals are represented by minor amounts of hematite and goethite. Hematite is essential opaque mineral occurs as very fine to fine grained aggregates, anhedral to subhedral in form. It also presents as irregular patches, thin films lining the essential minerals and also as a staining mineral over some grains cellophane and carbonates. Hematite sometimes replaces the internal structure of the biogenic material. But goethite presents as very fine to fine grained anhedral crystals in rare amounts; it is formed as alteration product after hematite where relics of hematite are still preserved in goethite.

3. Experimental

3.1. Sample Preparation

- i- The head sample was mixed thoroughly and divided into 8 similar parts; each part represents about 5 kg.
- ii- The first part which having P₂O₅ percent about 18.37% was sieved onscreen of 5 mm size. The oversize and undersize were chemically analyzed for P₂O₅ percent. The results are shown in Table 1 and Figure 1. From these results it can be seen that the percent of P₂O₅ in oversize was about 18.34%, while in undersize was 21.76%.

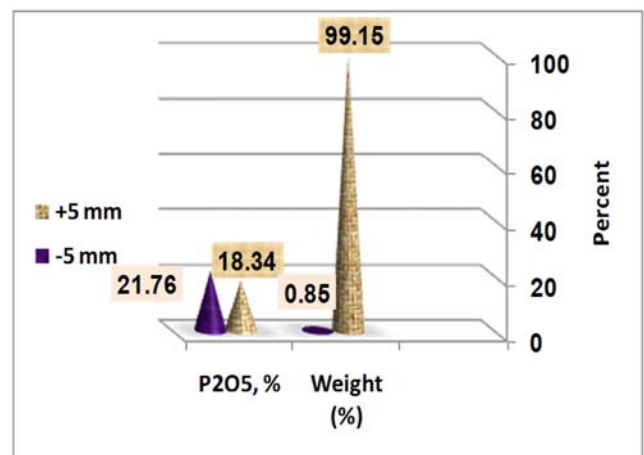


Figure 1. Results of sieving original sample on screen of size 5mm.

Table 1. Results of sieving the original sample on screen of size 5mm.

Screen size, mm	Weight, g	Weight, %	P ₂ O ₅ , %
+5	5007	99.15	18.34
-5	43	0.85	21.76
Head Sample			18.37

3.2. Procedure

In this paper, different alternative processes were carried out to concentrate and upgrade of phosphate ore tailings as follows:

i. The oversize resulted from sieving the original sample was comminuted according to the flowsheet shown in Figure 2. Two representative samples of undersize -5 mm were subjected to sieving and each size fraction was chemically analyzed. Results are shown in Table 2.

ii. A representative sample of crushed ore -5 mm was ground using hammer crusher in closed circuit with screen 1 mm. The ground sample (-1 mm) was sieve analyzed and each size fraction was chemically analyzed.

iii. The ground sample (-1 mm) was subjected to calcination at 740°C for one hour. The sample obtained from calcination was quenched with water in conditioning tank and agitated for 15 min. after which it was filtrated and dried at 105°C for 24 hours and then chemically analyzed for P₂O₅.

iv. A representative sample of crushed ore -5 mm was ground using hammer crusher in closed circuit with screen 1 mm. The ground sample (-1 mm) was ground again in disc mill until -200 μm. The product was de-slimed with screen 38 μm. The leaching experiments were carried out on fraction (-250+38) μm which having P₂O₅ percent of 18%. The tests were executed at different variables, i.e. solid to liquid ratio, leaching time, agitation speed, and acid to ore ratio.

v. Different sizes resulted from grinding process (-250 μm and -100 μm) were leached at acid to ore ratio 10%, leaching time 15 minutes, agitation speed 1200 rpm, and solid to

liquid ratio of 15%.

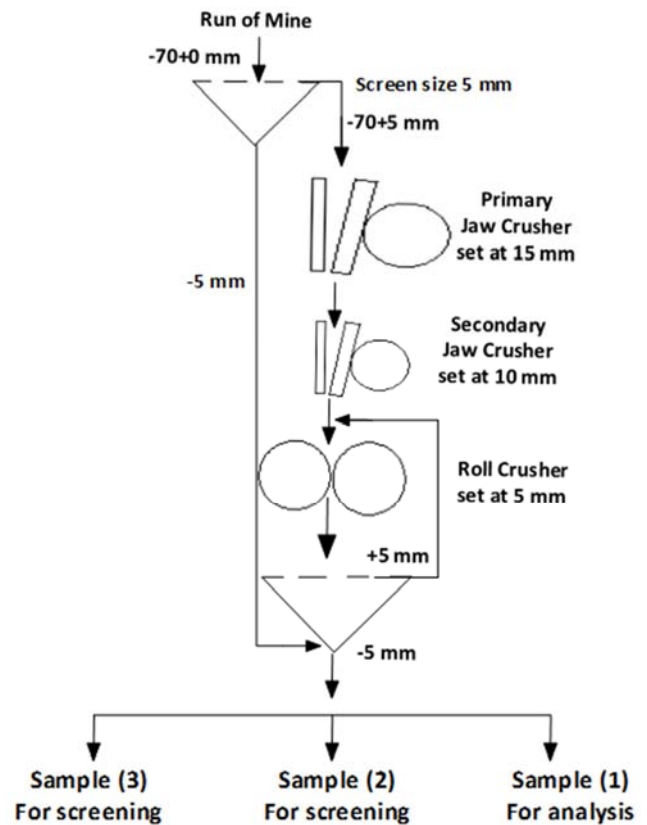


Figure 2. Flowsheet of crushing original sample.

