

## Behavior of some Radionuclides During the weathering and Alterations in sedimentary rock samples, Sinai, Egypt.

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**Abstract:** This study aims to examine the mobility of uranium and its isotopes and daughters during the acid leaching. This illustrates the transmission of radionuclide's from solid material (ore) to the liquid phase (leachate). In the same context the study of the daughter / parent ratios in the three phases, original sample, leach liquor and residue will give a complete picture about the behavior of these radionuclides during weathering and alterations of uranium-bearing rocks. Three samples of different sedimentary rock types from Farsh El Azraq area in southwestern Sinai were collected and prepared for various analyses. Wet chemical analyses were carried out for major oxides and trace elements. Natural radionuclides were measured by HPGe detector in original samples, pregnant solution and residuals. The results showed that the sum of activities of <sup>238</sup>U and its daughter in both pregnant solutions and residuals are nearly equal the activity in the original sample. The behavior of <sup>226</sup>Ra in the acidic leaching is completely different from <sup>238</sup>U and its isotopes. Selective leaching studies have shown that uranium isotopes are leached to the same extent but that is not observed in <sup>234</sup>U due to the  $\alpha$ -recoil phenomenon. The lowest leachability in all the samples is present in <sup>226</sup>Ra, <sup>214</sup>Pb and <sup>214</sup>Bi. These results can explain the difference in behavior between the uranium isotopes and <sup>226</sup>Ra series during natural weathering and alterations of uraniumiferous-rock materials.

**Keywords:** Radionuclides, Farsh El Azraq, HPGe detector, Sedimentary rock, Leaching.

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### I. Introduction

The <sup>238</sup>U, <sup>235</sup>U, and <sup>232</sup>Th are the parents of the three natural decay series, each of these series consists of many daughter products generated through successive decay of parent radionuclides. Natural uranium is a composite of the isotopes <sup>238</sup>U (99.28%), <sup>235</sup>U (0.72%) and <sup>234</sup>U (0.0057%) [Powell et al., 2007]. These isotopes have the same geochemical behavior. While on a mass basis there is far more <sup>238</sup>U than <sup>235</sup>U in a natural sample, the activity ratio is approximately 21.7 [Porcelli and Swarzenski, 2003]. The radioactive series reaches secular equilibrium when the activity ratios of daughter/parent are equal to unity [Wally El-Dine, 2008]. Radioactive disequilibrium arises in the natural radioactive series due to recent fractionation events (continuous or instantaneous processes) which lead to the loss or gain of radionuclides that are mobile in the ambient environmental conditions. The long-lived radionuclides in the natural radioactive series are generally used to assess the state of radioactive disequilibrium [E.S. Abd El-Halim et al., 2017]. The physical and chemical processes used to extract uranium from ore, such as crushing and acid treatment, produce large amounts of mill tailings. The principal aim of this work is to study radionuclide transfer from solid material (ore) to the liquid phase (leachate). Acid leaching is known to be predominant process for uranium recovery from ores, usually with sulfuric acid because its relatively low cost [Umanskii and Klyushnikov, 2012].

### II. Materials And Methods

#### 2.1. Study area

The study area (Fig.1) is located in the southwestern Sinai, Egypt. Where the samples collected from selected location; Farsh El Azraq according to GPS, it is located as the intersection of longitude 33° 25' and latitude 29° 59'. The studied area was chosen according to an environmental plan study.

Where, the collected samples represent what is known Um Bogma formation which is of lower carboniferous age (325 m.y).

