

Production of calcium monohydrogenphosphate from sebaiya phosphate ore leached by nitric acid

A.A.El-Zahhar^{1*}, M.M.Ali², A.M.Ahmad³, A.A.El-Asmy⁴

¹NuclChemDept (EGYPT)

²N.M.A, Cairo, (EGYPT)

³Abu Zaabal Fert. Co., Cairo, (EGYPT)

⁴ChemDeptFaculty of Science, Mansoure Univ, Mansura, (EGYPT)

E-mail: adelelzahhar@yahoo.com

ABSTRACT

The production of phosphoric acid and phosphate salt from Sebaiya phosphate ore was studied by leaching with nitric acid. Various factors affecting the process as particle size, leaching time, leaching temperature, phosphate rock/HNO₃ ratio and mixing speed, have been investigated to estimate the optimum phosphate ore dissolution in relation to impurity. The produced aqueous acidic solution was neutralized in such a way that a pure calcium monohydrogenphosphate (CHP) for animal fodder is precipitated. © 2013 Trade Science Inc. - INDIA

KEYWORDS

Sebaiya phosphate ore;
Nitric acid;
Direct production;
Calcium
monohydrogenphosphate.

INTRODUCTION

Phosphoric acid (H₃PO₄) is the second acid produced after sulfuric acid in volume over the world. It is primarily used for the manufacture of phosphate salts, which are used in turn for production of fertilizers, animals fodder and detergents.

The composition of phosphate ores varies from one deposit to another therefore, phosphate rocks from different sources are expected to behave differently upon acidification processes. Phosphate ores are of two major geological origins, igneous and sedimentary. The phosphate minerals in both types of ore are of the apatite group, like: fluorapatite, Ca₁₀(PO₄)₆(F,OH)₂ and francolite, Ca₁₀(PO₄)_{6-x}(CO₃)_x(F,OH)_{2+x}. Fluorapatite predominates in igneous phosphate rocks while francolite predominates in sedimentary phosphate rocks^[1].

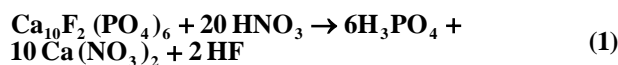
Phosphate ores occur in Egypt in three main provinces, Western desert, Nile valley, and Red sea. Added to these there are some phosphate-bearing sediments present in Wadi Qena, Wadi Araba, Esh El-Mallana range and Sinai^[2-4].

Phosphoric acid is produced by two methods, thermal and wet processes. The acid produced by thermal method is extremely pure, while the process is highly cost. The wet process is essentially based on the acidification of phosphate ores using mineral acid. The wet-process is the more popular because of increased demand for high-grade fertilizers, and energy saving (compared with thermal process)^[1,6].

The phosphoric acid produced by wet process represents 90% of the world current phosphoric acid. The wet process involves three subcategories, depending on the mineral acid used for the acidification. Sulfuric acid is used in acidification of phosphate rocks^[1,5-8]. Hydrochloric and nitric acids are also used in phos-

phoric production^[9,11-13,14-19].

When the phosphate rock treated with nitric acid, phosphoric acid and soluble calcium nitrate are formed according to the following equation:



The wet process involves firstly acidulating of crushed phosphate rock with excess nitric acid in a reaction vessel. The concentration of nitric acid used for acidulation should be within the range from 40 to 70%. The reaction temperature is in the range 120 - 130 °C, while the reaction time is ranged from 1 to 2.5 hrs. The produced phosphoric acid from this process contains 55 - 72 % by wt. P_2O_5 ^[10-13].

Food grade calcium monohydrogenphosphate has successfully produced from phosphoric acid produced by Abu-Zaabal Fertilizers and Chemicals Company by applying a process involves two steps. The first step includes clarification and defluorination of the produced crude phosphoric acid. The second step includes the precipitation of food grade dicalcium phosphate using calcium salt (calcium oxide, calcium hydroxide or calcium carbonate^[22]). The production of calcium monohydrogenphosphate for animal fodders usage was the goal of fertilizer industries. The production of calcium monohydrogenphosphate involves direct acidulation of phosphate rock with mineral acids. The process is much less mineral acid consumption than other conventional processes^[16, 19].

