

**LEACHING AND EXTRACTION OF URANIUM  
FROM MINERALIZED HAMMAMAT SEDIMENTS  
AT GATTAR-V LOCALITY, NORTH EASTERN  
DESTERT, EGYPT**

**By**

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**Abstract**

*Hammamat sediments situated at the contact with Gattar granite show a relatively rich uranium mineralization (G-V). The latter is mainly represented by yellow colored secondary uranium minerals that occur essentially along the cracks or else disseminated within the clay minerals. A mineralized technological sample assaying 0.12% uranium was prepared for the present leaching and extraction studies. The relevant leaching factors were determined as optimum indicated that 70 kg H<sub>2</sub>SO<sub>4</sub>/t and -50 mesh size ore are adequate to leach 87% of input uranium at 1/1 solid-liquid ratio for 4h leaching time at room temperature. The extraction experiments from the*

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*obtained leach liquor were carried out using D<sub>2</sub>EHPA/TOPO mixture in kerosene to prepare a highly pure uranium concentrate.*

### **Introduction**

Gebel Gattar granite is surrounded at its northern border by the Hammamat sediments of Gebel Um Tawat. This area is located in the northern Eastern Desert of Egypt between Latitudes 27° 05' and 27° 08' N and Longitudes 33° 13' and 33° 18' E (Fig.1).

The Hammamat sediments at the Gattar-V locality host an important uranium mineralization. Various geological and radiometric studies on the Hammamat sediments and their contact with Gattar granite have been previously carried out by several authors; namely Shalaby (1990), Salman et al. (1990) and Nossair (1994). These sediments are mainly composed of un-metamorphosed repeatedly intercalated succession of conglomerates, greywakes and siltstones. At the contact zone with Gattar granites the Hammamat sediments have been locally subjected to thermal metamorphism.

The marginal parts of Gattar pluton are medium- to fine-grained, highly fractured and strongly affected by wide-spread deuteric and post-magmatic hydrothermal alterations. The uranium mineralizations at the periphery of the Gattar granite are mainly associated with the hematitized and episyenitized zones. The cavities left after silica leaching are sometimes filled with secondary uranium

minerals and dark violet fluorite in addition to black clots of Mn and Fe oxides (Nossair, 1994). The uranium mineralization in the Hammamat sediments at Gattar-V locality is unique and is of particular interest since most of the uranium mineralization in the region is recorded in these sediments rather than intruded in the younger granites. On the other hand, the remobilization of uranium from granite could have taken place fairly the action of an effective transporting medium such as fluids moving along channels as represented by joints and cracks. The deposition of uranium in the Hammamat sediments is observed in the weak zones including bedding and foliation planes, joints, cracks and fissures. The frequent presence of abundant iron oxides in the altered rocks might have facilitate the precipitation of uranium from the ascending hydrothermal fluids as these oxides act as strong adsorbing agent. Shalaby (1990) suggested that the supergene (descending) mineralized fluids have percolated through the joints and fractures of the country fertile granite and redeposited uranium in the Hammamat fracture network.

Previous studies upon uranium leaching and in particular its extraction from Hammamat sediments mineralizations are actually limited. El Hazek (1995) in her studies for uranium agitation leaching from this occurrence has applied both sulfuric acid and alkaline reagents (sodium carbonate and bicarbonate) as well as some trials using sodium or ferric chloride. Uranium extraction from the

