ISSN NO. 2320-5407

Journal homepage: http://www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH

RESEARCH ARTICLE

Ion Exchange Recovery of Uranium and Rare Earths after their Selective Leaching from Abu-Tartur Phosphate Deposits, W.D., Egypt

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Manuscript Info

Manuscript History:

Received: 15 March 2015
Final Accepted: 18 April 2015
Published Online: May 2015

Key words:
Uranium, Rare Earths, Abu-Tartur,

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Abstract

A selective leaching procedure for uranium and the REEs is applied upon Abu-Tartur rock phosphate of the W.D. of Egypt using a mixed reagent of citric acid / calcium citrate in a manner to leave the phosphate values almost intact for later processing. The leaching conditions have thus been applied upon a representative sample of the weathered rock assaying 30 ppm U and 1058 ppm REEs. Before being processed, the representative sample was completely characterized through both mineralogical and chemical analyses of its major and some of the interesting trace elements. From the obtained leach liquor, uranium has been recovered by the anion exchange Amberlite IRA-400 resin and from the uranium-free effluent, the REEs were recovered via the cation exchange Dowex 50W-X8 resin.

1. Introduction

Abu -Tartur phosphorite lies at about 50 Km to the west of the Kharga town and covers an area of about 1200 Km^2 of the main Plateau between Kharga and Dakhla Oases in the Western Desert of Egypt and belongs to the Upper Cretaceous age. Besides the phosphorous value, rock phosphates could include a number of interesting trace elements where a small quantity of the Ca can isomorphously be substituted with many elements including Mg, Mn, Sr, Pb, Na besides uranium and rare earth elements. In the maintime, the (PO_4) radical can be partly replaced by small quantities of VO_4, AsO_4, SiO_4 and SO_4. Among the metal values, uranium ranges from 100 to 200 ppm in the sedimentary phosphates like that of Florida (USA) and down to only about 10 ppm in the igneous phosphates like that of Kola (Russia), while the REEs content in the sedimentary phosphates ranges from 0.1 up to about 0.5% as in Florida ore and can indeed attain up to about 1% as in the Kola ore.

Zidan (2014) has suggested that the sedimentary phosphorite rocks have been deposited in reducing environment with grey to black colour and would be changed to yellowish brown colour if subjected to geochemical weathering in a manner that the pyrite mineral would be changed to hematite and anhydrite or gypsum minerals by oxidation.

The recovery of the two interesting metal values associated with the phosphate rock, can either be recovered from the bulk acid solubilization products of the phosphate rock or else via their selective leaching. Thus, uranium can indeed be directly recovered from the wet process phosphoric acid product using the two main recovery techniques; namely solvent extraction and ion exchange. The former using octyl pyrophosphoric acid (OPPA), octyl phenyl acid phosphate (OPAP) and di (2-ethylhexyl) phosphoric acid and trioctyl phosphine oxide (DEHPA-TOPO) has actually been industrialized and 8 plants have been built and operated in the USA since 1976 **(IAEA, 1989)** in addition to those in Belgium, Canada, Taiwan and China. However, these plants have been stopped due to decreased uranium prices and too expensive production costs. Alternatively, the ion exchange recovery of U^{4+} has been suggested where in France, the MinmetRecherch process used the cationic chelating Duolite C464 resin containing the hydroxyl diphosphonic groups $R\text{-}C(OH)\text{-}(PO_3H_2)_2$ while **Ketzinel et al., (1971) and IAEA (1985)** have alsoused a cationic chelating resin with however amino phosphonic group (Duolite ES 467 resin). In this context, it has to be indicated that using H_2SO_4 acid would be difficult since most of the REEs would remain in the insoluble gypsum and their recovery from it would the difficult and uneconomic. Alternatively, in case of using nitric acid, both metal values would be co-leached with the phosphate values. However, such techniques are difficult and very expensive and several trends have tried to selectively leach the uranium and the REEs or the phosphate value itself. Thus, the selective processing of the phosphate ores, there are indeed two trends; namely a prior HCl acid leaching of the phosphate values as monocalcium phosphate in the presence of a reductant to keep uranium insoluble and upgraded in the residue in its tetravalent state **(Altair et al., 1958, Ketzinel et al., 1971 and El Shazly et al., 1972)**. In the second trend, both uranium and the REEs could first be differentially extracted leaving the phosphate values behind for their late processing. This has actually been realized for uranium by using citric acid calcium citrate mixture in a proper solid / liquid ratio by **Hussein (1980).** The latter has been based upon the fact previously observed by **Krumm in 1953** who has found that presence of calcium citrate would not allow phosphate solubilization; a matter which is due to the ability of the citrate anion to form un-ionized association compounds with calcium ions. Thus while uranium and the REEs can form stable uranium complexes with citric acid on one hand, presence of calcium citrate would hinder tricalcium phosphate solubilization. The reaction involved can be represented as follows:

$$3Ca_3(PO_4)_2 + 4\,H_3Cit \longrightarrow 3Ca(H_2PO_4)_2 + 2Ca_3Cit_2$$

$$UO_2^{2+} + HCit^{3-} \longrightarrow UO_2HCit^{-}$$

$$Ln^{3+} + HCit^{3-} \longrightarrow LnHCit$$

Finally, it has to be indicated that in the carbothermic reduction of phosphate ores, both uranium and the REEs would remain in the slag; viz:

$$2Ca_5F(PO_4)_3 + 4SiO_2 + 15C \xrightarrow{>1100^{o}C} 9CaSiO_3 + CaF_2 + 25CO + 1.5P_4$$

Although this option has successfully been tested for uranium extraction by **Guirguis in 1974** on a laboratory scale, it would actually be an energy expensive variant and would have a limited use in industry.

The present work is concerned with the recovery of both uranium and the REEs values from Abu-Tartur phosphate deposits and whose reserves are estimated to attain 1000 Mt. For this purpose, selective leaching of both metal values would be studied using the citric acid / calcium citrate mixed leachant in a proper ratio to keep the phosphate values almost intact. The obtained leach liquor would then be properly treated using the anion IX resin Amberlite IRA-400 for uranium recovery whereas the REEs would be subsequently recovered from the uranium-effluent solution using the Dowex 50W-X8 cation exchange resin. To realize these objectives, a representative sample of the weathered Abu-Tartur PR was collected and was found to assay 30 ppm U and 1058 ppm ΣREEs. The relatively low uranium assay is most probably due to weathering effects that have led to its partial leaching.

2. Materials and Methods

2.1. Material Characterization

The present work aimed to study the successive ion exchange recovery of uranium and the REEs after their selective leaching from Abu-Tartur phosphate rock (PR) using mixed citric acid ⁄ calcium citrate reagents. A representative weathered sample of this deposit was subjected to complete chemical analysis of both its major oxide constituents besides some trace elements by XRF. The former have been analyzed in the National Research Center (NRC) using Axios Advanced, Panalytical, Holland. In addition, the interesting trace elements have also been analyzed by XRF using the Philips X-unique-II Spectrometer of the Nuclear Materials Authority (NMA) which in fitted with an automatic sample changer PW 1510 (30 positions), Holland. Another sample thereof was also subjected to mineral identification in the labs of the NMA using an X-ray diffraction (XRD) of Phillips X-ray (PW3710) model with a generator (PW 1830) and Cu target tube (PW 223/20) operated at 40 KV and 30 mA.

2.2. Experimental Procedures

2.2.1. Selective Leaching of U and the REEs

Several experiments have been performed to determine the selective combined leaching conditions of U and the REEs from the studied Abu-Tartur PR using citric acid ⁄ calcium citrate mixed reagents. The studied conditions involved the effects of the mixed reagents concentration and that of the agitation time. In these experiments, 40 g sample portions, ground to -200 mesh size, of the working Abu-Tartur representative sample have all the time been used in a solid/liquid of 1/3.