Table 2. Sieve and chemical analysis of samples resulted from crushing process.

Analysis No.	Size, mm	Sample (2) weight, g	Sample (3) weight, g	Collected sample		
				Total weight, g	Total weight, %	P ₂ O ₅ , %
1	(-5+3.15)	53.37	38.09	91.46	28.42	18.74
2	(-3.15+2)	54.71	48.17	102.88	31.97	18.69
3	(-2+1.25)	13.31	16.32	29.63	9.21	17.97
4	(-1.25+0.63)	16.57	24.14	40.71	12.65	18.09
5	(-0.63+0.4)	4.84	8.29	13.13	4.08	19.25
6	(-0.4+0.25)	3.67	6.75	10.42	3.24	21.08
7	(-0.25+0.16)	3.69	7.97	11.66	3.62	20.64
8	(-0.16+0.1)	2.23	4.72	6.95	2.16	21.71
9	(-0.1)	5.00	10.00	15.00	4.66	21.38
Sum		157.39	164.45	321.84	100.00	18.93

4. Results and Discussion

4.1. Crushing and Sieving of the Head Sample

This technique utilizes the differences in differential friability between phosphate minerals and associated gangue and cementing matrix (carbonates, silica, and silicates). In

most cases, phosphate minerals are friable while gangue minerals are hard. By crushing and screening, the fine fraction will be rich in phosphates, but high percentage of P₂O₅ may be lost in the coarse fraction [10].

Cumulative weight% passed, cumulative P₂O₅% passed, and cumulative% of P₂O₅ recovery of samples in Table 2 were calculated and tabulated in Table 3 and shown in Figure 3.

Table 3. Cumulative weight% passed, cumulative P₂O₅% passed, and cumulative% of P₂O₅ recovery of samples resulted from crushing process.

Analysis No.	Size, mm	Wt. %	Cum. wt. % passed	Cum. P ₂ O ₅ % passed	Cum. % of P ₂ O ₅ recovery
1	-5	28.42	100.00	18.93	100.00
2	-3.15	31.97	71.58	19.00	71.86
3	-2	9.21	39.62	19.24	40.29

Analysis No.	Size, mm	Wt.%	Cum. wt.% passed	Cum. P ₂ O ₅ % passed	Cum.% of P ₂ O ₅ recovery
4	-1.25	12.65	30.41	19.63	31.54
5	-0.63	4.08	17.76	20.73	19.45
6	-0.4	3.24	13.68	21.17	15.30
7	-0.25	3.62	10.44	21.19	11.69
8	-0.16	2.16	6.82	21.48	7.74
9	-0.1	4.66	4.66	21.38	5.26