The present work aims at investigation of phosphoric acid production in wet process through leaching of phosphate rock with nitric acid. The experimental conditions that could affect the leaching process and product quality was studied as: particle size of phosphate rock, leaching time, leaching temperature, phosphate rock/ HNO_3 ratio and mixing speed (rpm). Sebaiya phosphate ore was used for this study to estimate the favored phosphate ore leaching conditions in relation to impurity and direct production of calcium monohydrogenphosphate (CHP).

EXPERIMENTAL

Materials

Nitric acid from MERCK, Germany, sulfuric acid

from FLUKA, Switzerland and phosphoric acid from BDH, England are chemical reagents grade and was used in the study. Calcium carbonate and calcium hydroxide from El-Nasr Pharmaceutical chemicals (ADWIC, Egypt) was used as a source of calcium ions. A compos-

TABLE 1 : Chemical analysis of sebaiya phosphate ore.

Constituent	%
P_2O_5	28.6
CaO	47.4
Fe_2O_3	2.3
F	2.9
SiO_2	6.5
Constituent	mg/kg
Cd	3.30
Pb	25.80
As	30.5

ite sample of Sebaiya phosphate ore was obtained from Abu Zaabal Fertilizers Co (AZFC). The chemical analysis of the phosphate ore is shown in TABLE 1.

Procedure

The reaction was performed out in a cylindrical 1 L reactor of 10 cm diameter, fitted with Teflon-coated stirrer and placed in thermostatically controlled water bath. The impeller tip speed was adjusted at 400 rpm unless otherwise stated. Filtration was performed using Buchner type filter of 4.6 in. diameter. Polypropylene filter cloth of 80 mesh aperture size and a vacuum pump were used for filtration.

The phosphate ore sample was crushed with a jaw crusher and then sieved using ASTM standard sieves to collect various sizes fractions for analysis. The sieved samples were dried in an electric oven at about 105 °C, cooled to room temperature and stored in closed desiccators. The dry samples were analyzed for P_2O_5 content and the results were given in TABLE 2.

Phosphate sample (5g) was transferred to the reactor with the proper determined amount of nitric acid solution. Defoamer (oleic acid) is added when necessary. After the desired reaction time, the leach slurry was immediately separated by filtration, while the remaining solids were dried and weighed. In the filtrate the P_2O_5 content was determined by a colorimetric method (spectrophotometer type Shimadzu UV 1208, using ammonium molybdate and ammonium

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metavanadate).

The production of calcium monohydrogenphosphate (CHP) was performed by adding 4 g of calcium carbonate to the proper amount of acidulation solution into the reactor. After appropriate reaction time, the produced CHP was filtrated, dried and weighted. The P_2O_5 content was determined in both CHP and the precipitation raffinate solution by colorimetric method (spectrophotometer type Shimadzu UV 1208, using ammonium molybdate and ammonium metavanadate).

RESULTS AND DISCUSSION

Leaching of phosphate ore using nitric acid

Leaching of Sebaiya phosphate ore was studied using nitric acid solution with studying different parameters that could affect the process. The studied parameters includes: particle size, reaction time, reaction temperature, nitric acid concentration, HNO_3 /phosphate ore mass ratio and stirring speed. The sieve analysis

TABLE 2 : Sieve analysis and P_2O_5 content of phosphate ore fractions.

Fraction no	Fraction, μm	Fraction wt, kg	Percent, %	P_2O_5 conc., %
1	1180	5.00	100.0	28.60
2	1180-1000	0.519	10.38	26.10
3	750 – 710	0.731	14.62	28.40
4	600 – 500	0.741	14.82	29.59
5	420 – 315	1.000	20.00	29.76
6	315 – 250	0.764	15.28	30.06
7	250 – 160	1.215	24.30	30.40

and P_2O_5 content in different fractions were analyzed and given in TABLE 2.