2.2.2. IX Recovery of U and the REEs

To study the IX recovery of both uranium and the REEs from the studied Abu-Tartur working sample, a proper leach liquors thereof was first prepared and subjected to the necessary pre-treatment **(Hussein, 1980)** prior to resin application**.** The purpose was to eliminate the applied leaching reagents; namely the calcium citrate by evaporation for its crystallization and the citric acid via ethylation. Thus, a 250 g ore sample portion of the working PR material was subjected to the leaching of its metal values using the studied citric acid ⁄ calcium citrate reagents (0.5 M each) in a S/L ratio of 1/3 for 2 hr at room temperature. After filtration and washing, the leach liquor and washings (2L) were subjected to evaporation down to 50 ml where most of the input calcium citrate has been crystallized. After filtration of the latter and which would be recycled, the filtered citric acid solution containing the leached uranium and the REEs was then gradually ethylated until precipitation of the soluble salts. After filtration, the citric acid /alcohol mixture would be subjected to distillation for later recycle while the precipitated salts including uranium and the REEs were redissolved and adequately washed with distilled water up to a volume of 1L. Since in the latter, uranium exists as an anion, it was first recovered using the Amberlite IRA-400 anion exchange resin while the REEs were subsequently recovered using the cation exchange resin Dowex 50W-X8 resin.

2.2.2.1. IX Recovery of Uranium

A 1 ml wsr of Amberlite IRA-400 anion exchange resin was packed in a small glass column of 1.5 cm diameter and the prepared 1 L solution of U/REEs was then passed through it a flow rate of only 1 ml/ min. and the effluent samples were collected every 100 ml for uranium analysis. After uranium adsorption, the loaded resin was eluted using 1M sodium chloride solution acidified with 0.15 N H_2SO_4 at a flow rate of 1ml/min and the eluate samples were collected every 10 ml for uranium analysis.

2.2.2.2. IX Recovery of the REEs

The uranium-free effluent containing the leached REEs was then subjected to their IX recovery using the cation exchange Dowex 50W-X8 resin. To realize this recovery, 5 ml wsr of latter was packed in a glass column of 1.5 cm diameter and the uranium effluent solution was then passed through at a flow rate of 1ml/min and the effluent samples thereof were collected every100 ml for REEs analysis. The loaded resin column was then eluted using 4 M HCl acid, at a flow rate of 1ml/min and the eluate samples were collected every 10 ml for REEs analysis.

2.3. Analytical Control Procedures

2.3.1. U Control Analysis

For uranium analysis in the different working experimental stream solutions, the oxidimetric titration method using ammonium metavanadate has been applied. For this purpose, a prior uranium reduction was performed by $(NH_4)_2Fe(SO_4)_2$ in the presence of diphenylamine sulfonate indicator until it changes to a slightly violet colour**(Davies and Gray, 1964)**.

U (g/l) = T.V1 / V. 1000 where V1: volume taken of NH_4VO_3 solution (ml)
V: sample volume (ml) and T: concentration of NH_4VO_3 to U (g/ml)

2.3.2. REEs Control Analysis

For the REEs analysis in the different working experimental stream solutions, the REEs content was spectrophotometically determined by Arsenazo III as a complexing agent and the absorbance was measured at 650 nm against proper standard solutions **(Marezenko, 1986)** using a Lambada UV/VIS spectrophotometer (Perkin-Elmer, USA).

3. Result and Discussion

3.1. Material Characteristics

3.1.1. Mineral composition

The obtained X-ray diffraction pattern of the study sample of Abu- Tartur phosphate rock sample and which is shown in Fig. (1) indicates that the its mineral composition involves the apatite mineral dahlite $(Ca,Mg,Na,H_3)_5(P,C)_3O_{12}(OH,Cl,F)$ which represents the main component. The latter is characterized by the main diffraction lines 2.81, 2.73, 3.46 and 1.94 Å. On the other hand, the associated gangue minerals include mainly gypsum ($CaSO_4$) with diffraction lines 7.56, 3,06, 4.27 and 2.68 Å and the quartz mineral (SiO_2) of diffraction lines 3.34, 4.26, 1.82 and 1.54 Å (Table 1).