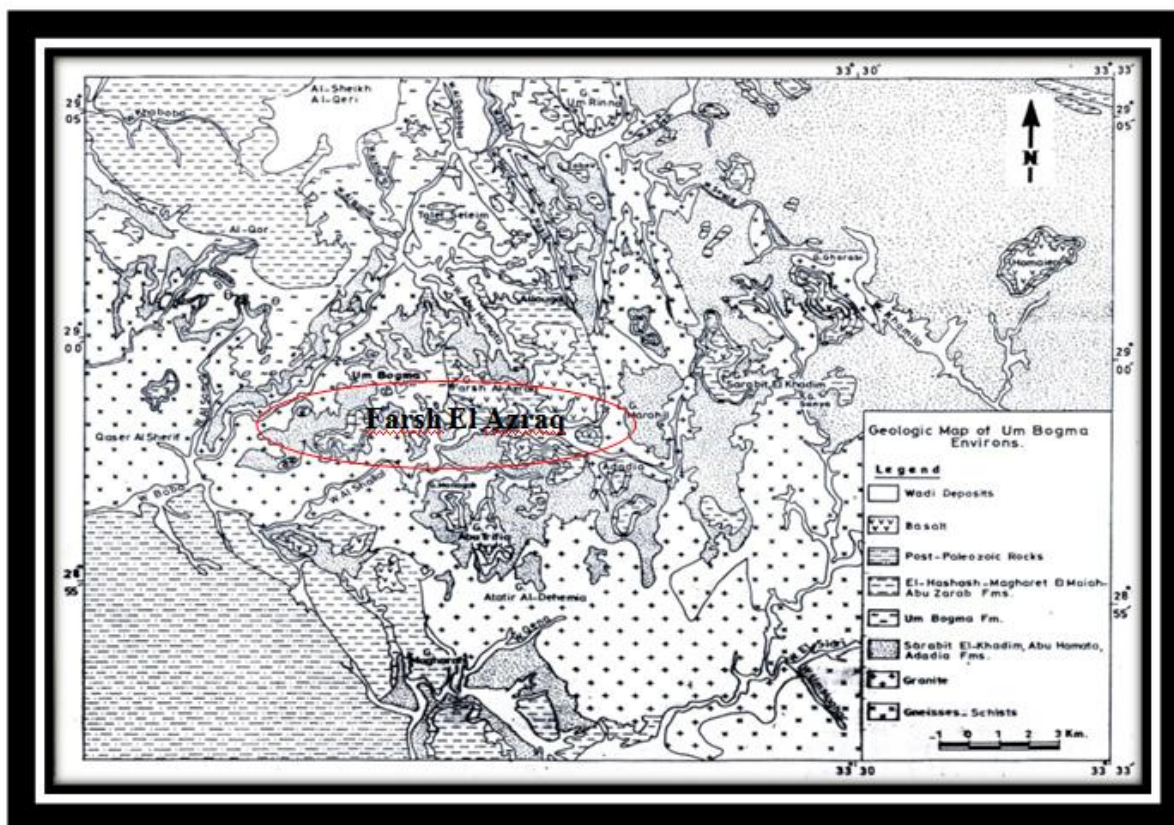
The Um Bogma Formation is the more important formation than the others as it hosts most of the radioactive anomalies and it is subdivided into three members known as:

(1) Lower Shaly-Ore Member comprises black shales with thin sandy dolomite bands and manganese-iron ore. Sharkawi et al. introduced the karstification process in this member and the formation of karstified dolostone

rocks with intrakarstic product housing manganese ores and caliche nodules [El Sharkawi M et al., 1989]. Aassy et al. introduced the lateritic profile section and the formation of gibbsite-bearing shale. This rock unit is highly radioactive when it is karstified and lateritized [El Aassy et al., 1999].

(2) Middle Marly Dolostone-Siltstone Member is also karstified and lateritized and consists of marl with siltstone and gibbsite-bearing siltstone. Its thickness is 6–8 m and moderately radioactive.

(3) Upper Dolostone Member is unconformably overlying the karstified and lateritized soil profile and consists of bedded dolostone with thin shale interbeds. The dolostone beds are present as step-like forms and in some parts are not deposited and laterally validated to grey clay stone [Ibrahim E. El – Aassy, 2011].