The leaching% (E%) of P_2O_5 was calculated as the following:

$$\text{Leaching E\% (P}_2\text{O}_5\text{)} = \frac{\text{Dissolved P}_2\text{O}_5\text{ (g) X 100}}{\text{Total amount of P}_2\text{O}_5\text{ in the ore (g)}} \quad (2)$$

The precipitation efficiency was calculated as:

$$\text{Precipitation efficiency E\%} = \frac{\text{Amount of P}_2\text{O}_5\text{ in CHP (g) X 100}}{\text{Total amount of P}_2\text{O}_5\text{ in the acidulate solution (g)}} \quad (3)$$

Effect of particle size

The effect of particle size on the phosphate ore

leaching using nitric acid was studied using phosphate ore of different particle size ($\leq 1180 \mu m$, 750- 700 μm , 600- 500 μm , 420- 315 μm , 315- 250 μm 250- 150 μm and 160- 63 μm). The leaching was of these fractions was studied at leaching time of 15 min, stirring speed of 400 rpm, temperature of 25 °C, nitric acid concentration of 2.5 % and acid/ phosphate ore mass ratio (L/S) of 25 ml/5 g. The results given in TABLE 3 representing P_2O_5 recovery% against and particle size, clarify that the phosphate ore particle size slightly affect the P_2O_5 recovery%. Therefore all investigations were carried out without particle size fractionation and frac-

TABLE 3 : Effect of particle size of phosphate ore on P_2O_5 recovery% from Sebaiya phosphate ore leached with nitric acid; time 15 min, temperature 25 °C, stirring speed 400 rpm, 1.0 M HNO_3 and L/S mass ratio 20 ml/ 5g.

Fraction μm	P_2O_5 leaching%
≤ 1180	20.5
1180 - 1000	15.9
750 – 710	17.2
600 – 500	17.5
420 – 315	20.6
315 – 250	20.8
250 – 160	20.9
160 – 63	20.5

tions with particle size $\leq 1180 \mu m$ was used to reduce production coasts.

Effect of reaction time

The leaching of phosphate ore using nitric acid solution was studied at different reaction time (1.0 to 60 min). The leaching process was studied using 10% nitric acid solution, reaction temperature of 25 °C, stirring speed of 400 rpm, L/S mass ratio of 25 ml/5 g and ore particle size $\leq 1180 \mu m$. The results represented in figure 1 reflect the increase of P_2O_5 with time, where the recovery% increased from 73.80 to 91.23% with increasing reaction time from 1.0 to 15 min. After 15 min there is a slight increase of P_2O_5 leaching with time. This finding indicates that the leaching reaction is fast under these experimental conditions and leaching time of 15min was considered in further experiments.

Effect of nitric acid concentration

The leaching of phosphate ore was studied using nitric acid solutions of different concentrations (2.5 -

20%). The leaching process was studied at leaching time of 15 min, stirring speed of 400 rpm, temperature of 25 °C, particle size fraction of $\leq 1180 \mu\text{m}$, and L/S ratio of 25 ml/5 g. The results given in figure 2 clarify that, when the acid concentration increased from 2.5 to 10%, the P_2O_5 recovery increased from 20.63 to 91.23%, while further increase in acid concentration than 10% led to slight increase in P_2O_5 leaching. This observation could be explained as; increasing the H^+ concentration increases the number of collisions with PO_4^{-3} ions in the phosphate ore crystal lattice and/or the H^+ ions collisions with PO_4^{-3} , HPO_4^{-2} , H_2PO_4^- in

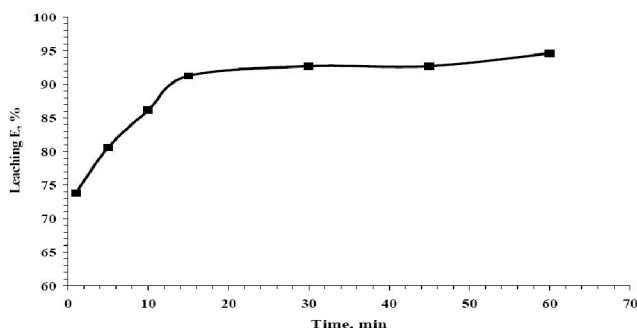


Figure 1 : Effect of reaction time on P_2O_5 leaching % from phosphate ore at 25 °C, stirring speed of 400 rpm, nitric acid concentration of 10 %, L/S mass ratio 25 ml/ 5 g and a particle size $\leq 1180 \mu\text{m}$.