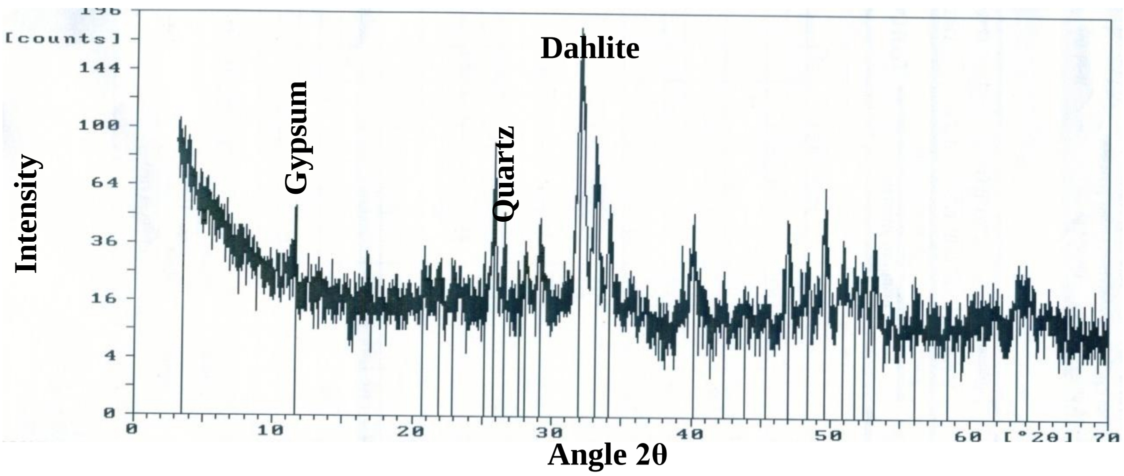

Fig. (1): X-ray diffraction pattern of the original Abu-Tartur PR sample

Table (1): The characteristic diffraction lines of the constituent minerals in the working Abu-TarturPR sample

Mineral	Characteristic lines (Å)				ASTM card
Dahlite	2.81	2.73	3.46	1.94	12-0529
Gypsum	7.56	3.06	4.27	2.68	6-0046
Quartz	3.34	4.26	1.82	1.54	5-0490

3.1.2. Chemical Composition

The major chemical composition of the representative working sample of Abu-Tartur PR is summarized in Table (2). From the latter, it is shown that P_2O_5 and CaO assay 21.82 and 45.61% respectively together with iron oxides and sulfur trioxide contents of up to 8.08 and 3.88% respectively. It is also interesting to refer to the relatively high percent of the loss of ignition (9.20%). On the other hand, the associated SiO_2 and Al_2O_3 are relatively low; viz, 5.89 and 1.72% respectively. In the meantine, it was found that the F assay is quite low attaining only 0.76%.

Table (2): Chemical composition of the working Abu-Tartur PR sample

Component	Wt. %
SiO_2	5.89
Al_2O_3	1.72
Fe_2O_3	8.08
MgO	1.01
CaO	45.61
Na_2O	0.58
K_2O	0.16
TiO_2	0.29
P_2O_5	21.82
MnO	0.34
F	0.76
Cl	0.17
SO_3	3.88
*L.O.I	9.20
Total	**99.51**

*L.O.I: Loss of ignition

To visualize the possible mineral composition, it was found interesting to subject the obtained chemical analysis to the calculation of the potential mineral composition (mode). To analysis this objective, the mineral formulae of apatite and clay were considered as equivalent to $Ca_{10}(PO_4)_6F_2$ and $Al_2O_3.2SiO_2.2H_2O$ respectively. Accordingly, the potential mode is shown in Table (3).

Table (3): Potential mineral composition of the working Abu-Tartur PR sample

Component	Wt.%	Component	Wt.%
Apatite	49.20	Iron oxides	8.08
Calcite	21.29	Clay	4.30
Dolomite	4.64	Quartz	4.49
Gypsum	8.35		
		Total	**100.35**

On the other hand, the interesting trace elements have also been analyzed together with the present rare earth elements. From the obtained results shown in Table (4), it was found that while V assays 103 ppm, Sr is highly enriched where it attains 1495 ppm. Also, while Cr, Co and Ni assay together about 170 ppm, Cu, Zn and As assay together 137 ppm. In the meantine, while U and Th assay only 30 and 18 ppm respectively, the REEs assay 1058 ppm. In the latter, the LREEs assay 756 ppm and whereas Yb assays only 18 ppm, Y attains up to 284 ppm. Finally, it might be interesting to add herein that Sc assay attains 76 ppm.