**Fig (1) : Geologic map of the studied area in southwestern Sinai, Egypt (Farsh AL-Azraq) [After El Aassy et al., 1986].**

## 2.2. Sampling

In this technique 3 samples, each sample represent (average 3 samples (A,B,C) where, " A-Base part, B-Middle part , C- Upper part" of the layer formation), therefore totally 9 samples of different types of sedimentary rocks collected from Farsh El Azraq area in Sinai as shown in **Table (1)** . These samples were prepared for  $\gamma$  – Ray spectrometric analyses by HPGe detector where the samples first dried, crushed and sieved through -60 mesh size. Weighted samples were placed in polyethylene bottles of 250 cm<sup>3</sup> volume. The bottles were completely sealed for more than one month to allow radioactive equilibrium to be reached before measured by the gamma spectrometer. This step was necessary to ensure that radon gas is confined within the volume and the daughters still also remain in the sample. Each sample was measured during an accumulating time 70000 sec. After each sample counting, an empty cylindrical polyethylene beaker was placed in the detection system, for a counting period of 70000 sec, in order to collect the background count rates. About 5 g of each sample was ground to -200 mesh for chemical analyses of major oxides and trace elements content, by the X-ray fluorescence technique (XRF).

**Table 1: Samples name, type and Description**

Sample name	Sample type	Description
FAZ 3	Siltstone (middle member)	Variiegated in colour, ferruginous, medium hard with Mn pockets.
FAZ 4	Siltstone (middle member)	Variiegated in colour, ferruginous, medium hard with kaolin at the base, yellow with black patches with sandstone lens, brown and coarse grained.
FAZ 7	Siltstone (middle member)	Reddish brown, highly ferruginous, medium hard with Mn pockets.

## 2.3. Radioactivity Measurements

### 2.3.1. Measurement Setup

High purity vertical germanium detector (HPGE) was coupled to a PC-computer with a special electronic card to make it equivalent to a multi-channel analyzer (MCA). The detector used has a single crystal of germanium with length 47.1mm, and crystal diameter 49.3mm. The system also contains the usual electronic components of preamplifier, amplifier and power supply. The detector was surrounded by a special heavy lead shield of about 10 cm thickness with inside dimension 28 cm diameter 40.5 cm height. The absolute detection efficiency of the HPGe detector was determined by using three well-known reference materials obtained from the International Atomic Energy Agency for U, Th and K activity measurements: RGU-1, RGTh-1 and RGK-1 [IAEA, 1987, Anjos et al., 2005]. The sample containers were placed on top of the detector for counting. The same geometry and size were used for both the samples and the reference materials [Pekala et al., 2010].

### 2.3.2. Data Analysis

The primordial  $^{238}\text{U}$  is the most abundant isotope of U (99.27 %) .It decays to  $^{234}\text{Th}$  with the emission of the  $\alpha$ - particle. Through two consecutive  $\beta$  - transitions,  $^{234}\text{Th}$  decays to  $^{234}\text{Pa}$  (half-lives of 24.10 days and 6.69 h, respectively) and to  $^{234}\text{U}$ , with the half-life time of 245,250 years, which decays to  $^{230}\text{Th}$  [Pekala et al., 2010]. Uranium-238 activity was determined indirectly from the gamma rays emitted by its daughter products ( $^{234}\text{Th}$  and  $^{234\text{m}}\text{Pa}$ ) whose activities are determined from the 63.3 and 1001 KeV photopeaks, respectively. The gamma-ray transitions of  $^{228}\text{Ac}$  (338.4, 911) KeV,  $^{212}\text{Bi}$  (727.3 KeV) and  $^{208}\text{Tl}$  (583.1 KeV) were used to evaluate the specific activity of  $^{232}\text{Th}$  [Technical Reports Series, 1989].

$^{226}\text{Ra}$  activity concentration was measured from 186.1 KeV after the subtraction of the 185.7 KeV of  $^{235}\text{U}$ . The concentrations of  $^{214}\text{Pb}$  and  $^{214}\text{Bi}$  were measured from (295.1, 351.9) KeV and (609.3, 1120.3 ,1238.1, 1764.5) KeV. For the actinium series gamma energies of 143.8 KeV, 163.4 KeV, 185.7 KeV and 205.3 KeV were taken to represent the  $^{235}\text{U}$  concentrations.  $^{40}\text{K}$  was determined directly from the 1460 KeV peak energy.  $^{234}\text{U}$  activity was determined directly from the gamma rays emitted from this nuclide at energies of 53.2 keV and 120.9 keV. [Technical Reports Series, 1989].