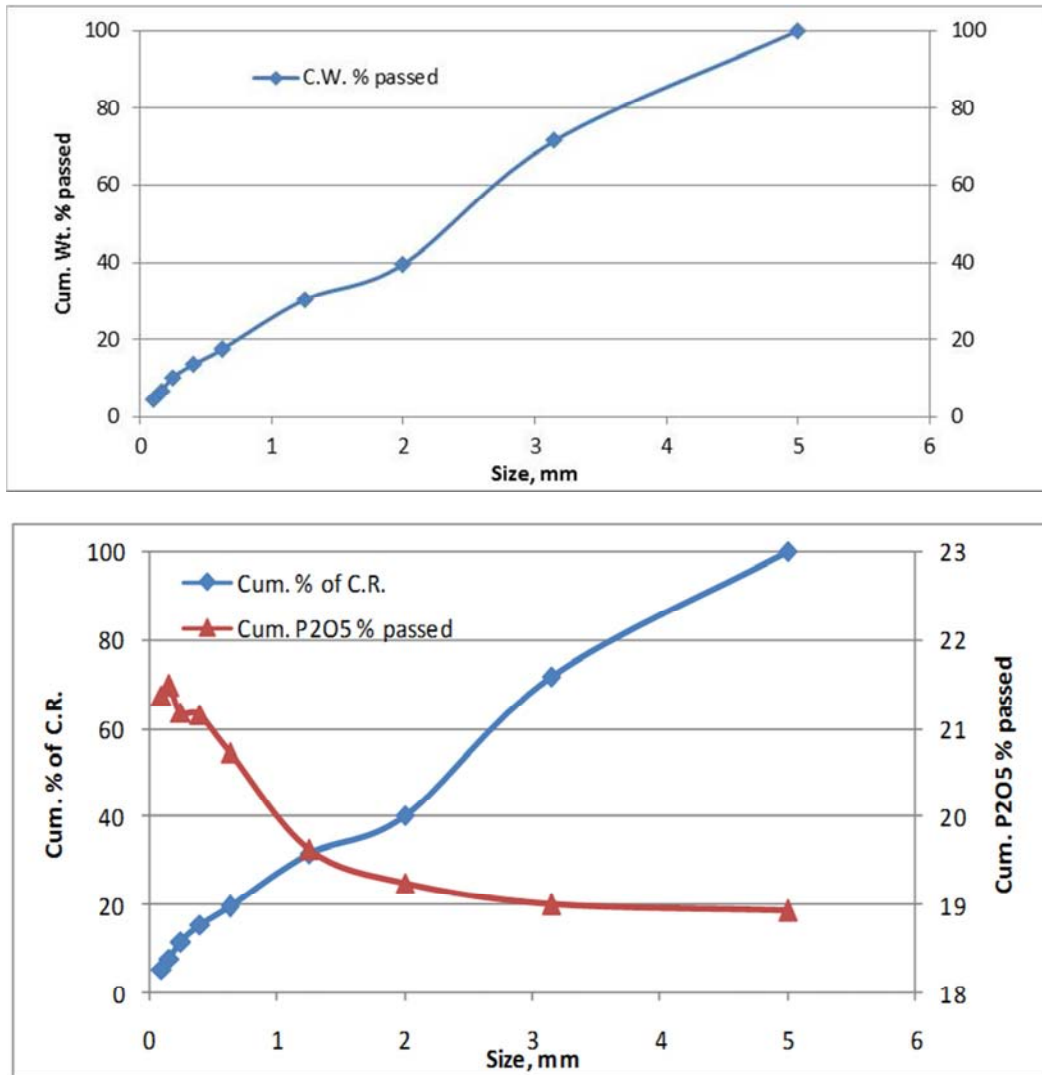


Figure 3. Cumulative weight% passed, cumulative P₂O₅% passed, and cumulative% of P₂O₅ recovery of samples resulted from crushing process.

Three alternatives of samples obtained from crushing and sieving were compared to obtain the highest grade of P₂O₅ with the greatest recovery. The results are shown in Figure 4 and tabulated in Table 4. From the results of different options, it was revealed that the second option (i.e. collection

of sizes below 0.400 mm) was the best one where the percent of P₂O₅ is 21.17%, recovery of P₂O₅ is 15.30%, and weight recovery equals to 13.68%. These results assured that the crushing process did not succeed to produce a suitable grade and recovery.

Table 4. Different alternatives of samples obtained from crushing and sieving.

Option No.	Particle size, mm	Wt. rec.%	P ₂ O ₅ %	Recovery of P ₂ O ₅ ,%
O1	(-0.630+0)	17.76	20.73	19.45
O2	(-0.400+0)	13.68	21.17	15.3
O3	(-0.250+0)	10.44	21.19	11.69

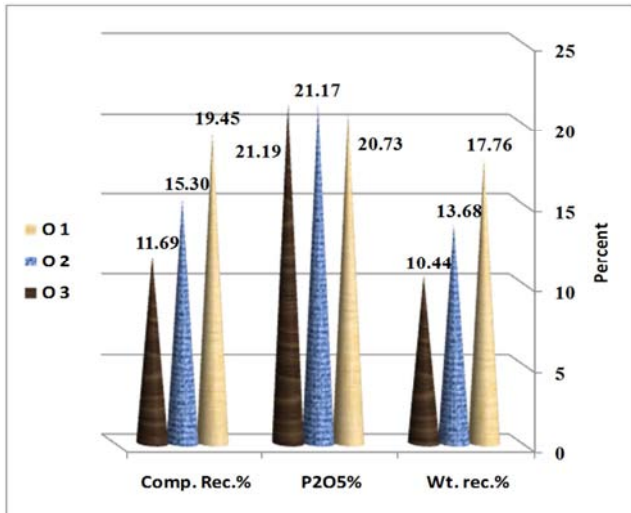


Figure 4. Different alternatives of samples obtained from crushing and sieving.

4.2. Calcination of Original Sample

Calcination is the process of heating the ore to a high temperature ranging from 800° to 1000°C to decompose the CaCO₃ and MgCO₃ to CaO, MgO and gaseous CO₂. The CaO and MgO formed are then removed as hydroxides by quenching the calcined product in water and washing. The most common chemical reagent used to enhance the removal of calcium and magnesium hydroxides is the ammonium chloride, NH₄Cl [11].

