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## NON-CONVENTIONAL APPROACH FOR IMPURITIES REDUCTION DURING AND AFTER MANUFACTURING OF PHOSPHORIC ACID

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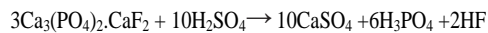
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### ABSTRACT

A non-conventional method to reduce the impurities in the wet process phosphoric acid (WPPA) was applied which divided into two parts: The first part deals with a process which utilizes phosphate rock, fluorosilicic acid, and sulfuric acid to produce phosphoric acid, calcium fluorosilicate, and calcium sulfate slurry by wet method. The second part studied the influence of hydroxyl-apatite (HAP) added to the produced acid for adsorption of uranium, fluorine, rare earth elements (REE), organic matter, and other undesirable components those affected the quality of phosphoric acid. The relevant conditions in the production and absorption processes have been optimized. The results show that, the obtained acid contains low values of suspended & organic matter (191.7 ppm), and fluorine (0.5%). The addition of HAP, in the form of bone powder, produce phosphoric acid contains low concentration of uranium, fluorine, and rare earth elements equals 85, 94.6 and 79% respectively.

### INTRODUCTION

The major constituent of phosphate rock, commonly known as apatite or fluorapatite, is represented by the formula  $\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2$ . Impurities are commonly present also, such as iron, aluminum compounds, calcium carbonate, and silica, but such impurities are of little or no effective value and are substantially eliminated in the process of phosphoric acid production. Such phosphate rock is commonly used as a starting material in the manufacture of phosphate fertilizers (Becker, 1983). In making the so-called "triple-phosphate" for example, the rock is acidulated with (70%) sulfuric acid in which the following reaction takes place:



The amount of  $\text{H}_2\text{SO}_4$  required is considerable being equivalent to the amount of calcium sulfate formed regardless of the source of the calcium, but the impure  $\text{CaSO}_4$  formed has practically no use whatever and in most cases creates a severe disposal problem. Thus acidulation of phosphate rock with  $\text{H}_2\text{SO}_4$  brings about a substantial economic loss, and many trials were performed to avoid this loss by eliminating the use of  $\text{H}_2\text{SO}_4$  for this purpose (Abdel Aal, 1989; Abd-el-Gawwad, 2003; Abdel Aal, 2000 and Singh and Vijayalakshmi, 2001). Hechenbleikner, (1991) states that when phosphate rock is acidulated with hydro-fluorosilicic acid, the phosphoric acid obtained is easily filtered and is of greater purity than that obtained in commercial WPPA plant. Recovering of the calcium, large part

of the fluorine from the phosphate rock in the form of valuable by-products (instead of the relatively worthless and discarded calcium sulfate), and the excess hydro-fluorosilicic acid was achieved. It is known that phosphate rock, which generally contains phosphorus in the form of  $\text{Ca}_{10}\text{F}_2(\text{PO}_4)_6$  can be acidulated with sulfuric acid to recover phosphoric acid by means of the "wet acid" process. The resulting phosphoric acid product, termed "Green acid" is suitable for use in fertilizer applications, but is not suitable for most other applications because of its high impurity level (Sweat et al., 2004; Lapple and Mountainside, 1967; Erickson and Bouffard, 1993; Oneill, 1980 and Vernooy and Michel, 2001).

One method for overcoming the problems of acidulation with sulfuric acid is described by Winter (1953). The process is carried out by acidulating phosphate rock containing silica with hydro-fluorosilicic acid ( $\text{H}_2\text{SiF}_6$ ) to obtain  $\text{CaSiF}_6$  and  $\text{H}_3\text{PO}_4$  as products of the reaction. The resultant phosphoric acid solution is then heated to volatilize any remaining hydro-fluorosilicic acid. This process also has serious drawbacks in which it is difficult to crystallize the calcium fluorosilicate from the phosphoric acid solution on a plant scale by merely volatilizing water from the hot  $\text{H}_3\text{PO}_4$  solution without encountering serious operational difficulties. This is due to the high solubility of  $\text{CaSiF}_6 \cdot 2\text{H}_2\text{O}$  in  $\text{H}_2\text{SiF}_6$  present in the phosphoric acid solution, which hinders recovery by crystallization (Laesen and Ravenholf, 1994).