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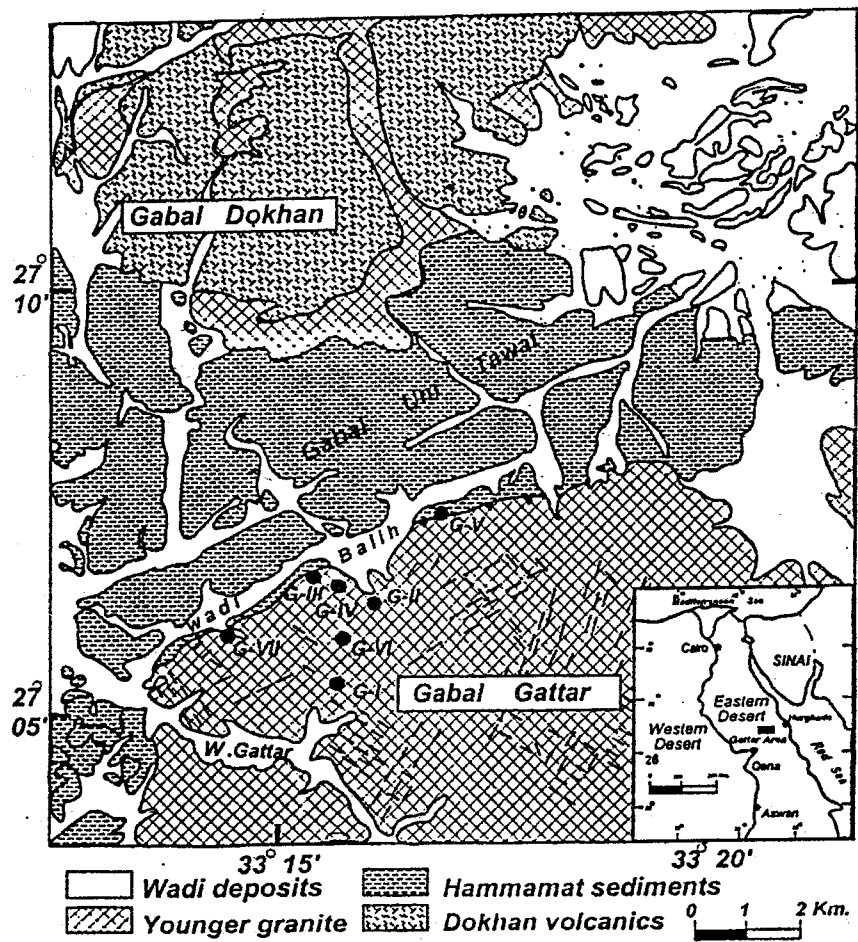


Fig.1: Location and geological maps of Gabal Gattar area ( after Ghobrial and Lotfi, 1967 )

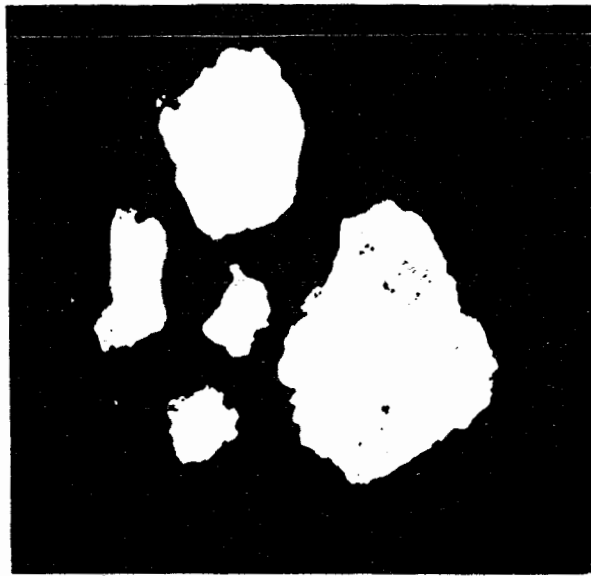
corresponding liquors was studied by anion exchange resin for the alkaline while from the acid liquor, a solvent extraction using tri-n-octyl amine was applied. Sayed (2000) has however, studied the static leaching of the mineralized Hamammat sediments after prior curing using sulfuric acid at 0.2 liquid-solid ratio for 24h. This was followed by uranium recovery from the leach liquor by direct precipitation with lime in one or two steps for subsequent precipitation by selective alkaline leaching.