The results of calcination process are tabulated in Table 5 and Figure 5. From these results; it can be seen that the calcination process raised the P₂O₅ from 18.93% to 22.4%, with weight recovery 83.75%, and P₂O₅ recovery 99.10%, due to the decomposition of calcium and magnesium carbonates.

Table 5. Results of calcination process.

Test No.	Sample	Weight, g	Weight, %	P ₂ O ₅ %	Recovery of P ₂ O ₅ , %
1	Original sample before calcination	103.6	100	18.93	100
2	Original sample after calcination	86.77	83.75	22.40	99.10

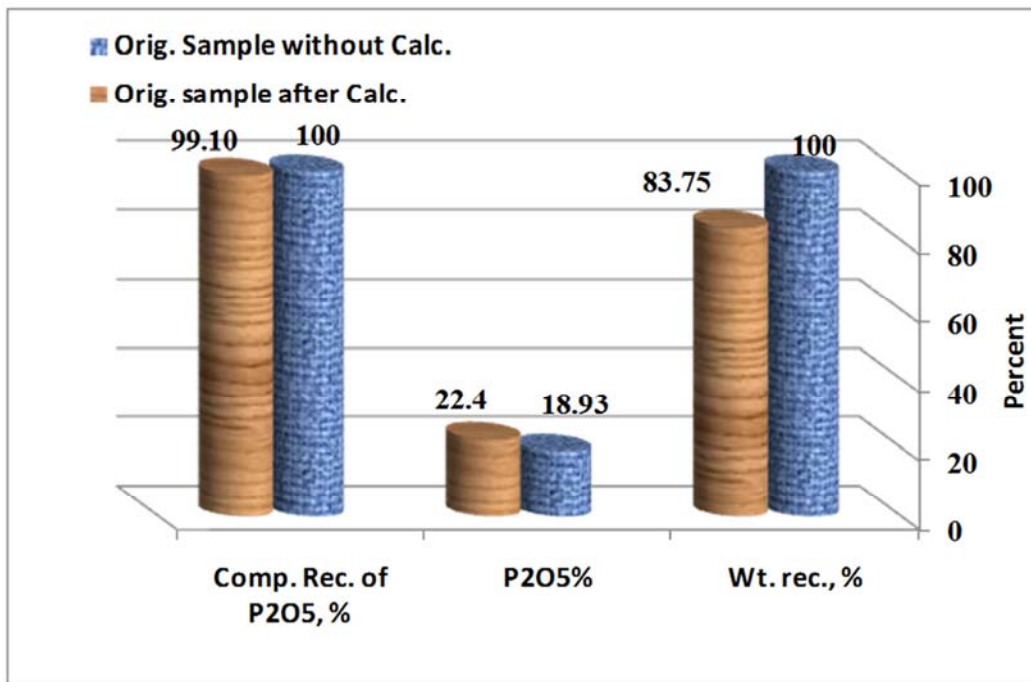


Figure 5. Results of calcination process.

4.3. Effect of Grinding Process

The obtained results were illustrated in Table 6 and Figure 6. Two options of sizes obtained from grinding and sieving were proposed to obtain the highest grade of P₂O₅ with the greatest recovery. The results are shown in Table 7 and Figure 7. From studying the different options, it was cleared that the first option (i.e. sizes below 100 μm) gave a percent

of P₂O₅ 23.88%, P₂O₅ recovery 42.37%, and weight recovery equals to 33.68%. The second option (i.e. sizes below 160 μm) gave P₂O₅ percent of 22.52%, P₂O₅ recovery of 53.25%, and weight recovery equals to 44.88%. These results revealed that the grinding and sieving processes succeeded to produce a suitable grade and recovery.

Table 6. Cumulative weight% passed, cumulative P₂O₅% passed, and cumulative% of P₂O₅ recovery of grinding samples.

Test No.	Size, mm	Weight,%	P ₂ O ₅ %	Size, mm	Cum. wt.% Passed	Cum. P ₂ O ₅ % Passed	Cum. rec. of P ₂ O ₅ ,%
1	(-1+0.63)	9.09	12.93	-1.00	100	18.98	100.00
2	(-0.63+0.40)	14.29	16.03	-0.63	90.91	19.59	93.81
3	(-0.40+0.25)	16.75	16.08	-0.40	76.62	20.25	81.74
4	(-0.25+0.16)	14.99	18.11	-0.25	59.87	21.42	67.55
5	(-0.16+0.10)	11.2	18.43	-0.16	44.88	22.52	53.25
6	(-0.10+0)	33.68	23.88	-0.10	33.68	23.88	42.37
Sum		100.00	18.98				

