El Nahas et al. The Arab Journal of Sciences & Research Publishing, Vol. 2 - Issue (3): 2016, 4, 21 P. 36-50; Article no: AJSRP/ N31116·

Mineralogical Investigation and Hydrometallurgical Characteristics of Uranium and Rare Earth Elements, Mineralization of El Hammamat Sediments, G. Gattar, Eastern Desert - Egypt

El Nahas, H.A., Abd-El fattah, N. A.* and El Monsif, M. Z
Nuclear Materials Authority, P.O. Box 530, El –Maadi, Cairo
*corresponding Email: nasraziz@ymail.com

Abstract

The uranium occurrence of GV is considered as perigranitic uranium deposits while all the others are considered as intragranitic ones. The principal economic minerals were separated and identified. Uranophane was found to represent the main uranium mineral while the REEs occur in the form of very fine disseminated grains of light elements or else in the form of heavy elements associated with the iron oxides and clay fraction of the host rock. For geochemical and hydrometallurgical works, a technological sample of (4000ppm U and 3500ppm REE) at the Northern part of the Eastern Desert, Hammamat GV mineralization of G. Gattar was collected. The collected ore material was then subjected to chemical processing involving sulphuric acid leaching and the dissolved U and REE were separated from the obtained leach liquor. Two alternatives have been studied, namely ion exchange for U following oxalate precipitation for the REEs or else selective successive precipitation.

Key Words: *El Hammamat, Uranophane, Leaching, Ion exchange, Precipitation.*

I. Introduction

Several uranium occurrences are recognized associated with G. Gattar younger granite pluton (alkali feldspar granite) in the Northern Eastern Desert of Egypt. The alkali feldspar granites are characterized by pink to red color sand range in size from medium to coarse grained. All these occurrence are situated inside the granite pluton in different shear zones except that of GV which is associated with the Hammamat sediments that are located in the northern periphery of G. Gattar at the intersection of latitude 27^{o} $07^{\backslash}$ $18^{\backslash\backslash}$N and longitude 33^{o} $17^{\backslash}$$42^{\backslash\backslash}$ E (Fig. 1). In other words, the uranium occurrence of GV is considered as perigranitic uranium deposits while all the others are considered as intragranitic ones. The two rock facies are traversed by W. Belih, which is filled with stream sediments. The Hammamat sedimentary rocks are well exposed at G. Um Tawat to the north of G. Gattar and range in color from light grey to dark grey and mainly comprise greywackes and siltstones. The greywackes, up to 35m thick, are highly foliated, laminated and display graded bedding ranging from pebbly greywacks at the bottom to fine-grained greywackes at the top. The siltstones in the studied area occur as thin layers especially along the contact with the alkali feldspar granites of G. Gattar. They are fine-grained and intercalated with the greywackes.

According to **Willis et al. (1988)** the Hammamat sedimentary rocks of G. Um Tawat were subjected to a low-grade metamorphic event (~530Ma; that seems to represent a lateral manifestation of a final pulse of alkaline magmatism concentrated in northernmost Egypt **(Stern and Hedge, 1985)**. Many alteration features were recognized along the contact of the Hammamat sedimentary rocks with the alkali feldspar granites, comprising strong hematitization, bleaching, fluoritization, carbonitization, koalinitization with some manganese dendrites **(Abdel Hamid, 2006)**. These alteration features may be due to the effect of hydrothermal solutions. The Hammamat sedimentary rocks are dissected by dyke swarms of acid and basic composition in addition to some quartz veins. **Shalaby (1990)** pointed out that the Hammamat sedimentary rocks are thrown down against the granite along a local reverse fault trending ENE-WSW and dipping from 45^{O} to 65^{O} SSE towards the granite. Along the sharp intrusive contacts with the adjacent Hammamat sedimentary rocks, the alkali feldspar granites also show variable degrees of alteration e.g. episyenitization (desilisification), hematitization, chloritization and silicification. In addition, some apophyses of alkali feldspar granites invaded the Hammamat sedimentary rocks.

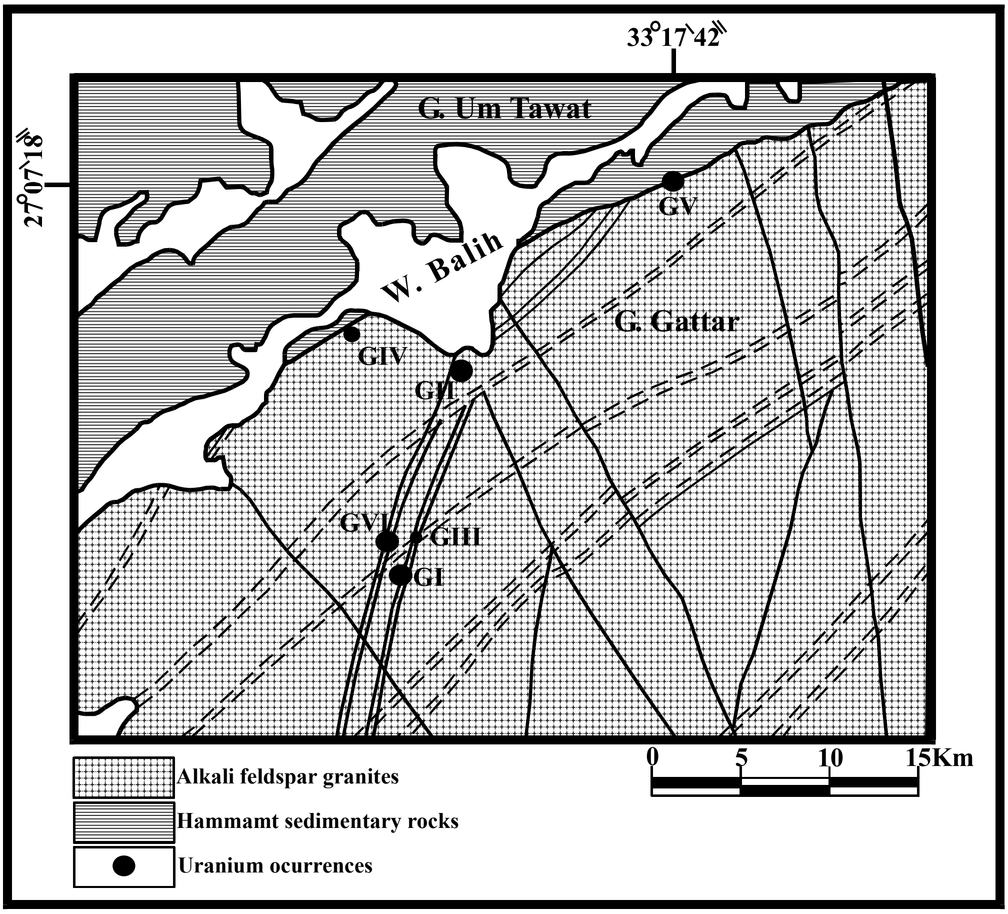


Figure (1): Geologic map of GV uranium occurrence in G. Gattar **(after Abu zaid, 1999)**

As a matter of fact, most of the uranium mineralizations at GV are restricted to the Hammamat sedimentary rocks especially along the contact between the two rock types exposed at the area. The alkali feldspar granites of G. Gattar are considered as the source of these mineralizations. According to **Shalaby (1990)**, the hypogene and supergene mineralizing fluids have percolated throughout the joints and fractures of the fertile alkali feldspar granites and released some of their uranium content then re-deposited their load in the fractures of the adjacent Hammamat sedimentary rocks.