Additional processes have also been suggested in which acidulation of the phosphate rock is carried out with other mineral acids, including hydrofluoric acid. A hydrofluoric acid-based process is generally impractical for commercial operation because the rate of rock attack with an acid of practical strength is too slow and because of the high cost of this acid (Schorr et al., 2013). Erickson and Bouffard (1996) studied the reaction of fluorosilicic acid with phosphate rock in order to produce wet process phosphoric acid and calcium fluo-

ride which is later reacted with sulfuric acid to produce hydrogen fluoride and calcium sulfate. The hydrogen fluoride is then stripped from the phosphoric acid and recovered as either anhydrous hydrogen fluoride or concentrated hydrofluoric acid or reacted with sodium carbonate to produce sodium fluoride/bi-fluoride.

Zhang et al. (2014) studied the mechanism and removal effect of uranium by HAP from acidic media. The effect of reaction time, pH value, initial concentration of uranium, dosage of apatite on adsorption of HAP for uranium was studied. The adsorption process was analyzed by thermodynamics and kinetics, and the adsorption mechanisms were determined by infrared spectroscopy, X-ray diffraction and scanning electron microscope. The result show that the removal capacity of uranium increases with initial concentration and the adsorption rate of HAP on  $\text{UO}_2^{2+}$  reaches 85%. Gauglitz et al. (1992) performed a process for immobilization of heavy metals by hydroxyl-apatite. The results indicate that, HAP immobilizes uranium and rare earth elements by forming different phosphate phases. Some of these phases can be identified by X-ray diffraction analysis (XRD), e.g. saleeite  $\text{Mg}(\text{UO}_2)_2 \cdot 9\text{H}_2\text{O}$  or meta-autunite  $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 6\text{H}_2\text{O}$ .

Mourabet et al. (2015) studied the removal of fluoride from aqueous solution by adsorption on hydroxyapatite (HAP) using response surface methodology. Fluoride removal efficiency was 86.3% at the optimum combination of process parameters. Dynamic adsorption data were applied to pseudo-first-order and pseudo-second-order rate equations, and the result shows that, the time data fitted well to pseudo second order kinetic model. According to the correlation coefficients, the adsorption of fluoride on the hydroxyapatite was correlated well with the Langmuir and Freundlich models.

Separative recovery with lime of phosphate and fluoride from an acidic effluent containing  $\text{H}_3\text{PO}_4$ , HF and/or  $\text{H}_2\text{SiF}_6$  was carried out by

Gouider et al. (2009). The data showed that fluoride has a beneficial effect on phosphate removal. Moreover, by acting on the precipitation pH, a selective recovery of fluoride and phosphate ions was possible either from phosphoric acid/hydrofluoric acid or phosphoric acid/hexafluorosilicic acid mixtures. The recovery process led to a fluoride removal efficiency of 97–98% from phosphoric acid/hydrofluoric acid mixture. Yulun et al. (2012) enhanced the adsorption of fluoride using Al modified calcium hydroxy-apatite, which possessed higher defluoridation capacity (DC) of 32.57 mgF<sup>-</sup>/g than unmodified HAP that have DC of 16.38 mgF<sup>-</sup>/g. The pH changes during the adsorption process suggested that the OH groups on the surface of Al-HAP acts as the adsorption sites. The more adsorption sites were formed on Al modified HAP, which possessed abundant surface hydroxyl groups, resulting in higher efficiency of F<sup>-</sup> removal.