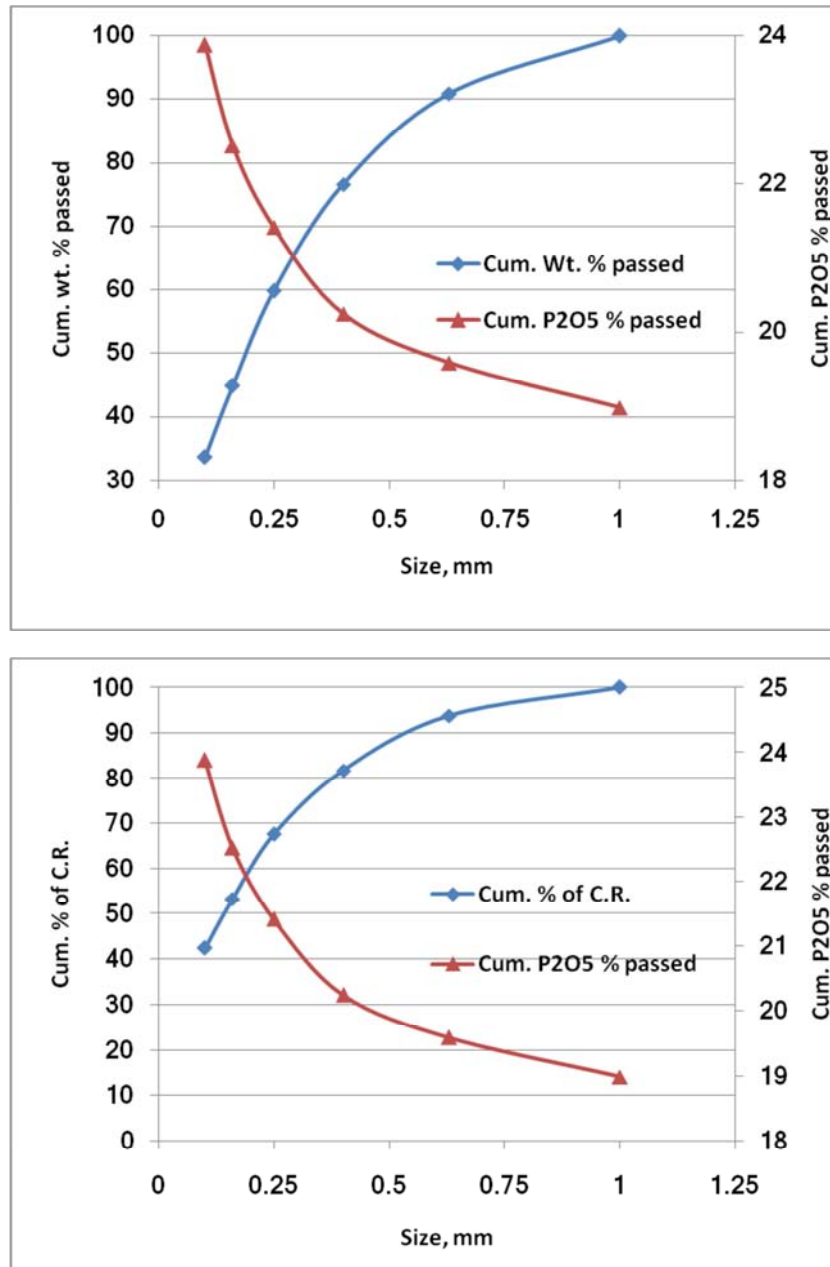


Figure 6. Cumulative weight% passed, cumulative P₂O₅% passed, and cumulative% of P₂O₅ recovery of samples resulted from grinding process.

Table 7. Different options of samples obtained from grinding and sieving processes.

Option	Size, mm	Weight recovery,%	P ₂ O ₅ ,%	P ₂ O ₅ rec.,%
O1	(-0.100+0)	33.68	23.88	42.37
O2	(-0.160+0)	44.88	22.52	53.25

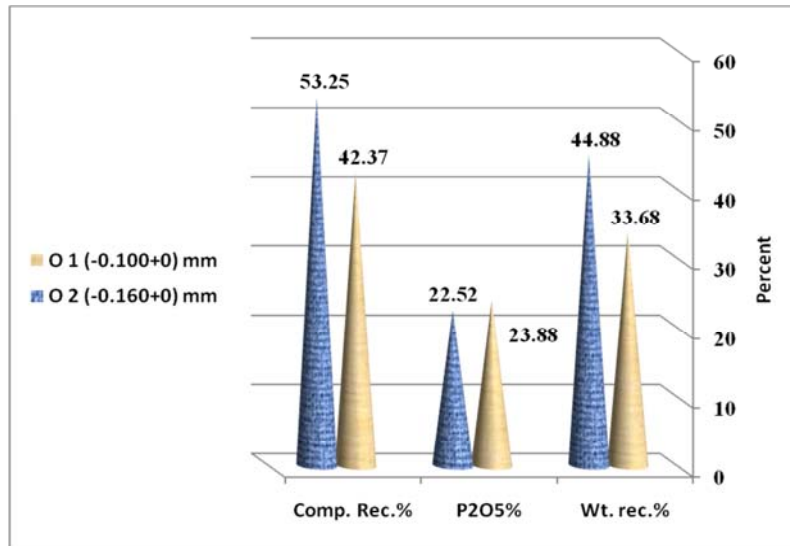


Figure 7. Different options of samples obtained from grinding and sieving processes.