Few hydrometallurgical works have been concerned with the recovery of both the REEs and U values from the mineralized Hammamat sediments at GV occurrence. These include mainly. **Mahdy et al (1990)** have studied the leaching characteristics of REE and U from the Hammamat Sediments of GV occurrence by different acids as well as by alkaline carbonate using both agitation and percolation techniques. In the former, the fixed conditions included 4 hr. agitation at room temperature and a solid - liquid ratio of 1/2. It was found that, while U is almost completely extracted by acid leaching using 100g/l acid, only 50% of the U was leached by 100 g/l Na_2CO_3. In percolation leaching using glass columns, 1 and 5g H_2SO_4/l, were used for 3 weeks or 1g/l of Na_2CO_3 or $NaHCO_3$ or else 0.5 g/l of each as well as 10 g/l Na_2CO_3. The obtained result indicated that acid leaching gave complete extraction after 4 to 5 days while 10g/l Na_2CO_3 resulted in 55% leaching in about 2 weeks and 10-20% leaching efficiency in the same period for the other alkaline reagents concentration.

El Hazek, (1995) has also undertaken a detailed leaching and recovery studies of U from a technological sample form the Hammamat sediments of Wadi Belih assaying up to 1.38% U and only 100ppm REE (Ce, Er, Nd, Sm and Yb). Leaching was performed by H_2SO_4 and Na_2CO_3 / $NaHCO_3$ besides NaCl and $FeCl_3$. Acid leaching indicated that 95% of U was leached by only 25 g/l acid for 4 hr at 50°C in a S/L ratio of 1/2 while alkaline leaching resulted in 85 and 91% leaching efficiencies at 90°C for 4 and 24 hr respectively. Leaching by NaCl necessitated addition of HCl whereas $FeCl_3$ solution was able to leach about 88% of the U content.

The present work is concerned with the definition of the mineralogical characteristics of Gattar GV mineralization as well as with its chemical processing for the recovery of both the U and RE metal values. For this purpose, heavy liquid separation has been performed followed by magnetic separation of the heavy fractions to separate the mineral grains for their analysis. In the hydrometallurgical processing, sulphuric acid leaching has been achieved for both U and REE while their separation was successfully studied by two alternatives. The first alternative has involved a proper separation of U by ion exchange and from the obtained effluent the REE has been precipitated as their oxalates. In the second alternative, the two metal values have been obtained by proper successive precipitation.

II. Experimental

II.1.Material Characterization

A technological sample representing the mineralized GV occurrence and assaying 0.4% U and 0.35% ∑REEs was first subjected to complete chemical analysis of the major oxides as well as for some interesting trace elements besides the tenor of the economic metal values. For this purpose, about 50 g sample weight was separated by quartering from the finely crushed representative sample before being ground down to – 150 mesh size. The pulverized sample was then analyzed using conventional wet chemical techniques **(Shapiro and Bannock, 1962)** Where as SiO_2, Al_2O_3, TiO_2 and P_2O_5 were determined using spectrophotometric methods, both Na and K were determined by the flame photometric technique. In these analyses, the estimated error for major constituents is not more than ±1%.On the other hand, the analyzed trace elements including Cr, Ni, Cu, Mo, V and Pb were estimated by the atomic adsorption technique using Unicam 969 model spectrometer fitted with auto gas box

Uranium was analyzed volumetrically where ammonium metavanadate was applied, after a previous uranium reduction by ammonium ferrous sulfate, in the presence of diphenylsulfonate indicator until its color end point changes to slightly violet red color **(Florence, 1970)**. On the other hand ∑REEs were analyzed spectrophotometrically using arsenazo (III) **(Marcezenko, 1986)**.

II.2. mineralogical Investigation

Five channel samples of the Hammamat sedimentary rocks were collected from GV uranium occurrence for mineralogical investigation. These samples were subjected to a proper comminution process to liberate the minerals from each other followed by quartering using both the John's splitter and the automatic rotary splitter to obtain representative samples. Each sample was subjected to the heavy liquid separation using bromoform (Sp. gr.2.81g/cm^3). The obtained heavy fractions were then subjected to magnetic separation using the Frantz Isodynamic Magnetic Separator (Model L1) using 0.2. 0.5, 1.0 and 1.5 current amperes. Each fraction obtained from the magnetic separation was mineralogically investigated using the Binocular Stereomicroscope, the Scanning Electron Microscope (SEM) and the X-ray Diffraction technique (XRD). From this study, several economic mineral have been identified and the most important of which comprise uranophane, beta uranophane, fluorite, fluorapatite, REE mineral, pyrite and barite

II.3. Experimental Leaching Procedure

The study of the leaching characteristics of both U and REEs from El Hammamat sedimentary GV mineralization was performed using sulfuric acid agitation leaching technique. The main relevant acid leaching conditions include the acid concentration, solid / liquid ratio, leaching time. At the end of each leaching experiment, the ore slurry was filtered, washed and completed up to volume for U and REE analysis.

III. Results and Discussions

III.1.Analytical Results of the Ore Material

Results of the average major chemical composition of the working El Hammamat technological sample are given in Table (1). From the latter, it is clearly evident that the ore is mainly composed of SiO_2, Al_2O_3 and, Fe_2O_3 and to a much lesser extent of TiO_2, MgO, CaO, Na_2O, K_2O and P_2O_5.The silica content attains 63.32% while Al_2O_3 and Fe_2O_3 assayed 14.45% and 7.94% respectively, besides CaO and MgO contents of 1.6% and 0.83% respectively. The economic trace and rare earth elements in the working sample are given in Table (2) which shows that both the REEs and U represent adequately economic interesting values; viz 4000 and 3500ppm respectively.