The present work was oriented to reduce the undesirable impurities during the manufacturing of phosphoric acid by the addition of H<sub>2</sub>SiF<sub>6</sub> to the rock, and to absorb the unwanted matter after the production of the phosphoric acid by the addition of hydroxy-apatite (HAP, in the form of bone powder). The main goal tends to minimize the environmental pollution during storage of byproducts, transportation and usage, as well as to achieve a decontaminated product that can be safely used in many industrial applications to prepare low cost useful products.

## EXPERIMENTAL

### Materials and Instruments

The working phosphate ore has been supplied from the Sebaiya region which was mixed with another one from the Abu-Tarture region, Egypt. Hydroxy-apatite (HAP) in the form of bone powder was provided from Pratap Chemical Industries LTD, India. Sulfuric acid, silicon di-oxide and hydro-fluoride were provided from Sigma-Aldrich Chemie, Germany. Spectrophotometric determina-

tions were carried out using Metertech UV/VIS SP8001 spectrophotometer, China. This spectrophotometer covers wave length from 200-1100 nm. Trace element analysis was obtained using Unicam 929 atomic absorption spectrophotometer (AAS), England. Calcium fluorosilicate samples were analyzed using energy dispersive X-ray spectroscopy (EDX), JSM-840A, JEOL, Tokyo, Japan. The analysis of bone powder (HAP) was presented in Table (1).

The (HAP) bone powder {Ca<sub>5</sub>(OH)(PO<sub>4</sub>)<sub>3</sub>} contains about 55.8% CaO, 43.2% P<sub>2</sub>O<sub>5</sub>, 1.79% water and very small traces of heavy metals, chloride, nitrogen, sulfate and iron.

Table 1: Analysis of hydroxyl apatite in the form of bone powder

Tests	Specification
Appearance	White solid
CaO	55.8%
P <sub>2</sub> O <sub>5</sub>	41.2%
Water	1.79%
Heavy metals	0.008%
Total N	0.007%
Chloride	0.003%
Sulfate	0.003%
Iron	0.001%

### Qualification Procedure

The acidulation of phosphate ore was carried out by addition of sulfuric acid, hydro-fluorosilicic acid, and sufficient amount of water. The produced acid was analyzed for determination of P<sub>2</sub>O<sub>5</sub>, its clarification, and other involved materials. The produced CaSiF<sub>6</sub> was evaporated, crystallized, and undergone chemical analysis. The absorption of U, F, ΣREE, and undesirable materials was performed by the addition of bone powder. The optimum conditions such as: reaction time, concentration of reactants, solid/ liquid ratio, and agitation speed were studied. For the analysis of uranium and heavy rare earths, the

concentration of initial sample was measured ( $C_o$ ) before absorption process. After each process, the aqueous solution was separated and analyzed for uranium and total rare earths contents ( $\Sigma$ REE). The absorption efficiency percent (E) % was calculated as follows:

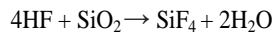
$$\text{Absorption efficiency (E) \%} = [(C_o - C_f)/C_o] \times 100$$

Where,  $C_o$  is the conc. of the initial sample and  $C_f$  is the conc. of treated one. The absorption efficiency (E) was expressed as enrichment percent (%) of studied materials.

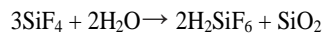
## RESULTS AND DISCUSSION

### Manufacturing Procedure

The hydro-fluorosilicic acid was prepared by the reaction of silicon dioxide with hydro-fluoric acid according to the following reaction:



The formed silicon tetra-fluoride is partially volatilized. The remainder would be hydrolyzed to form hydro-fluorosilicic acid and silica, as follows:



Hexa-fluorosilicic acid is also converted to a variety of useful hexa-fluorosilicate salts. During the process by-product fluorosilicic acid is intermittently recovered while the remaining acid is recycled (Erickson et al., 1993). The first step in the process comprises mixing hydro-fluorosilicic acid and sulfuric acid with phosphate rock to dissolve a substantial part of all its  $\text{P}_2\text{O}_5$  content and a substantial part of essentially all of its CaO content. The production of phosphoric acid from phosphate rock was achieved by applying certain conditions such as: stirring time 8 hrs, temperature  $70^\circ\text{C}$ ,  $\text{H}_2\text{SO}_4$ :  $\text{Ca}_3(\text{PO}_4)_2$  ratio is 1:1, added water  $\text{H}_2\text{O}$ :  $\text{Ca}_3(\text{PO}_4)_2$  ratio is 15:1, added phosphoric acid (15%  $\text{P}_2\text{O}_5$ )  $\text{H}_3\text{PO}_4$ :  $\text{Ca}_3(\text{PO}_4)_2$  ratio 10:1, (El-Shafie, 2008) and added hydro-fluorosilicic acid  $\text{H}_2\text{SiF}_6$ :  $\text{Ca}_3(\text{PO}_4)_2$  ratio 5:1.

Using proportions of rock and sufficient volume of two applied acids those are preferred in most cases, the general reaction is represented by the following equation to produce the so-called triple super-phosphate (Winter, 1953):



The analysis of major contents (%) and other trace elements (ppm) in mixed phosphate ore as well as manufactured phosphoric acid was shown in Table (2) and Figs (1&2).

Table 2 shows that, the produced acid have 29.6%  $\text{P}_2\text{O}_5$ , 3.95%  $\text{Fe}_2\text{O}_3$ , 3.25%  $\text{SiO}_2$ , 6.41% CaO, 0.5% F, and certain values of  $\Sigma$ REE, U, organic matter (OM) equal 410, 34.2, 191.7 ppm less than those in the commercial acid (607, 39.5, 472 ppm) respectively.

Calcium fluorosilicate is separated from the phosphoric acid by evaporating the solution and crystallizing the calcium salt. Calcium fluorosilicate is immediately available in most volumes, high purity, submicron and nano-powder forms. The analysis of major and trace contents in  $\text{CaSiF}_6$  &  $\text{CaSO}_4$ , the by-products from the acidulation of phosphate rock, was shown in Table (3).

Both of calcium salts contains a small amount of trace elements such as Cr, Ba, Co, Sr, Pb, Zn and V. The EDX analysis of  $\text{CaSiF}_6$  was shown on Figure (3).

### Absorption Investigation

Hydroxy-apatite (HAP) is a calcium phosphate similar to the human hard tissues in morphology and composition. An important characteristic of hydroxyl-apatite is its stability when compared to other calcium phosphates. Thermodynamically, hydroxy-apatite is the most stable calcium phosphate compound under physiological conditions as temperature, pH and composition of the body fluids (Deng et al., 2013). The addition of HAP in the form of bone powder to the phosphoric acid was studied. The factors affect the absorption process such as: time, conc. of phosphoric acid, amount of bone powder,

Table 2: Analysis of the mixed phosphate ore and produced phosphoric acid

Components	Phosphate ore	Produced acid	Commercial acid
<b>Conc. (%)</b>			
CaO	37.9	6.41	4.92
SO <sub>3</sub>	6.77	2.52	7.11
SiO <sub>2</sub>	12.1	3.25	4.75
P <sub>2</sub> O <sub>5</sub>	27.4	29.6	23.6
Fe <sub>2</sub> O <sub>3</sub>	9.03	3.95	6.85
K <sub>2</sub> O	0.17	0.04	0.08
Na <sub>2</sub> O	0.14	0.61	0.06
MnO	0.12	0.07	0.12
MgO	0.84	0.52	0.84
F	2.11	0.50	0.91
Σ Traces	0.18	0.06	0.17
<b>Conc. (ppm)</b>			
ΣREE	1410	410	607
U	95.6	34.2	39.5
Organic matter	504	191.7	472