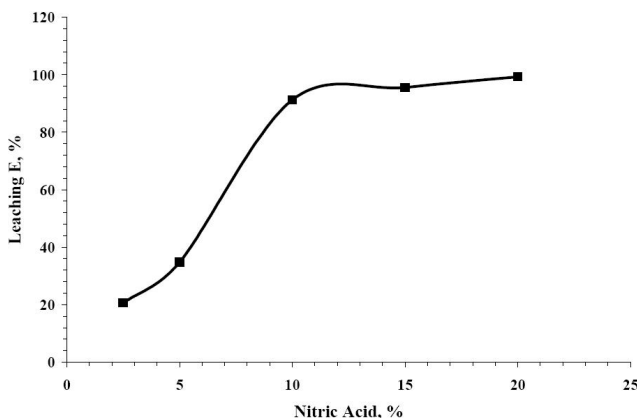


Figure 2 : Effect of acid concentration on P_2O_5 leaching % from phosphate ore at 25°C, reaction time 15 min, stirring speed of 400 rpm, L/S mass ratio 25 ml/ 5 g and ore particle size $\leq 1180 \mu\text{m}$.

aqueous phase. Based on the above results 10% nitric acid solution is appropriate for leaching of phosphate.

Effect of reaction temperature

The leaching of Sebaiya phosphate ore was studied using nitric acid solution at different reaction temperature (25-60 °C) (at reaction time 15 min, stirring

speed 400 rpm, acid concentration 10%, particle size fraction of $d^{75} 1180 \mu\text{m}$, and L/S mass ratio of 25 ml/5

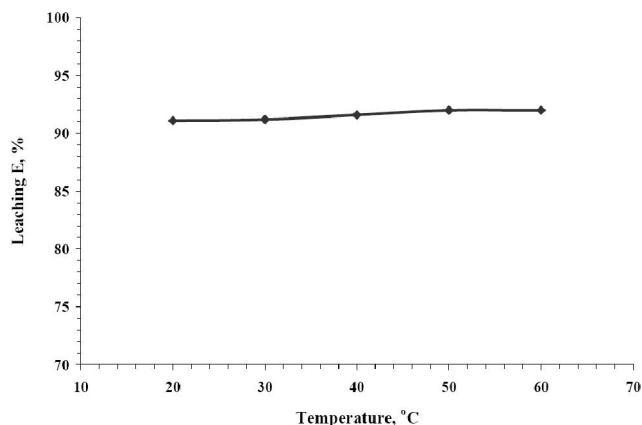


Figure 3 : Effect of reaction temperature on P_2O_5 leaching % from phosphate ore; reaction time 15 min, stirring speed of 400 rpm, acid concentration of 10%, L/S ratio 25 ml/ 5 g and ore particle size $\leq 1180 \mu\text{m}$.

g). The results given in figure 3 indicate that the reaction temperature has a slight effect on the reaction rate. Therefore, further experiments were performed at 25°C.

Effect of stirring speed

The leaching of Sebaiya phosphate ore was performed using 10% nitric acid at different stirring speed ranging (200 to 600 rpm) and reaction time of 15 min, L/S mass ratio 25 ml/ 5g, temperature 25 °C and ore particle size $\leq 1180 \mu\text{m}$. The results in figure 4 show very slight effect of stirring speed on the leaching process. Accordingly further experiments were carried out at mixing speed of 400 rpm.

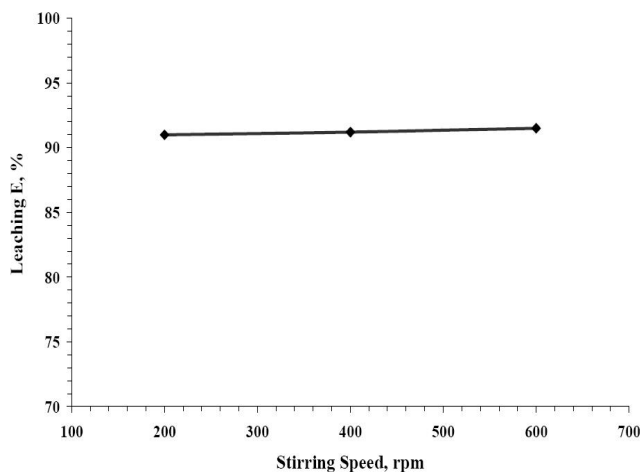


Figure 4 : Effect of stirring speed on P_2O_5 leaching % from Phosphate ore at L/S mass ratio 25 ml/ 5 g, temperature 25°C, time 15 min, acid concentration 10%, and ore particle size $\leq 1180 \mu\text{m}$.