Table 8 and Figure 8 illustrate the final results of grinding process. From these results it can be seen that the fraction (-1+0.630) should be sent to the waste dump because of the lower percent of P_2O_5 . An attrition process may be carried out to the fraction (-0.630+0.100).

Table 8. Final results of grinding and sieving processes.

Size, mm	Weight,%	P_2O_5 ,%	Remarks
(-1+0.630)	9.09	12.93	To waste dump
(-0.630+0.100)	57.23	17.06	To attrition process
(-0.100)	33.68	23.88	Final product

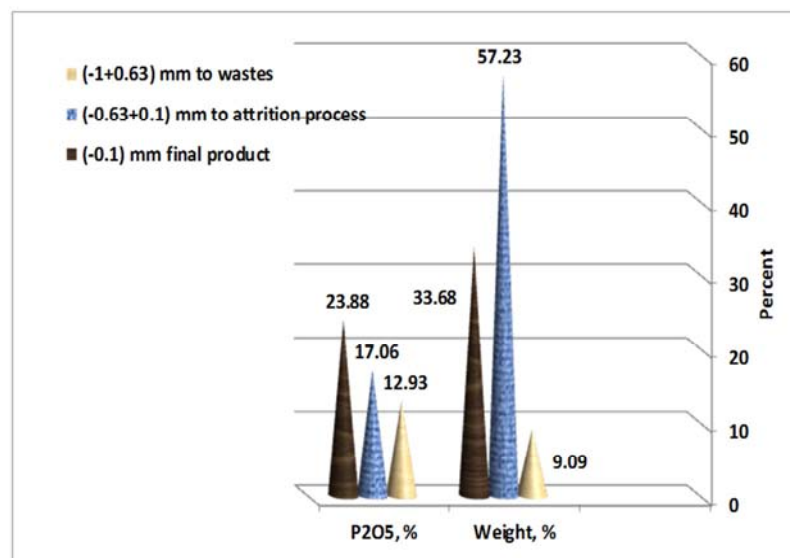


Figure 8. Final results of grinding processes.

4.4. Leaching Process with Acetic Acid

Calcium and magnesium carbonates are readily dissolvable in both mineral acids (strong acids) and organic acids (weak acids). In the case of calcareous phosphate ores, although mineral acids dissolve carbonates at high reaction rates, they also attack the phosphorus bearing minerals and cause losses in the P_2O_5 content of the ore if the intention is only to upgrade

the ore not to dissolve the phosphates. To avoid this problem, organic acids have been studied as carbonate leaching agents although their reaction rates are slow [12-13].

These organic acids may be expensive and will certainly add to the production cost. On the other hand, they are selective to leaching carbonates, their capital cost is low, they do not cause environmental hazards, and they can be recycled [12-13]. The organic acids most commonly used in carbonate

leaching are: acetic acid, citric acid, and formic acid.

The final results are shown in Table 9 and Figures 9 through 12. The different parameters studied were as follows:

- a. The solid to liquid ratio was changed from 10% to 30% at fixed agitation speed of 1200 rpm, constant leaching time, and acid to ore ratio 10% at 30 minutes. From these results, it can be seen that the best values were given at solid to liquid ratio 15% where the percent of P₂O₅ raised from 18% to 22.23% with weight recovery about 86%.
- b. The leaching time was changed with values 15, 30, and 45 minutes at fixed agitation speed of 1200 rpm, constant solid to liquid ratio of 15%, and acid to ore ratio 10%. From these results, it can be seen that the best values were obtained at 15 minutes where the percent of P₂O₅ raised from 18% to 22.35% with weight

- recovery about 86%.
- c. The leaching agitation speed was changed with values 800, 1200, and 1800 rpm at fixed leaching time of 30 minutes, constant solid to liquid ratio of 15%, and acid to ore ratio 10%. From these results, it can be shown that the best values were obtained at 1200 rpm where the percent of P₂O₅ increased from 18% to 22.23% with weight recovery about 86%.
- d. The values of acid to ore ratio were changed with values 10%, 16.7%, and 167.7% at leaching time 30 minutes, agitation speed 1200 rpm, and constant solid to liquid ratio of 15%. From these results, it can be revealed that the best values were obtained at acid to ore ratio 16.7% where the percent of P₂O₅ increased from 18% to 21.44% with weight recovery about 88.7%.

Table 9. Final results of leaching process.