Table (1): Chemical composition of the working GV technological sample of El Hammamat sediments

Component	wt %
SiO_2	63.32
Al_20_3	14.45
Fe_2O_3	7.94
CaO	1.61
MgO	0.83
Na_2O	0.61
K_2O	3.30
P_2O_5	0.43
MnO	0.1
Loss of ignition	5.4
Total	99.8

Table (2): Analytical assay of U and the REEs together with the other associated trace elements in the working GV technological sample of El Hammamat sediments

Trace element	Concentration	Trace element	Concentration
U	4000	Cu	240
REE	3500	Pb	296
Cr	80	Ni	157
Mo	64	V	600

III.2. Mineralogical Investigation of GV Occurrence

As mentioned above, mineral separation techniques have been applied upon S channel samples of GV mineralization at El-Hammamat sediments and several economic and accessory minerals have actually been separated. Microscopic investigations of the separated mineral grains besides their SEM images and EDX analysis have resulted in the identification of the following mineral species.

III.2.1.Uranophane [Ca (UO3)2 (SiO3)2 (OH)2 .5H2O]

It is a monoclinic uranyl silicate mineral fairly common in the oxidized zone of most uranium deposits **(Cebron, et al., 1993)**. Uranophane represents the most abundant radioactive minerals in GV uranium occurrence of G. Gattar and exhibits different grades of lemon yellow color and greasy luster (Fig. 2a). Some grains are tarnished with brown color due to staining of iron oxides. It mostly occurs as divergent clusters of prismatic to acicular grains, but few grains occur as anhedral massive crystals.

X-ray diffraction analyses are in accordance with ASTM card (8-442) of uranophane with the presence of diffraction lines of beta-uranophane (ASTM card 8-301). Uranophane is usually associated with the latter which represents indeed its dimorph, **(Gorman and Nuffield, 1955)**. SEM -EDX analyses of some uranophane mineral grains reveal that the mineral is composed essentially of U (about 80%), Ca and Si, with traces of Al, K and Fe (Fig. 3a).

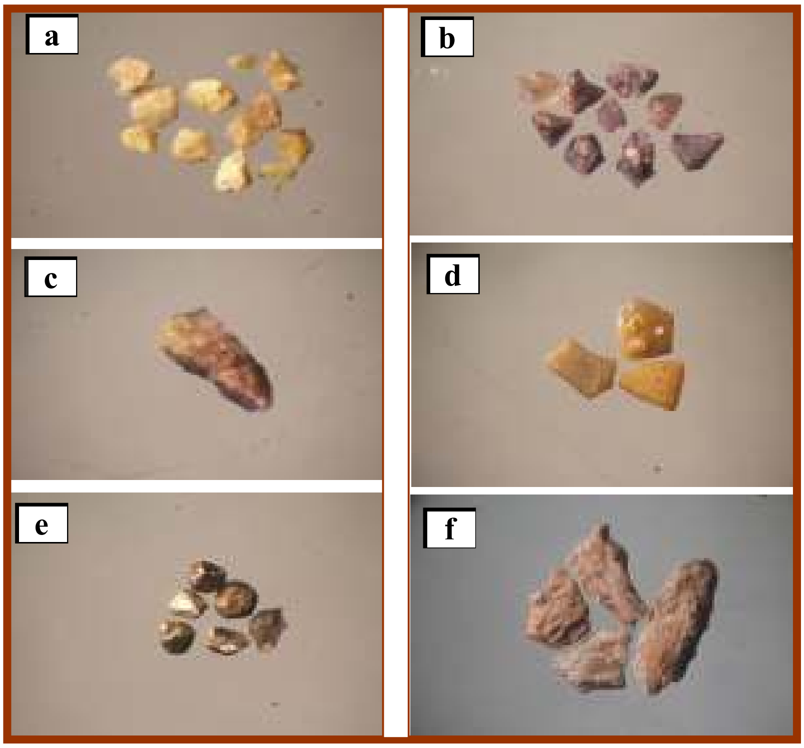
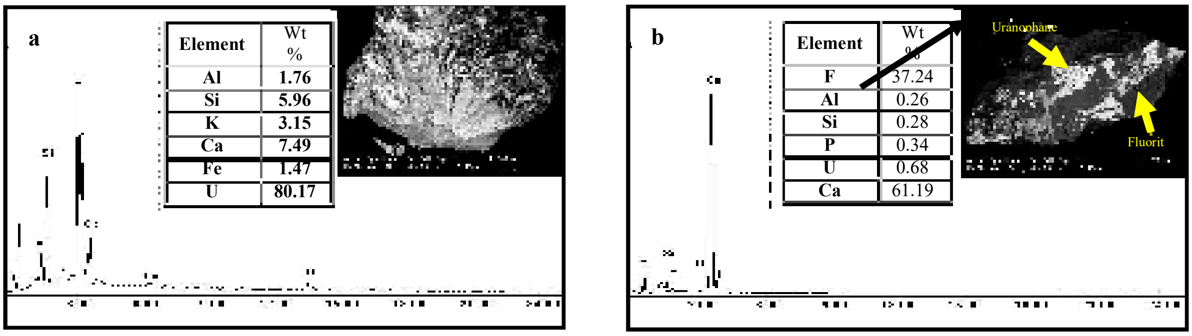


Figure (2): Photomicrographs showing the common heavy of mineral grains in the studied Hammamat sedimentary rocks; a) uranophane, b) fluorite, c) defect structure in fluorite, d) fluoapatite, e) pyrite, f) coexisting barite, wurtizite and calcite.