In the present paper, a detailed study of the acid leaching characteristics of the Hamammat sediments' mineralization was undertaken using sulfuric acid. This is followed by uranium extraction from the obtained sulfate pregnant liquor with the synergistic organic solvent mixture; namely di-2-ethyl hexyl phosphoric acid (D<sub>2</sub>EHPA) and tri-octyl phosphine oxide (TOPO) in kerosene. As a general rule, solvent extraction is preferred over ion-exchange methods for acidic leachates containing more than 1g/L uranium. For this purpose, a technological sample from the mineralized Hamammat locality of Gattar-V was properly collected. This sample assayed 0.12% uranium.

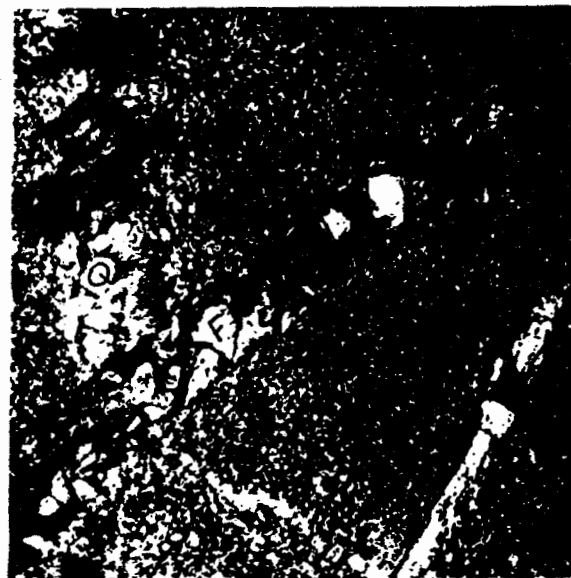
### **Mineralogical and Chemical Composition of the ORE Material**

Uranium minerals are represented mainly by the canary yellow colors of uranophane (Fig.2) and beta-uranophane. These minerals

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**Fig.2: Canary yellow colors of separated uranophane grains  
(under binocular X=360)**



**Fig.3: Dark violet fluorite veinlet invaded the studied sediments  
(P.P.L. X=35) F: fluorite Q: quartz**

occur as filling fractures in the sediments and are associated with secondary quartz and dark violet fluorite veinlets (Fig.3). Some uranium might occur also as adsorbed ions on the clay and iron oxide minerals. The host rock as represented by Hammamat sediments has been subjected to many types of alterations, including mainly; hematitization, kaolinization and silicification.

The XRD data showed that the host Hammamat sediments consist mainly of illite, montmorillonite and quartz (Table.1). These data were also confirmed by the the infrared spectrum (IR) of the studied sample. The latter shows indeed the pattern of a low-magnesium illite (Fig.4) and the slight variation in the spectrum is actually due to a variable content of magnesium (Wilson, 1994). This spectrum shows weaker absorption bands at 690 and 795  $\text{cm}^{-1}$ . The broad OH- stretching band near 3621  $\text{cm}^{-1}$  and 825 $\text{cm}^{-1}$  and 750 $\text{cm}^{-1}$  bands originated from Al-Mg-OH.

The collected mineralized composite sample chosen for the present work has been chemically analyzed. Table (2) shows the chemical analysis for the major and some trace elements using the proper wet chemical (Shapiro, 1975) and atomic absorption analytical methods respectively. Uranium analysis was performed by spectrophotometric technique using Arsenzo III method (Marczenko, 1976).