Table (4): Interesting trace elements analysis in the working Abu-Tartur PR sample

Element	ppm	Element	ppm
		U	30
Sc	76	Th	18
V	103		
Cr	77	La	201
Co	54	Ce	337
Ni	40	Nd	176
Cu	24	Sm	42
Zn	72	Yb	19
As	41	Y	283
Cd	2		
Sr	1495	**Total REEs**	**1058**

3.2. Optimization of the Selective Leaching Conditions

3.2.1. Effect of Citric Acid /Calium Citrate Molarity

To study the effect of the mixed selective leaching reagents of citric acid / calcium citrate upon the leaching efficiencies of both U and the REEs, 4 leaching experiments have been performed at room temperature using different concentrations thereof. While the latter was ranged from 0.05 to 0.5 M for each, the leaching time was fixed at only half an hour and the S/L ratio was fixed at 1/3. From the obtained results shown in Table (5), it was found that by increasing the molarity of the selective leaching reagents, the leaching efficiency of both U and the REEs has progressively been increased. Thus, while the uranium leaching efficiency has varied between about 50% and complete leaching, the leaching efficiency of the REEs has ranged between 30.0 and 84.3% at the input reagent concentrations of 0.05 and 0.5 M respectively. On the other hand, it is quite interesting to mention that the leached phosphate value is quite low and has even decreased from 0.34 down to only 0.10% at the input mixed reagents molarity of 0.05 and 0.5 M respectively.

Table (5): Selective leaching efficiency of U and the REEs from the working Abu-Tartur PR sample

1:1 Citric acid /Ca-citrate, M	Leaching Efficiency, %		
	U	REEs	P_2O_5
0.05	49.7	30.0	0.34
0.125	66.6	39.8	0.28
0.25	72.3	60.5	0.28
0.50	100.0	84.3	0.10

3.2.2. Effect of the Leaching Time

Having obtained a complete uranium leaching efficiency and up to 84.3% REEs leaching efficiency in a leaching time of only 0.5 hr, it was found that necessary to increase the latter to 2 hr. Thus, two more leaching experiments were performed to study the effect of extended leaching time upon the leaching efficiency of the rare earth elements. The former have been achieved at a leaching time of 1.5 and 2 hr using the obtained optimum concentration of the leaching reagent of 0.5 M for each in a S/L ratio of 1/3. The obtained results have indicated that the leaching efficiency of the REEs has increased to 90.8 and completion at 1.5 and 2 hr respectively. In the meantime, the leached P_2O_5 has only slightly increased to only 0.28 and 0.30 % respectively.

3.3. IX Recovery of U and the REEs

3.3.1. Pre-Treatment of the U and REEs Leach Liquor

To achieve the IX recovery of the studied two metal values from the citric acid/ calcium citrate leach liquor of Abu-Tartur PR, a proper 2 L leach liquor thereof was prepared using 250g ore sample that has been leached under the determined optimum leaching conditions. The unleached phosphate component of the ore was filtered and properly washed and the obtained leach liquor and the washings were made to 2 L volume and has thus was found to assay 3.75 and 133 ppm for uranium and REEs respectively while the assay of the associated P_2O_5 was quite negligible (0.8 ppm). To separate the leaching reagents for recycle, the pretreatment procedure adopted by **Hussein (1980)** for their successive separation of calcium citrate and citric acid has mostly been applied herein. Thus, the calcium citrate component of the prepared leach liquor was allowed to an evaporation step down to only 50 ml whereupon most of the calcium citrate has been crystallized while the U and the REEs assay has increased to 150 and 5320 ppm respectively. After filtration of the former, the obtained filtrate is mainly composed of citric acid solution-in which uranium and REEs are soluble as their citrate complexes-was then treated by ethyl alcohol to precipitate both metal values and which after filtration was found to weigh about 1.5 g and have some metal impurities e.g. Cr and Sr. The latter was then dissolved in 1L of distilled water and would thus assay 7.5 and 265 ppm of U and REEs respectively and was thus directed to successive IX treatment; namely anionic for U and cationic for the REEs. On the other hand, the filtrate composed of a mixed solution of ethyl alcohol and citric acid would then be subjected to proper distillation for their separation and recycle.