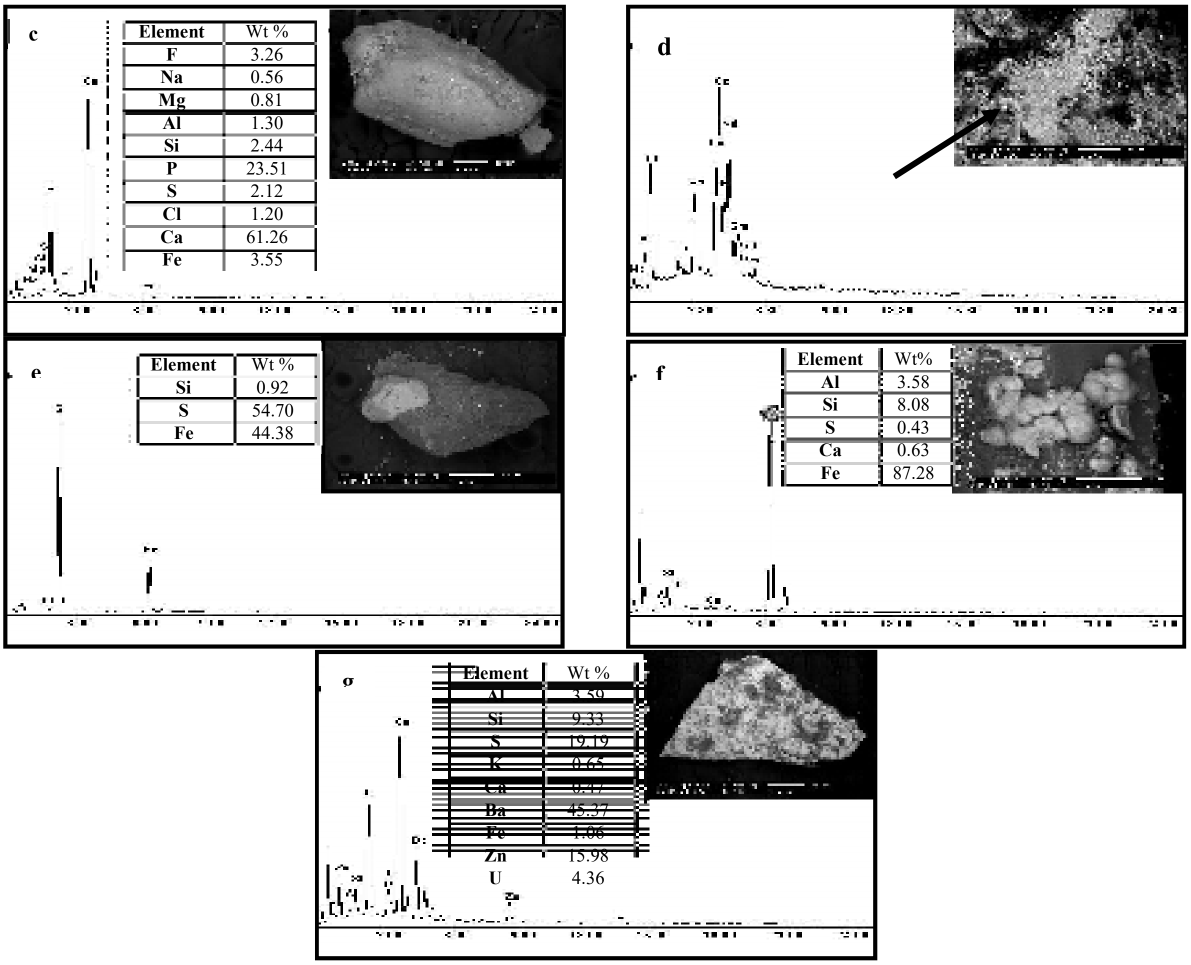
III.2.2 Fluorite [CaF2]

Fluorite commonly occurs in hydrothermal, pegmatitic and pneumatoytic veins, in greisens and in cavities in granites. In the studied Hammamat sedimentary rocks of GV, fluorite occurs as anhedral angular to subangular irregular fragments of vitreous luster. The color of this mineral range from pale rose, purple, violet, blue to deep blue colors (Fig. 2b). The coloration of fluorite may be due to the presence of radioactive materials in or around the fluorite mineral grains. Some fluorite mineral grains exhibit defect structure (Fig. 2c). **Raslan (1996)** concluded that rocks which are rich in secondary uranium minerals are usually rich in fluorite of deep blue to violet colors. From its EDX analysis the mineral is composed essentially of Ca and F with the presence of trace amounts of U, Si, P and Al. In addition some fluorite grains contain minute inclusions of uranophane (Fig. 3b).



Element	Wt %
Al	1.76
Si	5.96
K	3.15
Ca	7.49
Fe	1.47
U	80.17

Element	Wt %
F	37.24
Al	0.26
Si	0.28
P	0.34
U	0.68
Ca	61.19



Element	Wt %
F	3.26
Na	0.56
Mg	0.81
Al	1.30
Si	2.44
P	23.51
S	2.12
Cl	1.20
Ca	61.26
Fe	3.55

Element	Wt %
Si	0.92
S	54.70
Fe	44.38

Element	Wt%
Al	3.58
Si	8.08
S	0.43
Ca	0.63
Fe	87.28

Element	Wt %
Al	3.59
Si	9.33
S	19.19
K	0.65
Ca	0.47
Ba	45.37
Fe	1.06
Zn	15.98
U	4.36

Figure (3): EDX analysis and backscattered SEM images of the identified minerals separated from GV occurrence of el Hammamat sedimentary rocks; a) uranophane, b) fluorite with inclusions of uranophane, c) fluorapatite, d) very fine grains of a REE mineral e) euhedral pyrite in feldspar f) frambiodal texture of pyrite, g) particle grains of barite and wurtizite

III.2.3. Fluorapatite [Ca(PO4)3F]

Because of its large partition coefficients of REE, Th, U, Ba and Sr, apatite is considered as a major carrier of such elements **(Liu, et al., 2005)**. Fluorapatite in the studied GV occurrence is present in different forms; viz elongated blades, rod-like or massive granules, with rough surfaces. It displays smoky yellowish white to grayish green colors and vitreous luster (Fig. 2d). EDX mineral analysis of some Fluorapatite grains reveals that the mineral is composed mainly of Ca and P besides trace amounts of Fe, Cl, Al, Si, S, Mg and Na (Figure 3c).

III.2.4. REE-bearing mineral

The REE-bearing mineral has been detected by SEM-EDX analysis as disseminated very fine grains within quartz and feldspar of the Hammamat sedimentary rocks. This mineral may be formed through the interaction of the REE carrying hydrothermal solution with the rock constituents causing the absorption of the REE in these minerals through fractures. As shown in figure (3d) the REE-bearing, the mineral consists mainly of LREE, Ca and Si with minor amounts of Th and U. The most common REEs in this mineral are involve Ce followed by La and Pr.

III.2.5 Pyrite [FS2]

Pyrite is rare in the studied GV occurrence of El Hammamat sedimentary rocks where it occurs as euhedral to subhedral grains of golden black color and metallic to submetallic luster (Fig. 2e). SEM-EDX analysis indicates that Fe and S are the main constituents of the mineral with trace amounts of Si (Fig. 3e). Few pyrite grains exhibit frambiodal texture (Fig. 3f). Pyrite has most probably been formed as a result of microbiological processes in an anaerobic environment **(Love, 1964; Boctor, 1966)**.

III.2.6 Barite [BaSO4]

In the studied samples of GV occurrence of El Hammamt sedimentary rock, barite generally coexists with wurtizite ($ZnSO_4$) and calcite ($CaCO_3$) within some mineral particles of clayey appearance. These coexisting minerals are characterized by grayish white to bluish colors and are of earthy luster (Fig. 2f). The corresponding XRD data indicate the coexistence of these minerals; [barite (ASTM card no.5-448), wurtizite (ASTM card no.12-688) and calcite (ASTM card no.5-586)]. While the SEM analyses confirm; the presence of these minerals in the studied Hammamat sedimentary rocks (Fig. 3g).