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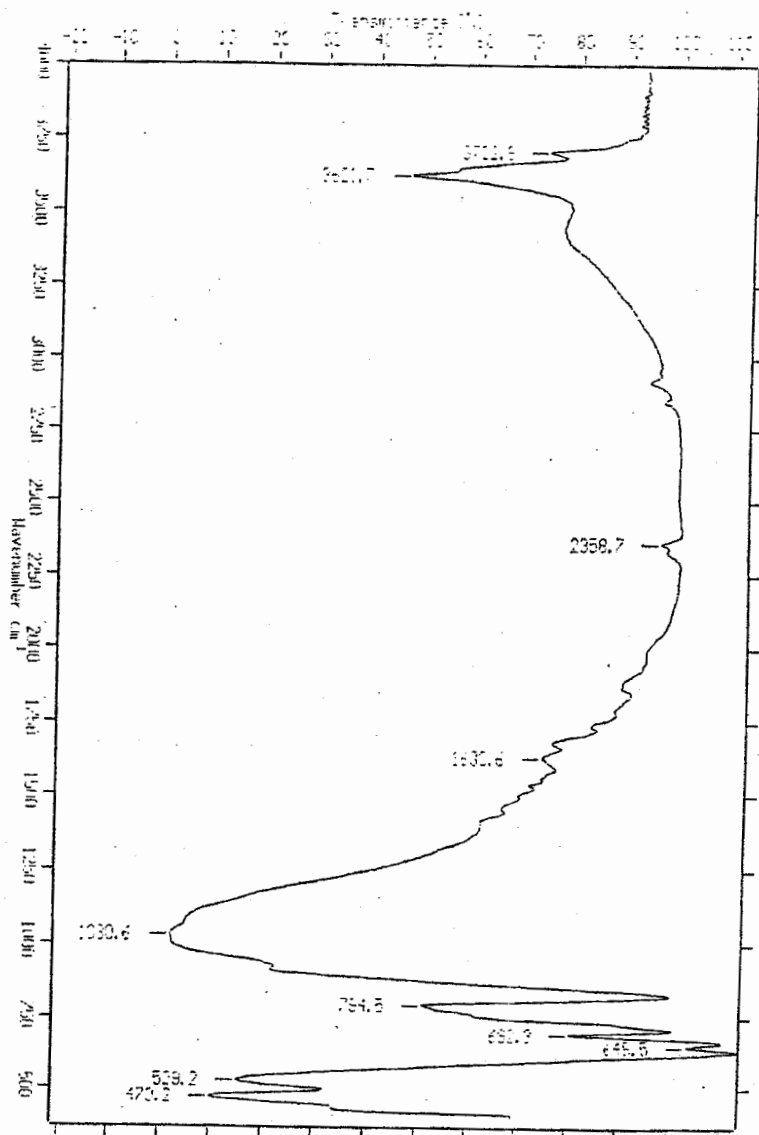


Fig.4: IR spectrogram of low-Mg illite of the studied Hammamat sediments.



**Table 1:** XRD of the working composite sample of Hammamat sediments ore material.

| Sample |      | Quartz<br>ASTM<br>No. (5-0490) |      | Montmorillonite<br>ASTM<br>No. (7-304) |      | Illite<br>ASTM<br>No. (9-334) |     |
|--------|------|--------------------------------|------|--|------|-------------------------------|-----|
| d A°   | I/I° | d A°                           | I/I° | d A°                                   | I/I° | D A°                          |     |
| 9.85   | 85   |                                |      | 9.70                                   | 60   | 9.90                          | 80  |
| 4.81   | 81   |                                |      | 4.85                                   | 60   | 4.90                          | 60  |
| 4.53   | 44   |                                |      | 4.48                                   | 100  |                               |     |
| 4.42   | 34   |                                |      |  |      | 4.46                          | 100 |
| 4.28   | 37   |                                |      |  |      | 4.29                          | 70  |
| 3.94   | 15   |                                |      |  |      | 3.88                          | 60  |
| 3.36   | 100  | 3.34                           | 100  |  |      | 3.36                          | 100 |
| 3.17   | 90   |                                |      | 3.22                                   | 80   | 3.10                          | 50  |
| 2.55   | 15   |                                |      | 2.59                                   | 60   | 2.57                          | 100 |
| 2.46   | 15   | 3.46                           | 12   |  |      | 3.45                          | 50  |
| 2.24   | 4    | 2.24                           | 6    | 2.23                                   | 60   |                               |     |
| 2.14   | 2    | 2.13                           | 9    |  |      | 2.14                          | 60  |
| 2.06   | 4    |                                |      | 2.10                                   | 40   |                               |     |
| 1.82   | 9    | 1.82                           | 17   |  |      |                               |     |
| 1.67   | 5    |                                |      | 1.67                                   | 60   | 1.65                          | 60  |
| 1.54   | 5    | 1.54                           | 15   | 1.51                                   | 60   |                               |     |
| 1.50   | 7    |                                |      | 1.49                                   | 40   | 1.50                          | 80  |