Table 3: Analysis of by-products CaSiF<sub>6</sub> and CaSO<sub>4</sub>

Components	CaSiF <sub>6</sub>	CaSO <sub>4</sub>
<b>Conc. (%)</b>		
CaO	24.7	26.4
Si	51.2	8.36
F	19.5	0.01
SO <sub>3</sub>	1.17	39.2
Fe <sub>2</sub> O <sub>3</sub>	8.52	5.26
<b>Conc. (ppm)</b>		
Cr	36	62
Ba	17	19
Co	20	16
Sr	12	8.0
Pb	16	11
Zn	53	74
V	23	18
U	41	23
Σ REE	321	542

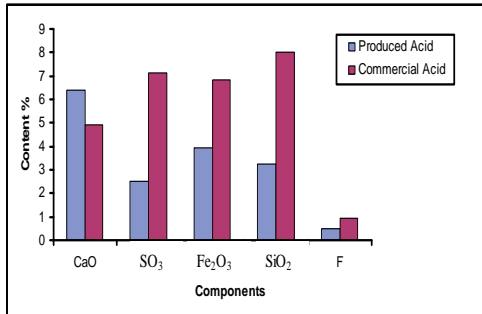
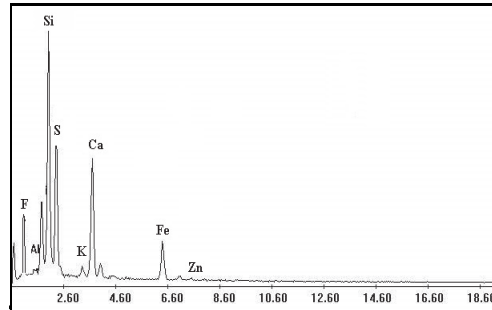
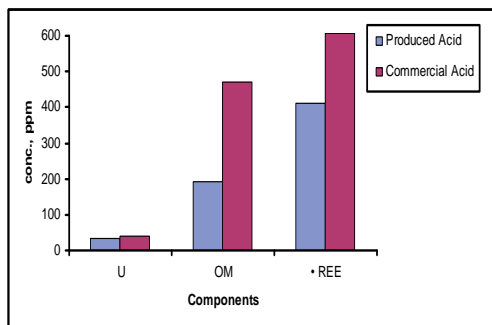
Fig. 1: Comparison between different components (%) in the produced phosphoric acid (29.6% P<sub>2</sub>O<sub>5</sub>) and commercial one (23.6% P<sub>2</sub>O<sub>5</sub>)

Fig. 3: EDX analysis for the produced calcium fluorosilicate

Fig. 2: Comparison between different trace elements (ppm) in the produced phosphoric acid (29.6% P<sub>2</sub>O<sub>5</sub>) and commercial one (23.6% P<sub>2</sub>O<sub>5</sub>)

agitation speed, and solid/liquid mass ratio were optimized as following:

#### Reaction time

At room temperature, the reaction time for the addition of HAP to the produced phosphoric acid was studied during 10-30 min. Agitation speed was 150 rpm. To 1L of phosphoric acid, 10 g of bone powder was added. The result was plotted on Figure (4).

The result indicates that, by increasing the time up to 20 min., the absorption efficiency increases to reach 41, 55 and 32% for U, F, and ΣREE respectively. The time 20 min. was