The net count rate under the most prominent photo peaks of radium and thorium daughter peaks is calculated from respective count rate after subtracting the background counts of the spectrum obtained for the same counting time. Then the activity of the radionuclide is calculated from the background subtracted area of prominent gamma ray energies. Activity of radium, thorium and potassium is calculated using the following equation:

$$\text{Activity (Bq/Kg)} = (\text{CPS} * 1000) / (\xi \times M \times I_{\gamma}) \quad (1)$$

Where CPS is the net count rate per second,  $\xi$  is counting the efficiency of the detector, M is Mass of sample in gram and  $I_{\gamma}$  is Intensity of the gamma spectral. The permissible activity levels for all types of sedimentary rocks are 32, 33, 45 and 412 Bq/kg for  $^{238}\text{U}$ ,  $^{226}\text{Ra}$ ,  $^{232}\text{Th}$ , and  $^{40}\text{K}$  respectively [UNSCEAR, 2010 ; El-Bahi S.M et al., 2017].

## 2.4. XRF Technique

The chemical analysis of major and trace elements in samples was carried out by using the X-ray fluorescence technique (XRF) .Practically the sample preparation is the most important step of any analytical technique. The solid samples were smashed to powder, by series of crushing steps to reduce it to an average grain size of a few millimeters [Raqual, 2006].

The X-ray fluorescence technique (XRF) using Philips Unique II unit fitted with an automatic sample changer PW 1510 (30 position), connected to a computer system using X-40 program for spectrometry. The detection limit of the measured elements by XRF technique was estimated to be 2 ppm for Rb,Nb,Ga,Y and Sr , 8 ppm for Pb and Cu and 5 ppm for other measured trace elements.

The trace elements concentrations are calculated from the programs calibration curves which were set up according to standards material as G-2 and GSP-1. The trace elements were measured by calibrating system under the conditions of Rh-target tube,LiF-420 Crystal,gas flow proportional counter (GFPC) ,Coarse collimators, vacuum, 30 kv and 40 mA for the determination of V,Cr,Ni,Cu,Zn and Ga, 70kv and 15 mA for Rb,Sr,y,Zr and Nb and 100kv and 10mA for the determination of Ba and Pb [R.Viswanathan,1989].

## 2.5. Leaching Procedure

The  $\gamma$  -measured samples were poured from the containers and prepared for leaching experiments, using HCL (Lab. grade) acid leaching on 150g sample weight under the conditions; solid/liquid ratio 1: 3, acid concentration (110gm/L), stirring time three hours and at room temperature. Filtration was carried out to separate leachate from residual which is dried at room temperature. The residual and leachate have been weighted and packed will in polyethylene bottles of 250 ml volume and stored for more than a month for measuring by HPGe detector. The leachability of the radionuclides was calculated according to the following equation:

$$\text{Leachability \%} = \frac{\text{Activity concentration in leachate}}{\text{Activity concentration in original}} \times 100 \quad (2)$$

### III. Results and Discussion

#### 3.1. Geochemical Characterization of Samples

Three sedimentary rocks samples ( FAZ 3 , FAZ 4 and FAZ 7 ) were prepared and analyzed chemically for determination of major oxides order of a (%) by XRF analysis in Nuclear Materials Authority laboratories is shown in **Table (2,3)**. The distributions of these components are of significant values of SiO<sub>2</sub> , Al<sub>2</sub>O<sub>3</sub> , Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> , CaO , MgO , Na<sub>2</sub>O , K<sub>2</sub>O , P<sub>2</sub>O<sub>5</sub> and L.O.I (loss on ignition), From **Table (2)**, the sample (FAZ 3) has high level of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and L.O.I which contents are 45.9, 20.49, 18.95 and 9.55 % respectively . The sample (FAZ 4) has high level of SiO<sub>2</sub> , Fe<sub>2</sub>O<sub>3</sub> and L.O.I which contents are 43.76, 17.78 and 15.75 % respectively . The sample (FAZ 7) has high level of SiO<sub>2</sub> , Al<sub>2</sub>O<sub>3</sub> and L.O.I which contents are 56.43, 12.34 and 11.9% respectively.