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**Table 2:** Chemical analysis of the working composite sample of Hammamat sediments ore material.

| Major oxide                      | Wt %         | Trace element | ppm  |
|----------------------------------|--------------|---------------|------|
| SiO <sub>2</sub>                 | 70.21        | Ba            | 96   |
| TiO <sub>2</sub>                 | 00.61        | Cr            | 84   |
| Al <sub>2</sub> O <sub>3</sub>   | 08.36        | Cu            | 68   |
| Fe <sub>2</sub> O <sub>3</sub> * | 07.08        | Ni            | 38   |
| MnO                              | 00.01        | Pb            | 360  |
| MgO                              | 03.68        | Sr            | 132  |
| CaO                              | 01.33        | Th            | 49   |
| Na <sub>2</sub> O                | 01.23        | U             | 1200 |
| K <sub>2</sub> O                 | 02.71        | Zn            | 417  |
| P <sub>2</sub> O <sub>5</sub>    | 00.92        |               |      |
| <u>L.O.I.</u>                    | <u>01.83</u> |               |      |
| Total                            | 97.97        |               |      |

\* Fe<sub>2</sub>O<sub>3</sub> was determined as total iron.

### **Leaching Characteristics of the Uraniferous Hammamat ORE Material**

In the present work, the uraniferous technological sample of Hammamat ore material was subjected to agitation leaching using sulfuric acid in order to determine the leaching characteristics of

uranium and the required leaching conditions. Leaching experiments were thus undertaken to determine the effects the acid concentration and in turn its consumption, the solid/liquid ratio as well as the leaching temperature and time.

**Effect of Acid Concentration:**

This effect was studied in the range of 10 to 100 g/L sulfuric acid while the other leaching conditions were fixed at 4h leaching time in a S/L of 1:1 at room temperature. From the obtained results represented in Fig.5, it is clearly evident that 70g/L acid gave a leaching efficiency of 87 %. This acid concentration would be equivalent to an acid consumption of 70kg/t ore. In the present work, the ore sample shows that about 40kg sulfuric acid/ton ore are consumed by the carbonate minerals (calcite and possibly dolomite). The relatively high MgO content is most probably attributed to clay minerals.

**Effect of Solid / Liquid Ratio:**

This factor was studied in the range of S/L ratio of 1:1 to 1:5 while fixing acid concentration at 70g/L at ambient temperature for 4h leaching time. Fig.6 shows the obtained results which indicate that decreasing the solid/liquid ratio results in decreasing the uranium concentration. This is actually due to the increased the leaching acid volume.

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#### **Effect of Leaching Temperature:**

The temperature effect upon uranium leaching efficiency was studied in the range of room up to 90 °C while the other leaching conditions were fixed at 70g/L acid and 4h leaching time in a S/L ratio of 1:1. The obtained results plotted in Fig.7 indicate a steady increase in the leaching efficiency and which attained about 91% at 90°C.

#### **Effect of Leaching Time:**

This effect was studied in the range of 30min. up to 10h while the other factors were fixed 70g/L acid at room temperature in a S/L 1:1. The obtained results are shown in Fig.8 and indicate that after 4h the obtained leaching efficiency attained 87%.

From the forgoing results, it can be concluded that a uranium leaching efficiency of about 87 % is easily obtained using mild leaching conditions. These include 70kg/t sulfuric acid at a solid/liquid ratio of 1/1 at room temperature for 4h leaching time on - 50 mesh size ore.