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Effect of HNO₃/phosphate ore mass ratio, L/S

The leaching of phosphate ore using nitric acid solution was studied at different acid/phosphate ratio (v/w) (2/1 - 6/1 ml/g); reaction time 15 min, stirring speed 400rpm, temperature 25 °C and ore particle size d⁷⁵ 1180 μm. The results in figure 5 indicate that, as the liquid/solid ratio increased from 2/1 to 6/1, the recovery% of P₂O₅ leaching% increased from 36.21 to 96.50%. This observation indicates that the decrease in bulk density (increase volume/solid ratio) increases in P₂O₅ leaching rate, which could be due to increase number of collision between H⁺ and PO₄³⁻ ions in the phosphate ore crystal lattice with increasing number of H⁺ in solution. Also the number of H⁺ available for attacking the phosphate ore increased with increasing acid/phosphate ratio. The optimum volume/solid mass ratio of 6 is the optimum ratio for leaching of phosphate ore using nitric acid solutions.

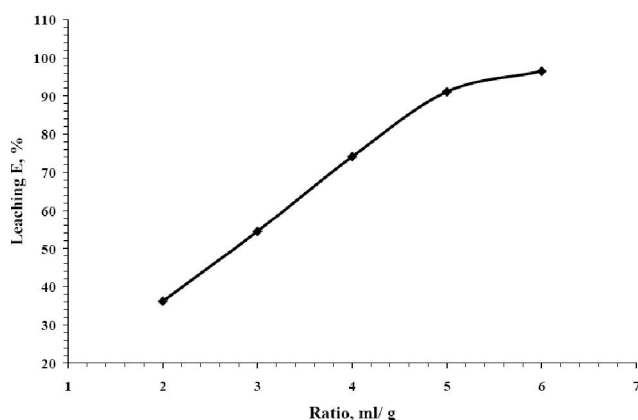


Figure 5 : Effect of HNO₃/phosphate ore ratio on P₂O₅ leaching % from Phosphate ore at stirring speed of 400 rpm, temperature 25°C, time 15 min, acid concentration 10%, and ore particle size ≤ 1180 μm.

Based on the above mentioned results on leaching of Sebaiya phosphate ore using nitric acid solution, it could be deduced that the optimum leaching conditions are: volume/solid ratio (L/S) at 6ml/g, acid concentration 10%, ore particle size ≤ 1180 μm, leaching time 15min, leaching temperature 25°C and stirring speed 400rpm.

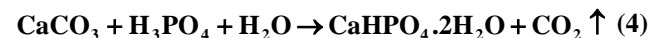
Accordingly, 600 ml of 10% HNO₃ was mixed with 100 g of Sebaiya phosphate ore of particle size ≤ 1180 μm and stirred for 15 min at room temperature. After filtration, the obtained acidulated solution was analyzed and the results were given in TABLE 4.

TABLE 4 : Analysis of the produced crud phosphoric acid solution (100 g phosphate ore, 600 ml HNO₃ 10 %, at 25°C, ore particle size of d⁷⁵ 1180 μm, and stirring speed 400rpm for 15 min).

Constituent	G	Constituent	ppm
P ₂ O ₅	27.55	Cd	0.65
Fe ₂ O ₃	1.41	Pb	2.51
F	1.10	As	6.00

Precipitation of calcium monohydrogenphosphate CHP

The precipitation of calcium monohydrogenphosphate (CHP) from the produced acidic solution was studied using calcium carbonate as source of calcium. Different parameters that could affect the precipitation process efficiency were studied as: mixing time, acid solution to calcium carbonate mass ratio, reaction temperature and stirring speed. The precipitation of calcium monohydrogenphosphate (CHP) from the produced acidic solution occurs according to the following equation:



Effect of mixing time

The precipitation of CHP from the produced acid solution was studied at different mixing times (1 - 60 min), reaction temperature 25°C, stirring speed 400rpm and solution to solid mass ratio L/S 50ml/4g. The results given in figure 6 clarify that as the mixing time increases from 1 to 15 min, the precipitation efficiency increases from 5.6 to 68.1%, while further increase in mixing time slightly increases the precipitation efficiency. Therefore, 15 min could be considered as appropriate