II.3 Hydrometallurgy of U and REE from GV Occurrence

For studying the hydrometallurgical and characteristic of U and the associated REEs in the studied GV occurrence of El Hammamat sediments, the acid leaching agitation technique was first applied using sulfuric acid. For this purpose, the relevant leaching conditions have actually been studied to optimize their proper value. To recover uranium from its leach liquors, there are three major techniques that can be applied; ion exchange resins, solvent extraction and direct precipitation. The use of the suitable method for uranium recovery depends primarily on its concentration in the leach liquors. A combination of more than one recovery technique such as ion exchange and solvent extraction could also be applied **(Tunley and Nel, 1974).**

III.3.1 Optimization of the Relevant leaching Factors

III.3.1.1 Effect of acid concentration

Several acid leaching experiments have been performed using mainly sulfuric acid at different concentrations (50, 100,150,200 g/l) in a solid /liquid ratio of 1:2 at room temperature for 2h upon ore sample ground to -150 mesh size. The obtained results shown in Fig (4) indicated that the leaching efficiency of the REEs attained 52%, 67%, 75% and 80%, while that of uranium it were 45%, 60%, 67% and 77%, respectively. These results indicate that the best concentration acid is 200 g/l for both the REEs (80%) and uranium (77%). Increasing the leaching efficiency for uranium and REEs behind the latter the acid concentrations above 200 g/l dose not seem to results in any perceptible increase. Therefore work was oriented towards increasing the solid liquid ratio at 200 g/L.

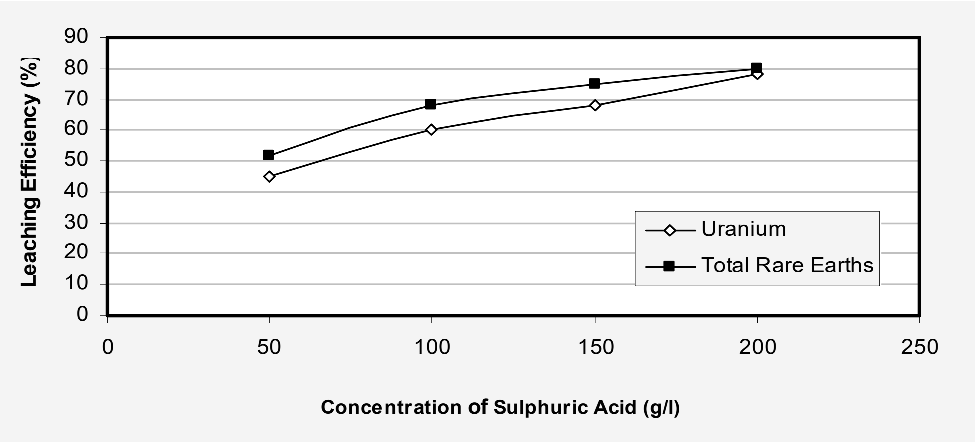


Figure (4): Effect of acid concentration on the leaching efficiency of U and REE

III.3.1.2 Effect of the solid / liquid ratio

The work was then oriented towards studying the effect of the solid / liquid ratios 1:2, 1:3, 1:4.During this study the other leaching conditions were fixed at 200 g/L H_2SO_4, a stirring time of 2 hours, and ore material ground to a grain size of -150 mesh, at room temperature. The obtained leaching efficiencies of REE and uranium under these conditions are plotted in fig (5).

From the obtained data it was indicated that a suitable solid/ liquid ratio 1:4 was found optimum for both the leaching efficiencies REEs and U, which increased to 98.9% and 97% respectively. This may be attributed to the presence of some acid consuming rock constituent.

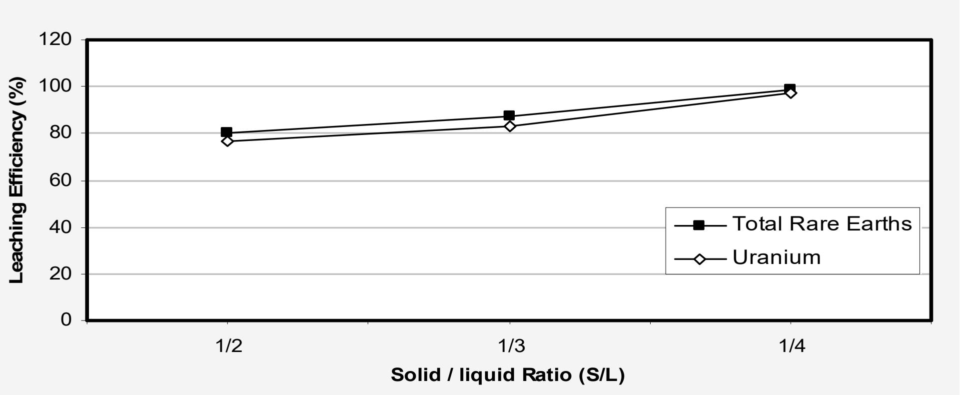


Figure (5): Effect of S/L ratio on the leaching efficiencies of U and REE

III.3.1.3 Effect of leaching time

In a trial to decrease the leaching time below 2 hrs at the studied leaching condition, two leaching experiments have been performed for 1/2 and 1 hr to be compared with that at 2 hr. In these two experiments, the other factors were fixed at a S/L ratio 1/4, 200 g/l sulfuric acid with -150 mesh grain size at room temperature. The obtained leaching efficiencies of both U and REE are plotted in fig. (6) in comparison with that performed at 2 hrs.

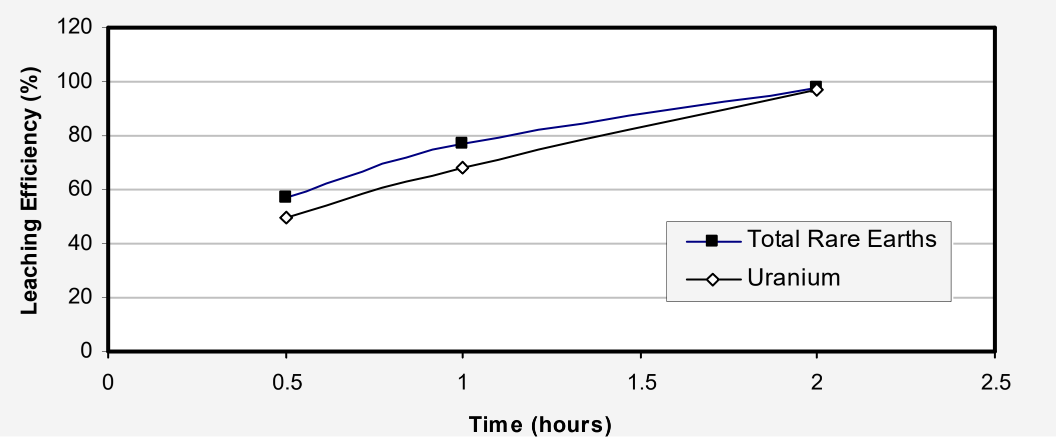


Figure (6): Effect of leaching time on the leaching efficiencies of REE and U

The obtained data show that by increasing stirring time from half to two hour the leaching efficiency of REEs increased from 57.5 to 98% while for uranium leaching it was found to increase from 48 at 1/2 hr to 97% at 2 hr.