#### **Uranium Recovery by Liquid – Liquid Extraction**

The obtained leach liquor of Hamammat ore material assays 1.05 gU/L and could be extracted by the liquid-liquid extraction method. The type of the organic solvent depends on the nature of the uranium

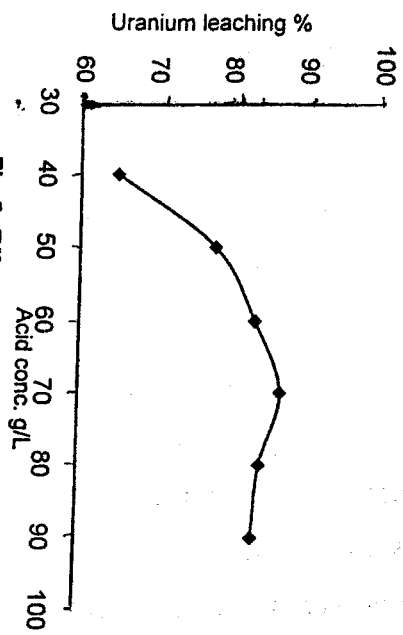


Fig.5: Effect of acid concentration

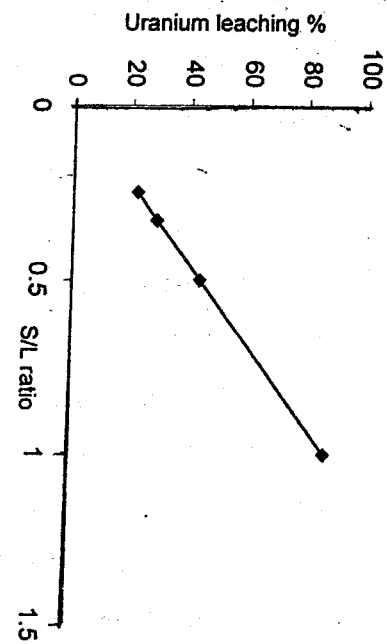


Fig.6: Effect of solid/liquid ratio

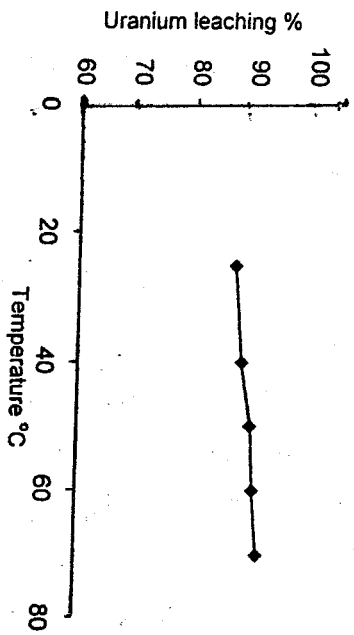


Fig.7: Effect of temperature

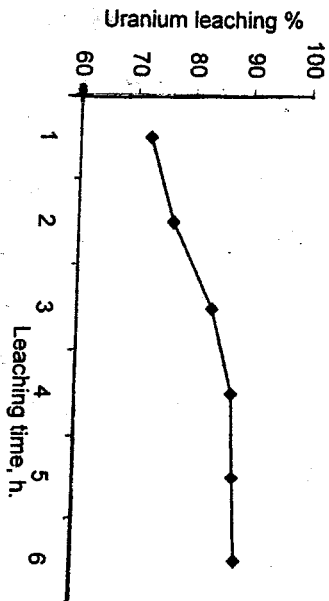


Fig.8 Effect of leaching time

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species in the aqueous phase, which depends in turn on the type of the leaching medium. In sulfuric acid medium,  $\text{UO}_2^{2+}$  is present in a dynamic equilibrium with uranyl disulfate or trisulfate anionic complexes and hence acidic organo-phosphorous or alkyl-amine solvents can be used for uranium recovery.

In the present work, Di (2-ethyl hexyl) phosphoric acid ( $\text{D}_2\text{EHPA}$ ) is used as an extractant. It belongs to the organophosphorous acidic extractants and was used in combination with trioctyl phosphine oxide extractant (TOPO) in a manner to improve its efficiency. According to Sato (1962),  $\text{D}_2\text{EHPA}$  in kerosene extracts uranium from sulfuric acid solutions by the ion exchange mechanism while TOPO will increase its efficiency. The synergic solvent mixture for the present work was prepared by dissolving the proper weight necessary for the working molarity of each solvent in Sunset kerosene of Misr Petroleum Co.