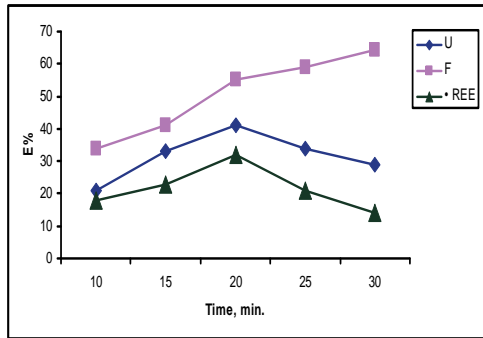


Fig. 4: Effect of time on the absorption efficiency (E) % on phosphoric acid

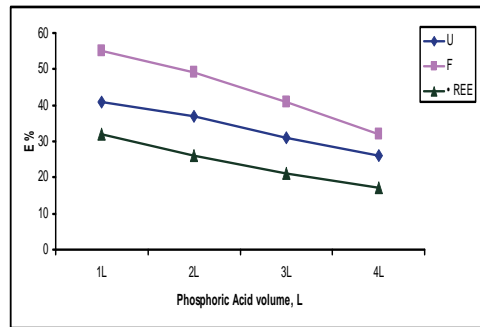


Fig. 5: Effect of phosphoric acid volume on the absorption efficiency (E) %

the optimum one for absorption of undesirable impurities from phosphoric acid.

#### Phosphoric acid volume

Different volumes of acid (1- 4L) were subjected to 10g of HAP addition for 20 min. at room temperature and standard conditions. The effect of phosphoric acid volume on the absorption process was shown on Figure (5).

The data show that, the absorption efficiency for U, F and  $\Sigma$ REE was decreased with the volume of acid. The viscosity of acid was increased with concentration. The mobility of HAP and the mass transfer was restricted and immobilized (Gauglitz et al., 1992).

#### Solid/liquid ratio

The amount of HAP, which added to 1L of phosphoric acid, was varied between 10-50g. The absorption process was carried out during 20 min. at room temperature with agitation speed of 150 rpm. The effect of amount of HAP was illustrated on Figure (6).

The data show that, by increasing the amount of HAP to 30 gm, the absorption efficiency (E) % was increased to reach 74, 85, and 67% for U, F and  $\Sigma$ REE respectively. After this value the acid became saturated with high amount of HAP (Chohey, 2004).

#### Agitation speed

The effect of agitation speed on the ab-

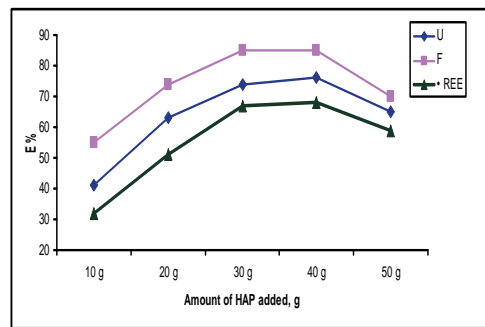


Fig.6: Effect of amount of HAP on the absorption efficiency (E) %

sorption process was studied in range 150-300 rpm. At room temperature, 30g of HAP were added to 1L of phosphoric acid during 20min. The effect of agitation speed was shown on Figure (7).

The result shows that, by increasing the agitation speed to 250 rpm, the absorption efficiency % was increased to become 80, 93 and 76% for U, F and  $\Sigma$ REE respectively, which give rise to good and high mass transfer between the reactants (Savova et al., 2005).

#### Choice of optimum conditions

Based on the results, the optimum conditions required for absorption of U, F and  $\Sigma$ REE from produced phosphoric acid were: contact time of 20 min., 30g of hydroxy-apatite (in the form of bone powder) to 1L of



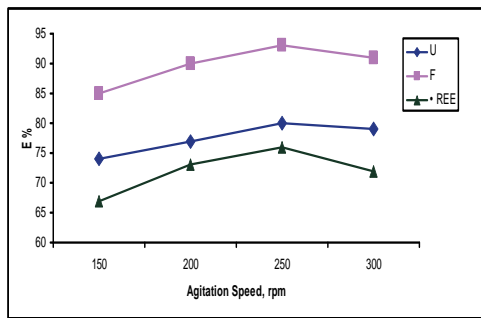


Fig. 7: Effect of agitation speed on the absorption efficiency (E) %

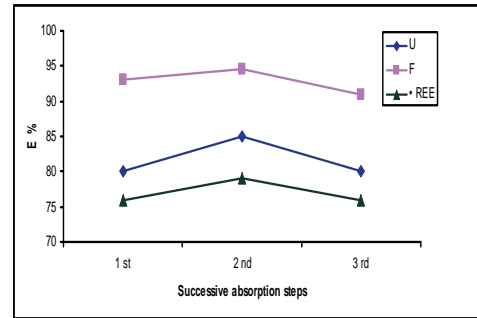


Fig. 8: Effect of successive step on absorption processes

phosphoric acid, and agitation speed of 250 rpm. This conditions lead to absorb of 80, 93 and 76% of U, F and  $\Sigma$ REE respectively.