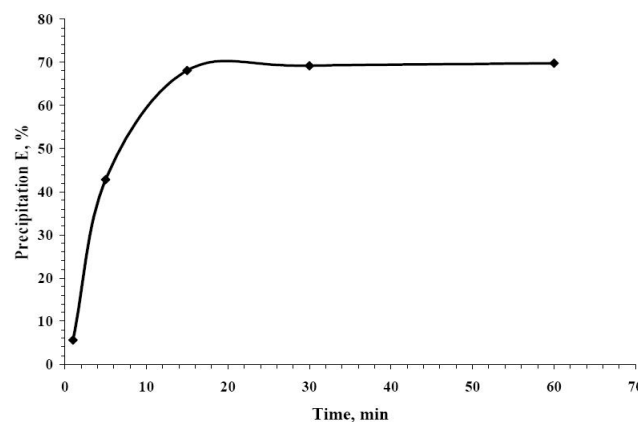


Figure 6 : Effect of mixing time on precipitation% of CHP from the produced acidic solution at 25°C, stirring speed of 400 rpm, L/S mass ratio 50 ml/4.0 g.

mixing time for CHP precipitation.

Effect of acid solution to calcium carbonate mass ratio L/S

The precipitation of CHP was studied as a function of acid solution/ calcium carbonate (L/S) ratio within range 50ml/3g -50ml/6g. The procedure was performed at 25°C, mixing time 15min and stirring speed 400rpm. The results presented in figure 6 indicate that, increasing the acidic solution/calcium carbonate ratio from 50 ml/3.0 g to 50 ml/6.0 g, increase the precipitation efficiency from 22.08 to 99.3%. Further increase in L/S ration produce very slight increase in precipitation efficiency, consequently the ratio 50 ml/5.0g was considered in further.

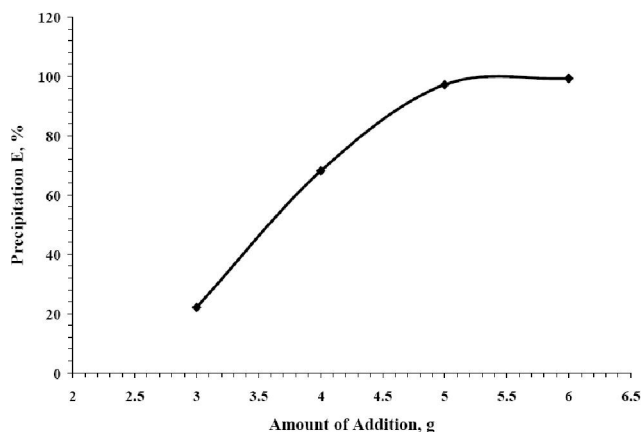


Figure 7 : Effect of acidic solution/calcium carbonate ratio (L/S) on the precipitation of CHP, at 25°C, stirring speed 400 rpm, reaction time 15 min.

Effect of reaction temperature

The precipitation of CHP was performed at different temperatures (20 - 60 °C) at reaction time 15 min, stirring speed 400 rpm and acid solution/calcium carbonate ratio of 50 ml/4 g. The obtained results in figure 8 show a slight effect of temperature on the precipitation efficiency (20-40 °C), while at (40 - 60 °C), the precipitation efficiency decreases. This could be due to the solubility of precipitated calcium monohydrogenphosphate (CHP) at high temperature. Therefore, room temperature is favored for calcium monohydrogenphosphate precipitation process using calcium carbonate as calcium source.

Effect of mixing speed

The precipitation of CHP was studied at different mixing speed (200-800 rpm), 25°C, reaction time

15min and L/S ratio of 50ml/5g using calcium carbonate as calcium source. The results in figure 9 indicate the increase of precipitation efficiency from 88 to 93 % as the stirring speed increases from 200 to 800 rpm. These results reflect a slight effect of stirring speeds upon the precipitation efficiency of CHP, as the reaction rate slightly affected by mixing speed. Consequently, 400 rpm could be considered in precipitation of CHP using calcium carbonate as calcium source.