A stock Hammamat pregnant liquor prepared by leaching and concentration by evaporation to half of its original volume to assay 2.1 g U/L for the extraction experiments. In studying the extraction characteristics of the prepared liquor by the proposed mixture, batch equilibrium experiments were carried out. In each experiment equal volumes of the aqueous and organic liquors were shaken for 5 minutes using laboratory glass separating funnel. After shaking of the two phases they were left for 5 minutes for settling and aliquot portions of the aqueous phases were taken for uranium analysis while that in the

organic phases was calculated by difference. From the latter, the distribution coefficient was calculated.

Fixing the  $D_2EHA$  at 0.25M, a batch equilibrium test was first performed at the above-mentioned conditions. The obtained uranium distribution coefficient was found to be 9.50. As mentioned before, TOPO was used to increase the extraction efficiency of the working solvent. To study the optimum molarity value of TOPO, 4 batch equilibrium tests were undertaken in which  $D_2EHPA$  concentration was fixed at 0.25M while that of TOPO was increased from 0.02M to 0.08M. From the obtained results (Table3), the uranium distribution coefficient increased to 11.35 at 0.02M and further increased to 15.15 at 0.06M TOPO. Thereafter, the uranium distribution decreased however to 12.13. Accordingly, the optimum TOPO concentration matching with the obtained maximum value of uranium distribution coefficient attains 0.06M. In other words, the optimum synergistic mixture molar ratio is about  $4D_2EHPA : 1TOPO$

**Table (3) :** Effect of TOPO concentration on uranium extraction from Hammamat leach liquor by 0.25 M  $D_2EHPA$  in kerosene

| TOPO concn., M | Uranium concn. g/L |           | $D_a^0$ |
|----------------|--------------------|-----------|---------|
|                | Org. phase         | Aq. Phase |         |
| 0.00           | 1.90               | 0.20      | 9.50    |
| 0.02           | 1.93               | 0.17      | 11.35   |
| 0.04           | 1.95               | 0.15      | 13.00   |
| 0.06           | 1.97               | 0.13      | 15.15   |
| 0.08           | 1.94               | 0.16      | 12.13   |

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### **Construction of McCabe-Thiele Diagram for Uranium**

#### **Extraction:**

In order to determine the number of theoretical extraction stages for uranium by the proposed solvent mixture in a counter current system, an extraction McCabe-Thiele diagram was constructed. For this purpose, a series of batch equilibrium tests was performed using the prepared stock Hammamat pregnant solution as contacted in variable ratio with the working synergistic mixture of D<sub>2</sub>EHPA/TOPO in kerosene. The attained equilibrium uranium concentrations in the two phases were plotted as an equilibrium curve (Fig.9). To the latter, a suitable operating line whose slope represents the flow ratio of the two phases was properly fitted. From the latter, it is evident that an O/A flow ratio of 0.28 (O/A) would result in 3 to 4 theoretical stages for uranium extraction. In this way, uranium in the raffinate would assay 100 to 10ppm respectively.

### **Construction of MaCabe-Thiele Diagram for Uranium Stripping**

To construct the McCabe-Thiele diagram for uranium stripping from Hammamat loaded D<sub>2</sub>EHPA/TOPO solvent mixture; the latter was loaded with 6.68gU/L and it was decided to perform stripping by (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> solution (200 g/L). The uranium equilibrium