#### Successive absorption step

In the successive absorption processes, the treatment of residual U, F, and  $\Sigma$ REE in the phosphoric acid was carried out on 3 steps. Under the optimum experimental conditions, the treated phosphoric acid samples were subjected to successive absorption process. The results are represented on Figure (8).

From the results, the absorption efficiency for U, F and  $\Sigma$ REE increases in the second step of process to reach the value of 85, 94.6 and 79 % respectively. On the other hand, the third step has no effect on the absorption of U, F and  $\Sigma$ REE from phosphoric acid that produced by non-conventional approach.

The proposal flow sheet for the overall process included the production and absorption step was illustrated on Figure (9).

The physical properties indicate that the obtained acid from non-conventional approach have green clear color with high density in comparison with the commercial one of dark brown color of high amount of humic acid (Table 4) produced in Abu-Zaabal Company for chemicals and fertilizers.

#### CONCLUSIONS

A successful absorption procedure of

phosphoric acid, produced by acidulation of phosphate rock with hydro-fluorosilicic acid and sulfuric acid in the so-called wet process phosphoric acid (WPPA), has been developed in the present work. This was possible through absorption of U, F and  $\Sigma$ REE by using hydroxy-apatite in the form of bone powder. The corresponding relevant factors were studied and the conclusions can be summarized as follows:

1- A method for manufacture of substantially pure calcium fluorosilicate and phosphoric acid, from mixed phosphate rock acidulated by hydro-fluorosilicic acid and sulfuric acid, leads to produce acid contains 29.6%  $P_2O_5$ , 3.95%  $Fe_2O_3$ , 6.41% CaO, 0.5% F and traces of some undesirable elements. The acid contains also 34.2 and 410 ppm of U &  $\Sigma$ REE respectively, with low values of organic matters, humic acids (191.7 ppm), and unwanted suspended materials.

2- The absorption of U, F and  $\Sigma$ REE by Hydroxy-apatite (HAP) in the form of bone powder was carried out at room temperature during 20 min. by the addition of 30 gm of HAP to 1L of the phosphoric acid produced by non-conventional approach. The absorption process gives rise to absorb of 85, 94.6 and 79% of U, F and  $\Sigma$ REE respectively after two successive steps. Most of uranium undergoes into  $CaSiF_6$  and  $CaSO_4$  residue due to the ionic radii similarity of calcium and uranium.