**Table (2): The concentration of the Major elements in (%) for the samples.**

Major Oxides	FAZ 3 %	FAZ 4 %	FAZ 7 %
SiO <sub>2</sub>	45.9	43.76	56.43
Al <sub>2</sub> O <sub>3</sub>	20.49	13.85	12.34
Fe <sub>2</sub> O <sub>3</sub>	18.95	17.78	11.13
Ti <sub>2</sub> O	0.55	0.65	0.75
CaO	1.62	3	2.94
MgO	0.2	0.72	0.89
Na <sub>2</sub> O	0.67	1.35	0.75
K <sub>2</sub> O	0.88	1.14	1.45
P <sub>2</sub> O <sub>5</sub>	0.93	0.89	0.85
L.O.I	9.55	15.75	11.9

From **Table (3)** It's clear that, The concentrations of trace elements in sedimentary rock samples at Farsh El Azraq Area ( FAZ 3 , FAZ 4, FAZ 7 ) .The concentrations of **Cr** (Chromium) vary from 314 to 370 ppm , **Cu** (Copper) vary from 283 to 2566 ppm , **Ni** (Nickel) range from 558 to 888 ppm , **Zn** (Zinc) range from 6290 to 10000 ppm , **Zr** (Zirconium) vary from 343 to 633 ppm , **Sr**( Strontium ) varyfrom 90 to 153 ppm , **Y**( Yttrium) vary from 134 to 249 ppm , **V**(Vanadium) vary from 183 to 256 ppm, **Pb** (Lead) range from 134 to 189 ppm and **Ba** (Barium) vary from 1820 to 2136 ppm . Where, these Results shown in **Fig (2)**.

**Table (3): The concentration of the trace elements in (ppm) and Rare earth for the selected samples.**

Element	FAZ 3	FAZ 7	FAZ 4
Cr	352	370	314
Cu	1402	283	2566
Ni	888	558	713
Zn	10000	6290	7930
Zr	357	633	343
Ga	11	22	u.d
Sr	92	153	90
Y	226	134	249
Rb	6	40	5
V	183	256	213
Nb	1	2	1
Pb	134	189	175
Ba	1979	1820	2136
RREs	1884.5	1639.25	1906.25

#### 3.2. Radiometric Measurements and Leaching Experiments

The results of  $\gamma$ -detection of radionuclides in the original samples (Bq/Kg), residuals (Bq/Kg) and leachates (Bq/l) are collected in **Table (4)**. The activity concentration of <sup>238</sup>U in the original samples, residuals and pregnant solutions are higher than the typical world average value of 33 Bq/Kg [UNSCEAR, 2010]. On the other hand the behavior of <sup>226</sup>Ra in the acidic leaching is completely different from <sup>238</sup>U and its isotopes. It is almost lower than the leachability of <sup>238</sup>U and <sup>234</sup>U under the same conditions in all samples. So most of the <sup>226</sup>Ra remains in the leachate ore (residuals) [Fernandes et al., 2006]. The behavior of each radionuclide in the samples shows that the <sup>238</sup>U, <sup>226</sup>Ra, <sup>214</sup>Pb and <sup>214</sup>Bi and <sup>40</sup>K activity concentrations are near equally in the Summation of Residual and Solution compare with the original for all samples. The activity concentration of <sup>226</sup>Ra in the original samples, residuals and pregnant solutions are higher than the typical world average value of 50 Bq/Kg, [UNSCEAR, 2010] except the pregnant solutions FAZ 4. For <sup>232</sup>Th, all the original samples and the residuals are

higher than the permissible level 50 Bq/Kg. For <sup>40</sup>K, all the original samples and the residuals are lower than the permissible level 500 Bq/Kg except the original samples FAZ 7 .The activity concentration of <sup>232</sup>Th and <sup>40</sup>K for all the pregnant solutions are lower than the typical world average.