III. 3.2 Potentialities of U and REEs product recovery

To recover both metal values from the prepared leach liquor, it has previously been mentioned this has been made by two alternatives. In the first, U was first recovered by its selective exchange upon an anion exchange resin followed by REEs recovery from the resin effluent by their precipitation as oxalate. In the second alternative, a two step precipitation procedure was applied; namely oxalate precipitation of REEs followed by U precipitation as sodium diuranate at pH 12 from the REEs filtrate solution. To study the potentiality of these two alternatives, proper leach liquor was performed using the optimized leaching factors and this solution was found to assaying 0.975 g/l U and 0.85g/l REEs.

III.3.2.1 ion exchange recovery of U and oxalate precipitation of REE

The ion exchange recovery of uranium is based upon the fact that hexavalent uranium can exist in sulfate leach liquors as two anionic complexes viz, $[UO_2 (SO_4)_3]^{-4}$ and $[UO_2 (SO_4)_2]^{-2}$as well as the neutral form $UO_2 SO_4$, besides the uranyl cation $[UO_2]^{+2}$. The relative abundance of these species depends mainly upon their equilibrium constants as well as on the sulfate content of their acid solution. According to the chemical characteristic of the studied metal values, it can be mentioned that the REEs, Al, Zn besides most the undesired metal impurities exist in their cationic forms except uranium which exists in both cationic and anionic forms. Therefore, an anion exchanger resin would be selective for uranium recovery from the obtained sulfate leach liquor. In the latter, the competing anions include mainly SO_4^{2-}, HSO_4^- and some ferric sulfate anionic complexes $[Fe(SO_4)_2^{-1}$, $Fe(OH) (SO_4)_2^{-2}]$ which would be strongly adsorbed at exceeding pH 1.8, while below this value HSO_4^- would be strongly adsorbed**.(Merritt,1971; Preuss ; and Kunin , 1965)**.

A pyrex glass was packed with 25 ml of wet settled Dowex 1 ion exchanger over a glass wool. The pH of the prepared leach liquor was firstly to adjusted 1.8 using 5% NaOH solution and then was passed through the prepared resin bed allowing a contact time 1.25ml/min. Uranium adsorption was continued until the ion exchanger was saturated (influent concentration = effluent concentration) and periodical samples were collected every 100 ml for analysis. The obtained result shown in Table (3) indicate that the obtained uranium saturation level has attained 1.497 g/25ml wsr i.e. about 60 gU/l wsr realizing about 80% of the theoretical capacity (75 g/l). This relatively low saturation capacity is most probably due to high, HSO_4^-, SO^{-2} and $Fe_2 (SO_4)_2^{-2}$ content of the working GV leach liquor. After attaining uranium saturation the working resin column was washed with distilled water before uranium elution using NaCl solution acidified to 0.1M by sulfuric acid using a contact time of 0.5 ml/min. The eluate samples were collected every 100 ml for uranium analysis and the data are represented in table (4).

Table (3): Analytical results of uranium in GV effluent sulfate leach liquor using Dowex 1 and its adsorption efficiency

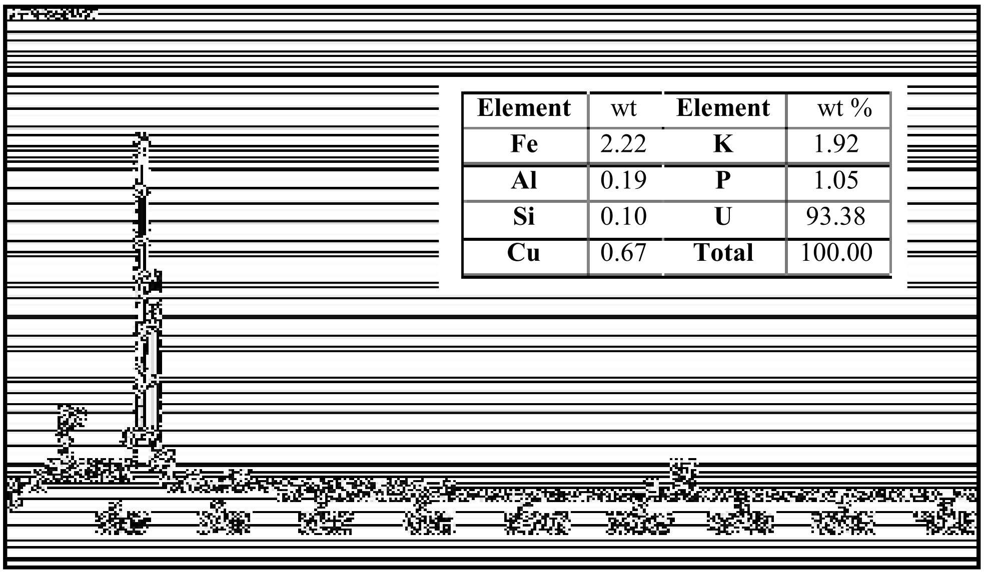
Effluent sample No. (100ml)	Uranium content mg/100ml	Uranium Adsorption, (%)
1	Nil	100
2	Nil	100
3	Nil	100
4	Nil	100
5	Nil	100
6	Nil	100
7	Nil	100
8	Nil	100
9	Nil	100
10	Nil	100
11	Nil	100
12	0.009	90
13	0.013	86
14	0.023	76
15	0.040	58
16	0.055	43
17	0.062	36
18	0.073	25
19	0.082	15
20	0.0975	0

Table (4): Analytical results of uranium in the obtained eluate samples of the saturated Dowex 1working resin column

Eluate sample No. (100 ml)	Uranium content (mg/100ml)
1	**0.0192**
2	**0.115**
3	**0.420**
4	**0.315**
5	**0.260**
6	**0.128**
7	**0.110**
8	**0.065**
9	**0.00**