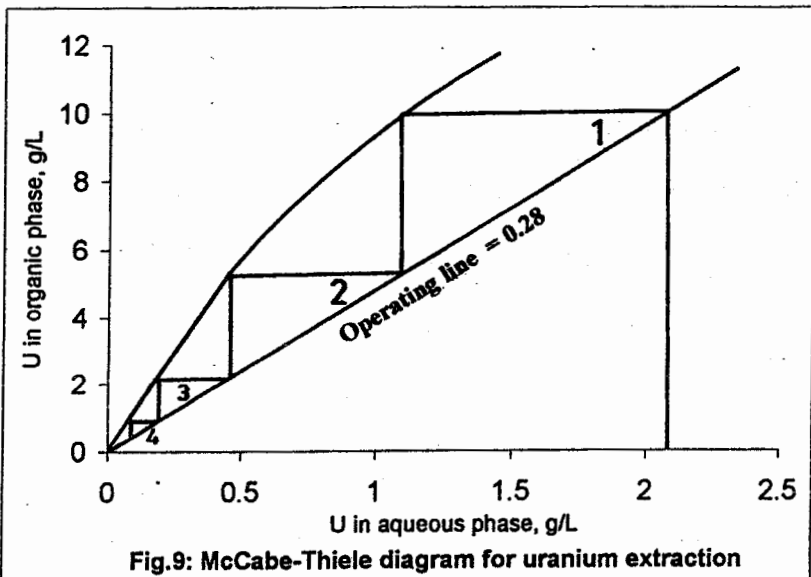


Fig.9: McCabe-Thiele diagram for uranium extraction

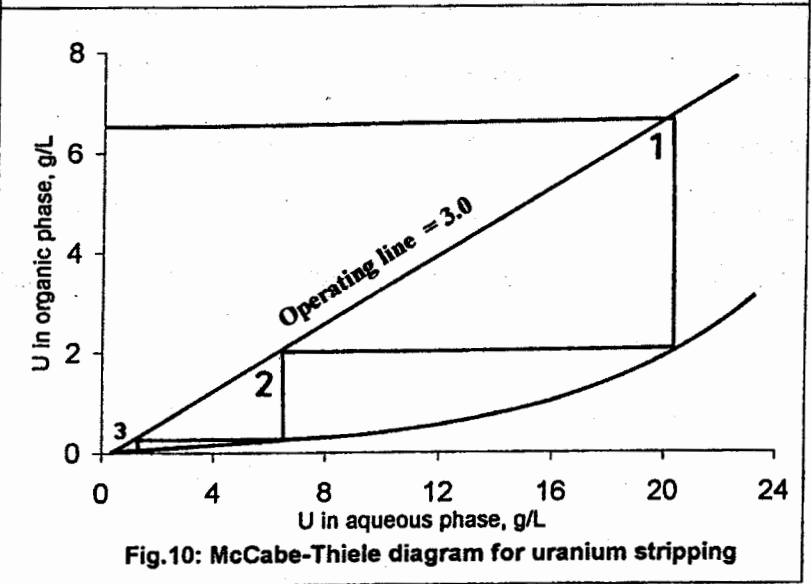


Fig.10: McCabe-Thiele diagram for uranium stripping

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data were obtained by shaking different O/A ratios using a contact time of 5 min. and a settling time of 5 min. From the obtained data shown in Fig. (10), it is clearly evident that it would be possible to have an aqueous strip solution assaying up to about 21 g/L and deplete uranium in the recycle solvent to less than 0.4 g U/L. This has actually been achieved by fitting an operating line of a slope O/A of about 3.0 and which resulted in three theoretical stages.

### Conclusion

A uraniferous sample representing the mineralization of Hammamat at Gattar-V locality assaying 0.12 was subjected to chemical processing for the preparation of pure uranium concentrate. A uranium leaching efficiency of about 87% was obtained using mild agitation leaching conditions. These include 70kg/t sulfuric acid in a solid/liquid ratio of 1/1 at a room temperature for 4 hours leaching time on -50 mesh size ore.

Uranium recovery experiments were carried out by solvent extraction using a synergic mixture of 0.25 M D2EHPA/0.06 M TOPO in kerosene. Studied parameters for solvent extraction at equilibrium showed that, 4 theoretical stages are required at a flow

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rate O/A of 0.28 for almost complete uranium extraction and 3 stripping stages are required to strip uranium in ammonium carbonate solution. Finally, precipitation of almost pure yellow cake (ammonium diuranate) by ammonia was obtained by calcination at 850 °C resulted in U<sub>3</sub>O<sub>8</sub> with a recovery exceeding 98.5%.

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