3.3.2. IX Recovery of U

3.3.2.1. U Adsorption

The prepared precipitate containing both the uranyl and the RE citrates (1.5 g) which was dissolved in 1 liter of distilled water was then subjected to uranium recovery using the anion exchange resin Amberlite IRA-400. However, before uranium adsorption, the pH of the prepared leach liquor was adjusted at 1.7 followed by passing through the prepared resin column containing 1 ml of Amberlite IRA-400 wsr using a flow rate of 1 ml/min. The obtained effluent was collected every 100 ml for uranium analysis and the obtained results are tabulated in Table (6). From the latter, it is clearly evident that uranium breakthrough has rapidly occurred in the 6th sample; a matter which is due to both the high flow rate and the low resin height in the column. In the 10th effluent sample, uranium assay has attained 5 ppm and proper calculation of the loaded uranium has indicated that 6 mgU has been loaded.

Table (6): Uranium adsorption results upon Amberlite IRA- 400 anion exchange resin

Effluent Sample No. (100 ml)	U Conc., mg/L	Loaded U, mg
1-5	Nil	3.75
6	1.0	0.65
7	2.0	0.55
8	3.0	0.45
9	4.0	0.35
10	5.0	0.25
Total adsorbed U		6.00

3.3.2.2. U Elution

After washing the loaded resin sample by distilled water, the adsorbed U has been eluted using 1M sodium chloride solution acidified with 0.15N H_2SO_4 at a flow rate of 1 ml/min. The obtained eluate solutions were collected every 10 ml for uranium analysis (Table 7) and which exhibits the famous bell-shaped concentration curve with the major peak at the 5th eluate sample (116 mg U/ L).The total eluted uranium has attained 5.98mg U indicating an almost complete elution.

Table (7): Uranium elution results of the loaded Amberlite IRA-400 resin column

Eluate Sample No. (10ml)	U Conc., mg/L	Eluted U, mg
1	39.1	0.39
2	49.0	0.49
3	59.4	0.59
4	99.0	0.99
5	116.0	1.16
6	98.0	0.98
7	58.1	0.58
8	37.4	0.37
9	27.3	0.27
10	16.6	0.16
Total eluted U		5.98

3.3.3. IX Recovery of REEs

3.3.3.1. REEs Adsorption

The uranium effluent from the anion resin has then been subjected to the prepared cation exchange Dowex 50W-X8 resin for REEs recovery using the prepared 5 ml wsr volume packed in 1.5 cm diameter glass column at a flow rate of about 0.5 ml ⁄ min. The effluent samples collected every 100 ml were then analyzed for their REEs content and the obtained results are shown in Table (8). From the latter, it was found that the adsorbed REEs has attained 244 mg. Similar to uranium adsorption, the early breakthrough in the 6 th the effluent sample is due to both high flow rate and the low resin height in the column.

Table (8): REEs adsorption results upon Dowex 50W- X8 cation exchange resin

Effluent Sample No. (100 ml)	REEs Conc., mg/L	Loaded REEs, mg
1-4	Nil	106.0
5	10.0	25.5
6	20.0	24.5
7	30.0	23.5
8	40.0	22.5
9	50.0	21.5
10	60.0	20.5
Total adsorbed REEs		244.0

3.3.3.2. REEs Elution

Before REEs elution, the REEs-loaded resin column was washed with 2 M HCl to remove any adsorbed possible impurities. From the obtained elution data shown in Table (9) using 4 M HCl at a flow rate of 1 ml/ min, it is evident that the latter exhibits the famous bell-shaped concentration curve with the major peak at the 5th eluate sample, which assayed up to about 8.20 g REEs/L. Proper calculation of the eluted REEs in the obtained eluate samples hasindicated a total of 243 mg REEs i.e. an almost complete elution.