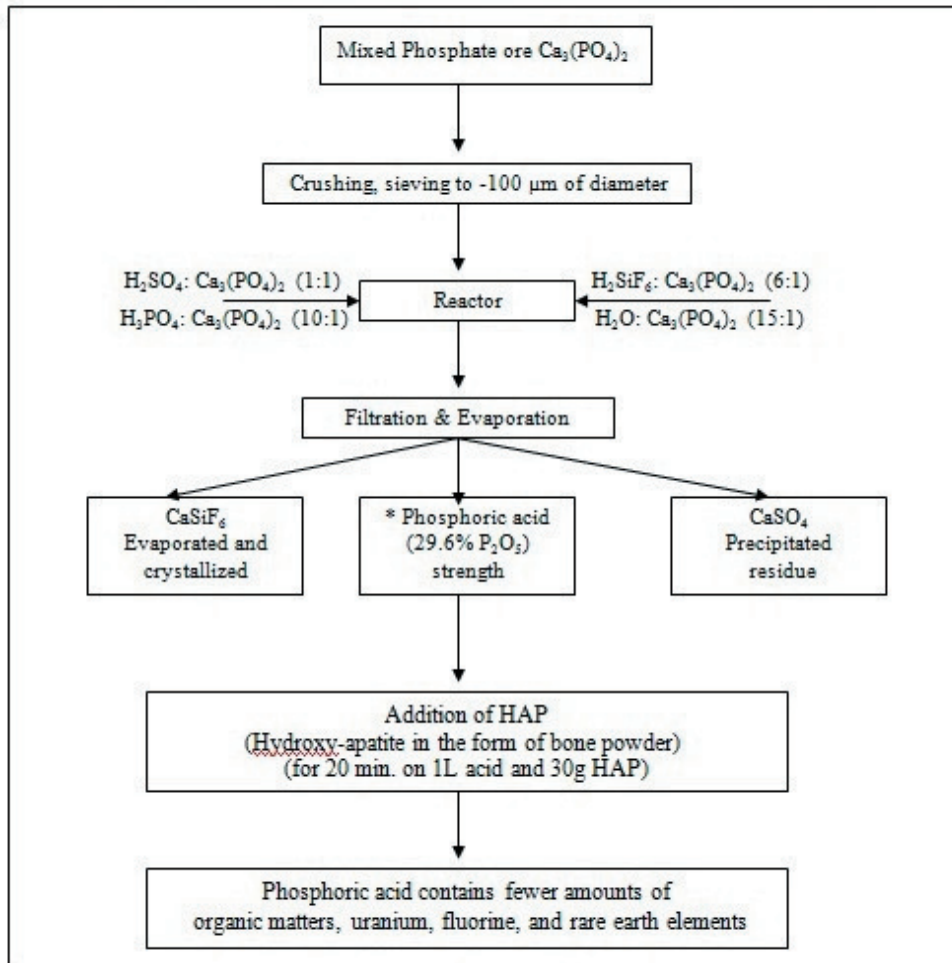


Fig. 9: Proposal flow sheet for manufacturing and absorption process

Table 4: The physical properties of the produced and commercial phosphoric acid

Character	Specification	
	Produced phosphoric acid	Commercial phosphoric acid
Appearance	green	dark brown
Odor	Slight acid odor	Slight acid odor
pH	< 2	< 1
Density	1.67	1.53



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## مقاربة غير تقليدية لخفض الشوائب أثناء وبعد تصنيع حمض الفوسفوريك

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تم تطبيق طريقة غير تقليدية لخفض نسبة الشوائب الناتجة خلال عملية تصنيع حمض الفوسفوريك بالطريقة الرطبة. ولقد انقسمت الدراسة إلى جزئين أساسيين ، الجزء الأول يتناول عملية إذابة صخور الفوسفات في حمض الهيدروفلوروسيليكات وحمض الكبريتيك المركزين لإنتاج حمض فوسفوريك وفلوروسيليكات الكالسيوم السداسية التي تتفاعل مع حمض الكبريتيك لترسيب كبريتات الكالسيوم (الفوسفوجبس). أما الجزء الثانى فيتناول عملية امتصاص اليورانيوم والفلور والعناصر الأرضية النادرة بواسطة مركب الهيدروكسي اباتيت المستخدم في شكل مسحوق عظام. وتمت دراسة جميع الظروف المناسبة المؤثرة علي عمليتي الانتاج والامتصاص، وقد اظهرت النتائج أن الحامض الناتج يحتوى على اقل كمية من المواد العضوية والعوالق (٧,١٩١) جزء في المليون ونسبة قليلة من الفلور (٥,٠%)، بالإضافة إلي إمتصاص ما يعادل 85% من اليورانيوم و٩٤,٦% من الفلور و٧٩% من العناصر الأرضية النادرة بإستخدام مسحوق العظام عند افضل وانسب الظروف.