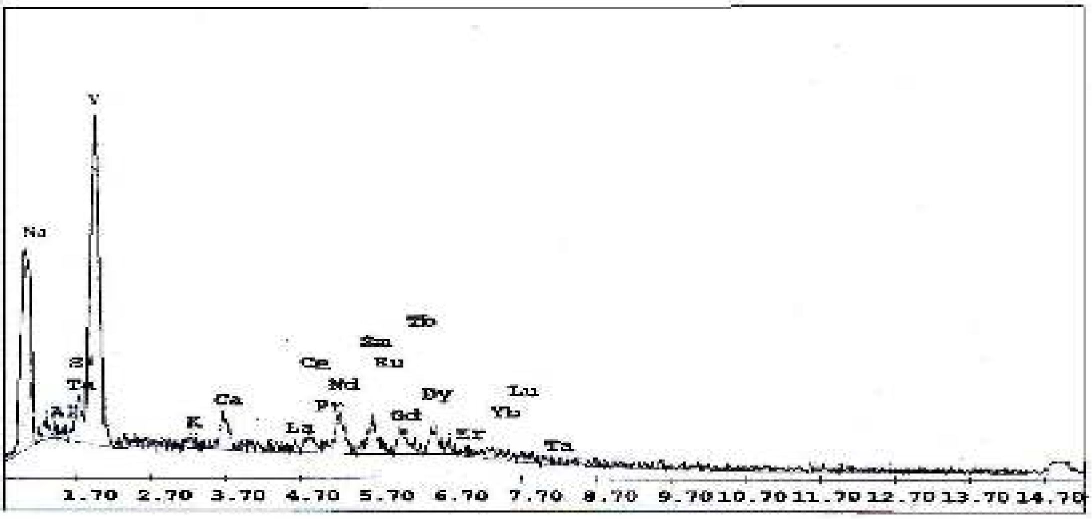
From the collected eluate samples, uranium was precipitated with ammonia solution at pH 7 and the final product was then calcined to U_3O_8 and subjected to ESEM-EDX analysis (Fig. 7). From the latter, it is clearly evident that uranium assay attains about up to 93% with relatively low levels of the associated impurities. In the meantime, uranium in the obtained product was titrimetrically analyzed against metavanadate and was found assay about 88%.

Figure (7): ESEM-EDX analysis of calcined Gattar V uranium cake prepared by Dowex 1 ion exchange resin



Element	wt	Element	wt %
Fe	2.22	K	1.92
Al	0.19	P	1.05
Si	0.10	U	93.38
Cu	0.67	Total	100.00

On the other hand, the uranium-free effluent samples were collected and subjected to oxalate precipitation of the REEs content. After adjusting the pH to 2.8 using NaOH solution, oxalic acid was added until a pH value of about 1.3. The obtained oxalate precipitation was filtered, washed and then calcined at 900 ^{0}C and subjected to ESEM-EDX analysis and the obtained data are shown in Fig. (8). From the analytical results of the obtained REO concentrate, it is evident that it assays a total of about 82% REE while the heavy REE fraction in this concentrate attain up to 76% as represented by Er, Yb, Lu, Y and Dy. In this regard, it is interesting to refer to the analysis of the separated grains of RE-bearing mineral that has magnetically been obtained which indicated that it is mainly composed of light REE. In other words, it is most probable that the heavy rare earth elements which largely constitute the obtained REO (calcined oxalate precipitate) are mostly adsorbed on the iron oxides (about 8%) and clay fraction of the working Hammamat sediment G-V ore material.

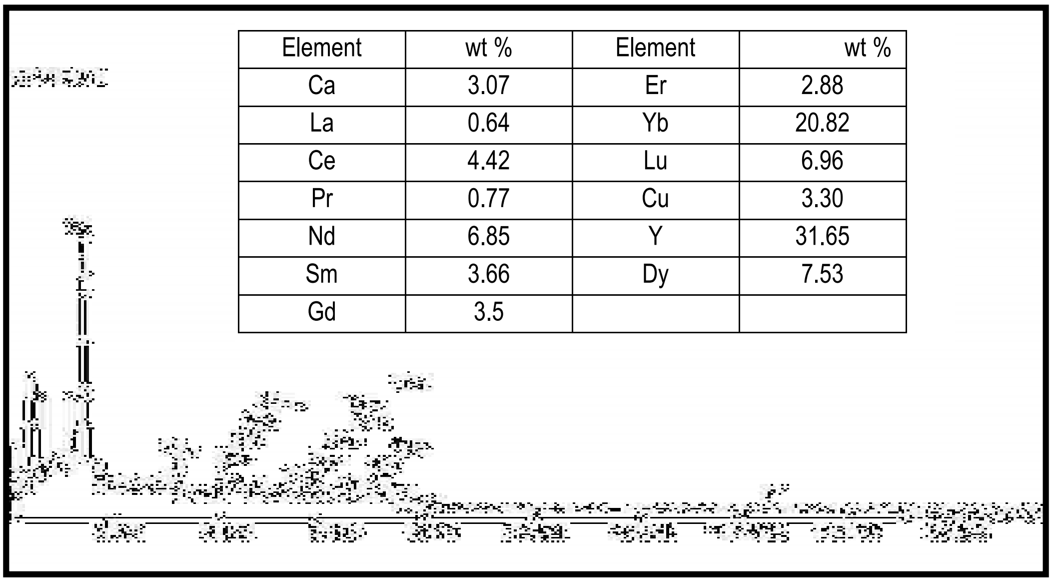


Element	wt %	Element	wt %
Na	10.07	Er	1.23
La	1.64	Yb	10.82
Ce	4.42	Lu	3.96
Pr	0.97	Ca	1.30
Nd	6.85	Ta	1.99
Sm	3.66	Y	41.57
Gd	4.5	Dy	5.56

Figure (8): ESEM-EDEX analysis of calcined REEs oxalate precipitation from GV sulphate leach liquor after uranium separation

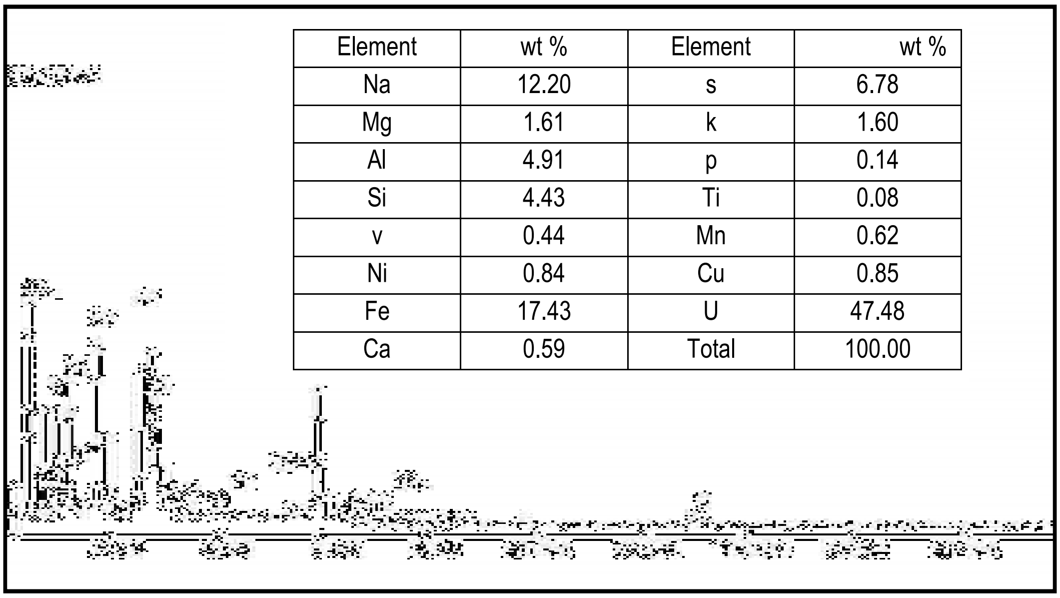
III.3.2.2. Recovery of REEs and uranium product by selective precipitation