Table (9): REEs elution results of the loaded Dowex 50W-X8 resin column

Eluate Sample No. (10ml)	REEs Conc. mg/L	Eluted REEs, mg
1	900	9
2	1100	11
3	2100	21
4	3000	30
5	8200	82
6	3100	31
7	2300	23
8	1600	16
9	1200	12
10	800	8
Total eluted REEs		243

4. Conclusion

From the above studied leaching behavior for uranium and the REEs from the studied weathered sample of Abu-Tartur phosphate rock, it can be concluded that the optimum conditions that would result in complete leaching of both these two metal values include:

Citric acid /Ca-citrate concentration:	0. 5M (each),
Agitation time:	2h
S/L ratio:	1/3
Temperature:	Room temperature

The obtained leach liquor has successfully been treated for the recovery of both the leaching reagents for their recycle and during which, the leached values were precipitated followed by their water dissolution. From the latter, both uranium and the REEs have then been successfully recovered via ion exchange resins. Thus, Amberlite-IRA anion exchange resin was used for uranium recovery while recovery of the REEs from the uranium-free effluent was achieved using the Dowex 50W-X8 cation exchange resin. The obtained results have indeed been translated in a generalized flow sheet showing clearly the successive required unit operations (Fig. 2).

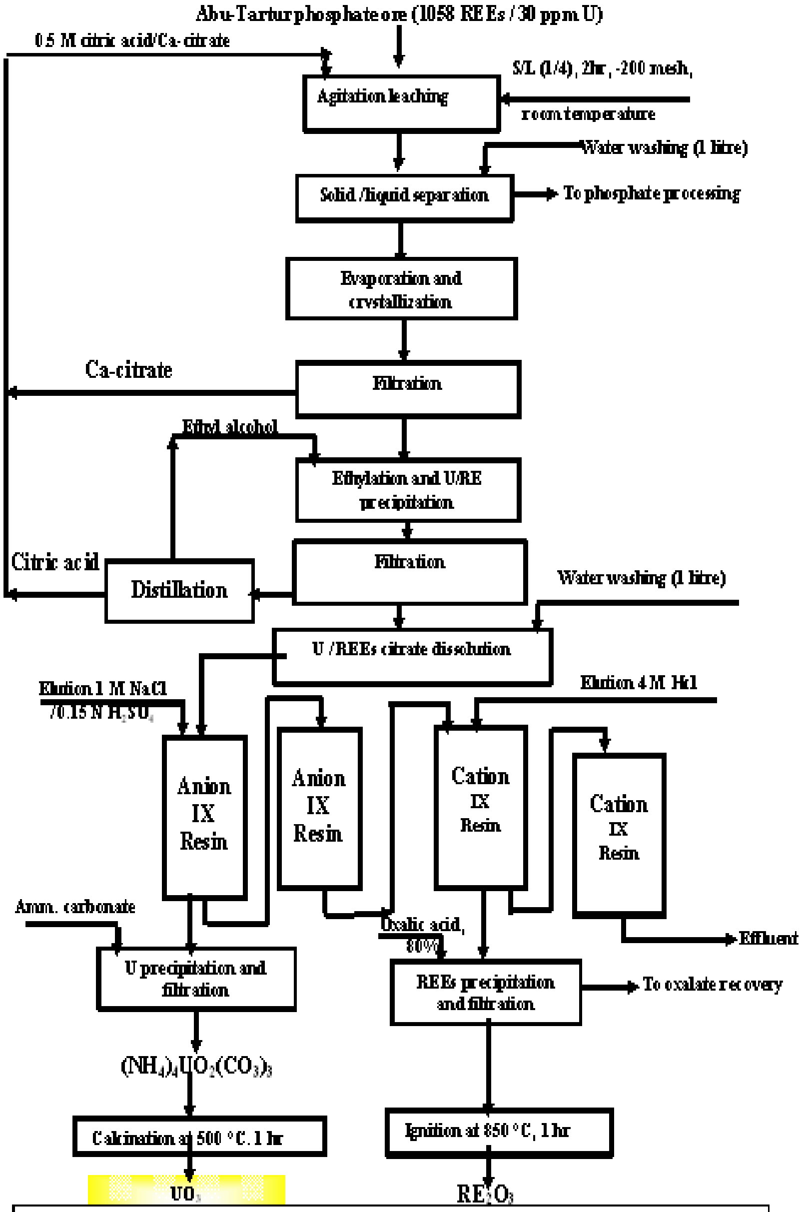

Fig. (2): Generalized technical flowsheet for the recovery of uranium and the REEs from Abu-Tartur PR

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