Exp. No.	S/L ratio,%	Leaching time, min.	Agitation speed, rpm	Acid/ore ratio,%	Weight recovery,%	P ₂ O ₅ ,%
1	10	30	1200	10	86	22.1
2	15	30	1200	10	86	22.23
3	30	30	1200	10	85.3	21.58
4	15	15	1200	10	86	22.35
5	15	30	1200	10	86	22.23
6	15	45	1200	10	85.5	21.58
7	15	30	800	10	85.3	21.9
8	15	30	1200	10	86	22.23
9	15	30	1800	10	85.7	21.77
10	15	30	1200	10	93.3	20.27
11	15	30	1200	16.7	88.7	21.44
12	15	30	1200	167.7	85.3	20.92

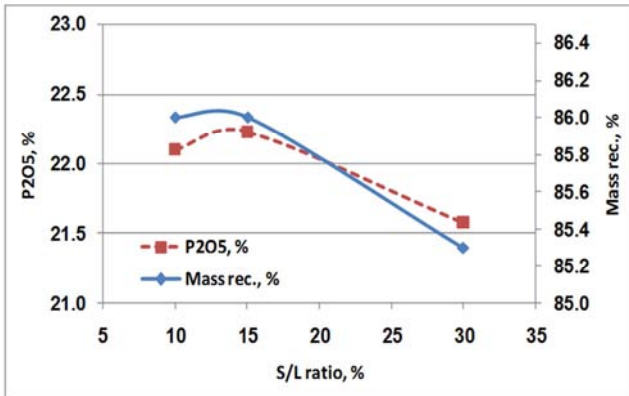


Figure 9. Effect of solid to liquid ratio on weight recovery and P₂O₅%.

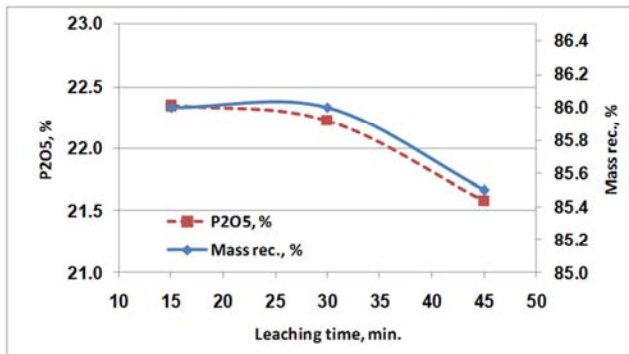


Figure 10. Effect of leaching time on weight recovery and P₂O₅%.

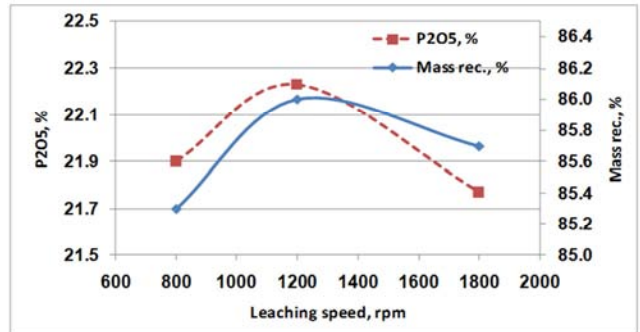


Figure 11. Effect of agitation speed on weight recovery and P₂O₅%.

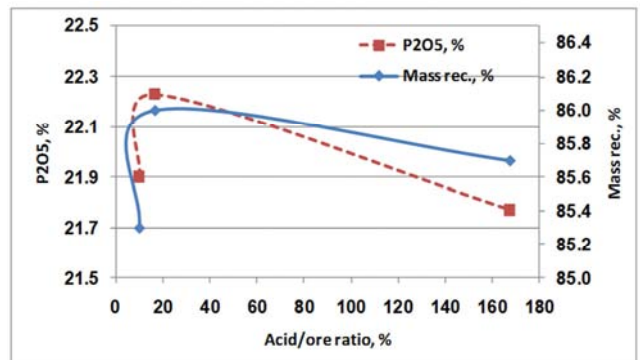


Figure 12. Effect of acid to ore ratio on weight recovery and P₂O₅%.

4.5. Leaching of Some Particle Sizes Produced from Grinding Process

The obtained results are illustrated in Tables 10 & 11 and Figure 13. From these results, it can be revealed that leaching process on size -100 μm raised $\text{P}_2\text{O}_5\%$ from 23.88% to 28.44% with weight recovery 84%. The weight recovery and

P_2O_5 recovery with respect to the original feed were 28.29% and 42.39%, respectively. The leaching process on size -250 μm increased $\text{P}_2\text{O}_5\%$ from 21.42% to 25.51% with weight recovery 86.33%. The weight recovery and P_2O_5 recovery with respect to the original feed were 51.69% and 69.47%, respectively.

Table 10. Leaching conditions of some particle sizes produced from grinding process.

Acid/ore ratio, %	Leaching speed, rpm	Leaching time, min.	S/L ratio, %	Exp.
10	1200	15	15	-100 μm
10	1200	15	15	-250 μm

Table 11. Final results of leaching process of some sizes produced from grinding process.