In the direct precipitation alternative, the REE have first been selectively precipitated by the addition of 20% oxalic acid to the sulfate leach liquor of GV ore materials. This procedure has proved advantageous in avoiding iron precipitation through its reduction to the ferrous state. ESEM-EDX analysis of the obtained REO concentrate after calcination of their oxalate precipitate is shown in Fig.(9). From the latter, it is clearly evident that the obtained REO concentrate is mostly composed of heavy REEs. This method consumed additional amount of oxalic acid to convert all the ferric iron to ferrous oxalate which would precipitate at pH 8.5. In other words the present uranium content would combine with oxalic acid to form uranium – oxalate complex which would not be decomposed before increasing the pH value to 12 using NaOH i.e it wouldn't be contaminated with iron. From ESEM-EDX analysis of the calcined uranium precipitate shown in Fig.(10), it is indicated that uranium assays about 48%. i.e a highly impure concentrate.



Element	wt %	Element	wt %
Ca	3.07	Er	2.88
La	0.64	Yb	20.82
Ce	4.42	Lu	6.96
Pr	0.77	Cu	3.30
Nd	6.85	Y	31.65
Sm	3.66	Dy	7.53
Gd	3.5		

Figure (9): ESEM-EDEX analysis of calcined REEs oxalate selective precipitation from GV sulphate leach liquor before uranium separation



Element	wt %	Element	wt %
Na	12.20	s	6.78
Mg	1.61	k	1.60
Al	4.91	p	0.14
Si	4.43	Ti	0.08
v	0.44	Mn	0.62
Ni	0.84	Cu	0.85
Fe	17.43	U	47.48
Ca	0.59	Total	100.00

Figure (10): ESEM-EDX analysis of calcined U cake after its direct precipitation of the sulphate leach liquor Gattar V ore at pH12.

Due to the highly impure nature of the uranium concentrate obtained from direct precipitation, it was found that it is necessary to subject the sodium uranate precipitate to a purification step through its dissolution in a 20% sodium carbonate solution. Accordingly, uranium would selectively dissolve as its uranyl carbonate, namely.

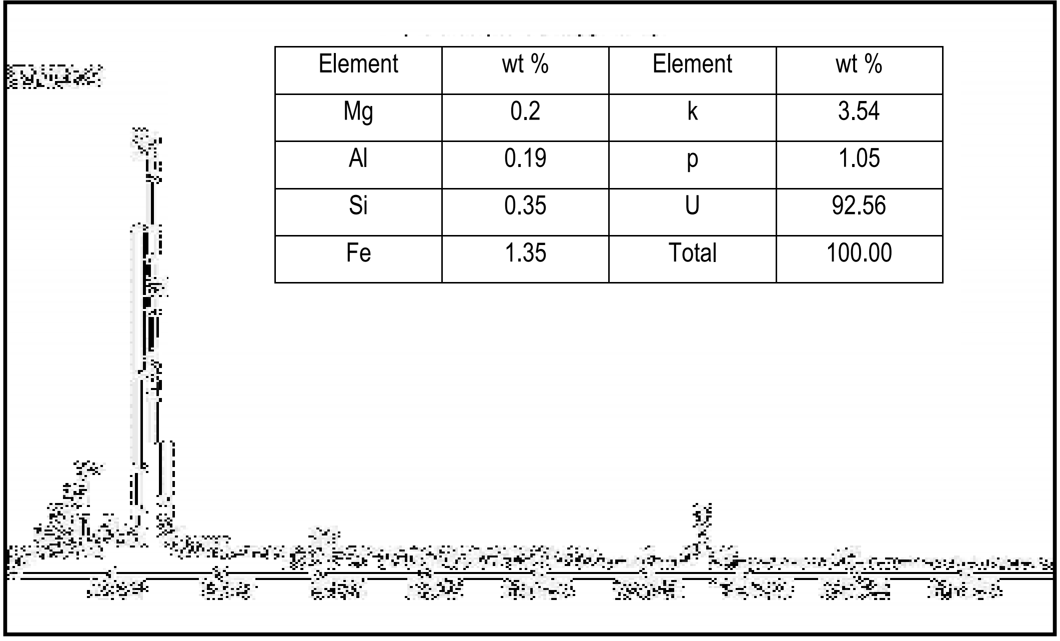
$$UO_3 + 3Na_2CO_3 + H_2O \longrightarrow Na_4UO_2(CO_3)_3 + 2NaOH$$

From this solution, uranium recovery has been accomplished by acidifying the uranium carbonate complex to a pH less than 5.2 followed by precipitating the diuranate according to the following equation:

$$Na_4UO_2(CO_3)_3 + 6HNO_3 \longrightarrow UO_2(NO_3)_2 + 4NaNO_3 + 3CO_2 + 3H_2O$$

$$2UO_2(NO_3)_2 + 6NH_3 + 6H_2O \longrightarrow (NH_4)_2U_2O_7 + 4NH_4NO_3 + 3H_2O$$

The ammonium diuranate precipitate was then calcined and subjected to ESEM-EDEX analysis as shown in Fig.(11). From the latter, it is clearly evident that uranium assay attains up to about 92.5 % with relatively low levels of the associated impurities.



Element	wt %	Element	wt %
Mg	0.2	k	3.54
Al	0.19	p	1.05
Si	0.35	U	92.56
Fe	1.35	Total	100.00

Fig.(11): ESEM-EDEX analysis of calcined uranium cake after purification

Conclusion

From the foregoing mineralogical and potential hydrometallurgical studies of G-V mineralization Hammamat sediments it was possible to define the interesting mineral and preparing some products of both lanthanides and uranium from their sulphate leach liquor. This has been possible by using two alternatives. In the first alternative anion exchange resin (Dowex-I) was used for U adsorption and it was possible to obtain a loading capacity of 55.5g U/l wet settled resin which is equivalent to about 73% of theoretical capacity. Uranium was eluted from the resin using 1M NaCl solution acidified to 0.1 H_2SO_4 at a contact time of 0.5 ml/minutes. The U effluent solution was then directed to recovery of lanthanides by oxalate precipitation after pH adjustment to 2.8. The second alternative depends on the direct precipitation where the lanthanides were first separated by using oxalate precipitation followed by uranium recovery at pH 12. Comparison between the two alternatives would prove that the first alternative would be more economic and efficient.

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