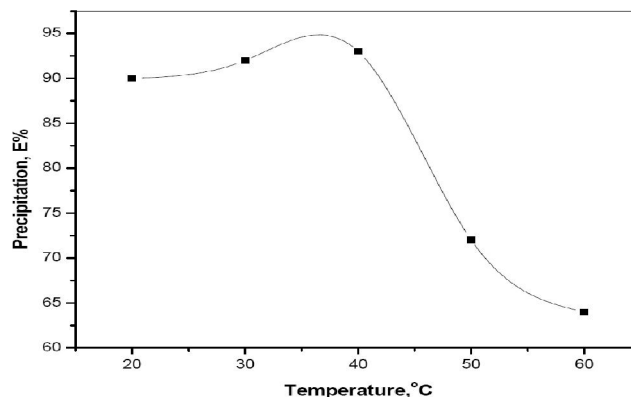


Figure 8 : Effect of temperature on CHP precipitation % from the produced acidulate solution at a stirring speed 400 rpm, reaction time 15 min, L/S ratio 50 ml/ 5 g.

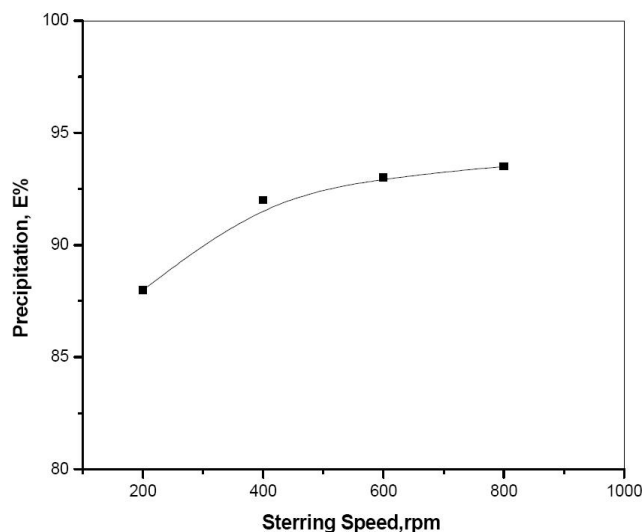


Figure 9 : Effect of stirring speed on CHP from the produced acidulate solution at 25°C, reaction time 15 min, L/S mass ratio 50 ml / 5.0 g.

Based on the above discussed results on CHP precipitation from the produced acidulate solution; a procedure for CHP production was developed. In this respect, 1000 ml of the produced acidulate solution was reacted with 100 g of calcium carbonate; mixing speed 400 rpm, reaction time 15 min and temperature 25°C.

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The precipitate was filtered and dried at 105 °C for 5 h. The produced CHP was analyzed and presented in TABLE 5 with the standard analysis of CHP given in the ISO-9001:2000. The results in TABLE 5 clarify that the specifications of CHP produced by the developed method is combatable with ISO-9001:2000 specifications.

TABLE 5 : Analysis of CHP produced from reaction of 100 g calcium carbonate with 1000 ml acidulate solution for 15 min at 25°C and stirring speed of 400 rpm –together ISO-9001:2000 standard specifications.

Characteristics	Produced DCP	Standard*
Solubility in water	Partially insoluble	Partially insoluble
Solubility in Citric acid 2 %	97 %	97 %
P	17.46 %	18 % Min
Ca	22.5 %	23 % Min
F	0.10 %	0.18 % Max
Cd	< 2 ppm	10 ppm Min
As	4.5 ppm	10 ppm Min
Pb	2.1 ppm	10 ppm Min
Moisture	14.5 %	14.5 %

* According to ISO-9001:2000 standard.

CONCLUSION

The leaching studies of Sebaiya phosphate ore using nitric acid solutions revealed that the optimum conditions for the leaching process are: particle size $\leq 1180 \mu\text{m}$, HNO_3 (10%), temperature (25 °C), HNO_3 /phosphate ore mass ratio L/S 6ml/1g, stirring speed (400 rpm) and reaction time 15 min. The leaching efficiency reached 96.5% under these conditions. Calcium monohydrogenphosphate (CHP) was successfully precipitated from the produced acidulate solution using calcium carbonate as calcium source. The precipitation efficiency reached more than 97% using acidulate solution/calcium carbonate mass ration of 10, reaction time 15 min and stirring speed 400 rpm at 25 °C. The specifications of the produced CHP are combatable with the ISO-9001:2000 standard specifications.

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