P_2O_5 rec. with resp. to original feed, %	$\text{P}_2\text{O}_5\%$ after leaching	$\text{P}_2\text{O}_5\%$ before leaching	Weight recovery with resp. to original feed, %	Weight recovery, %	Exp.
42.39	28.44	23.88	28.29	84.00	-100 μm
69.47	25.51	21.42	51.69	86.33	-250 μm

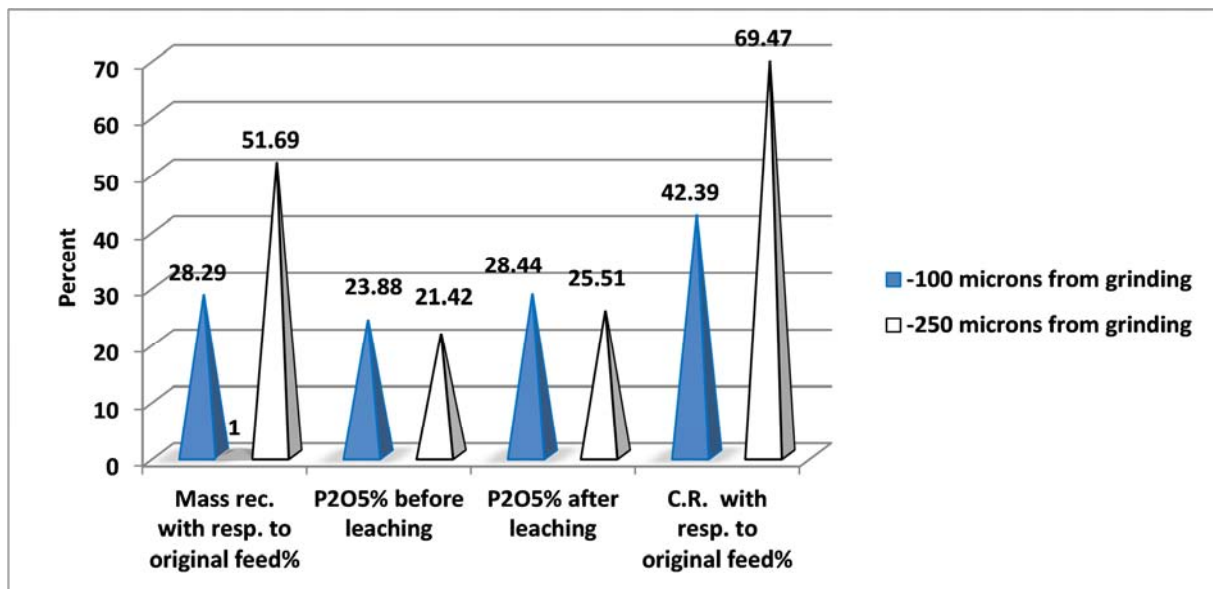


Figure 13. Final results of leaching process of some sizes produced from grinding process.

5. Conclusions

Three alternative samples obtained from crushing and sieving were compared to obtain the highest grade of P_2O_5 with the greatest recovery. The second option (i.e. sizes below 0.400 mm) was the best one where the percent of P_2O_5 is 21.17%, recovery of P_2O_5 is 15.30%, and weight recovery equals to 13.68%.

The calcination of feed sample upgraded P_2O_5 from 18.93% to 22.4%, with weight recovery of 83.75%, and P_2O_5 recovery of 99.10%.

Two options of sizes obtained from grinding and sieving were separated to obtain the highest grade of P_2O_5 with the greatest recovery. The first option (i.e. sizes below 100 μm) gave P_2O_5 percent of 23.88%, P_2O_5 recovery of 42.37%, and weight recovery equals to 33.68%. The second option (i.e. sizes below 160 μm) gave P_2O_5 percent of 22.52%, P_2O_5 recovery of 53.25%, and weight recovery equals to 44.88%.

Leaching process using acetic acid gave the best results at the following conditions: a) solid to liquid ratio 15% where the percent of P_2O_5 raised from 18% to 22.23% with weight recovery about 86%, b) Agitation time 15 minutes where the percent of P_2O_5 raised from 18% to 22.35% with weight recovery about 86%, c) Agitation speed 1200 rpm where the percent of P_2O_5 increased from 18% to 22.23% with weight recovery about 86%, d) Acid to ore ratio 16.7% where the percent of P_2O_5 increased from 18% to 21.44% with weight recovery about 88.7%.

The leaching process of size -100 μm raised $\text{P}_2\text{O}_5\%$ from 23.88% to 28.44% with weight recovery 84%. The weight recovery and P_2O_5 recovery with respect to the original feed were 28.29% and 42.39%, respectively. The leaching process on size -250 μm increased $\text{P}_2\text{O}_5\%$ from 21.42% to 25.51% with weight recovery of 86.33%. The weight recovery and P_2O_5 recovery with respect to the original feed were 51.69% and 69.47%, respectively.

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