Figure (2): The concentration of trace elements by XRF in the selected samples from Farsh El Azraq Area

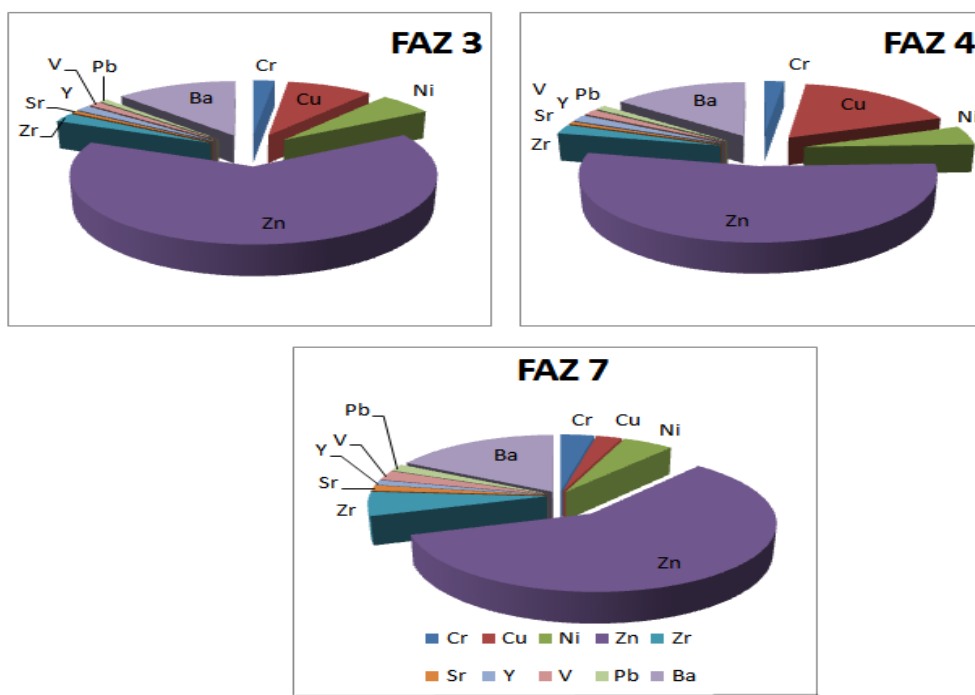


Table (4): Activity concentrations of radionuclides in original samples, residuals and leachates (pregnant solution) in sample FAZ 3, FAZ 4 and FAZ7

Sample FAZ 3					
Radionuclide	Original (Bq/kg)	Residual (Bq/kg)	Solution (Bq/l)	Leachability (%)	Summation (Residual + Solution)/ (Original) (%)
<sup>238</sup> U series					
<sup>234</sup> Th	2286.51	1958.2	295.9	12.94	98.6
<sup>234m</sup> Pa	2762.25	2355.57	354.13	12.8	98.11
<b>Average</b>	<b>2524.38</b>	<b>2156.885</b>	<b>325.015</b>	12.9	98.3
<sup>234</sup> U	2872.11	<b>2113.59</b>	<b>547.35</b>	19.1	92.65
<sup>226</sup> Ra subseries					
<sup>226</sup> Ra	3918.15	<b>3740.13</b>	<b>56.55</b>	1.44	96.90
<sup>214</sup> Pb	1317.88	1237.2	32.75	2.48	96.3
<sup>214</sup> Bi	1105.13	1036.03	34.18	3.09	96.84
<sup>235</sup> U	<b>117.309</b>	<b>100.58</b>	<b>14.97</b>	<b>12.8</b>	<b>98.50</b>
<sup>232</sup> Th series					
<sup>228</sup> Ac	71.5810	61.5738	8.71	12.16	98.18
<sup>208</sup> Ti	78.79	68.39	9.31	11.8	98.62
<b>Average</b>	<b>75.1855</b>	<b>64.9819</b>	<b>9.01</b>	<b>11.98</b>	<b>98.41</b>
<sup>40</sup> K	<b>410.67</b>	<b>246.4</b>	133.1	32.41	92.41
<sup>238</sup> U/ <sup>235</sup> U	21.5	21.44	21.7		
<sup>234</sup> U/ <sup>238</sup> U	1.137	0.9799	1.68		
<sup>226</sup> Ra/ <sup>238</sup> U	1.55	1.73	0.17399		

Table (4): Continue.

Sample FAZ 4					
Radionuclide	Original (Bq/kg)	Residual (Bq/kg)	Solution (Bq/l)	Leachability (%)	Summation (Residual + Solution) / (Original) (%)
<b><sup>238</sup>U series</b>					
<sup>234</sup> Th	2147.94	1792.26	317.73	14.792312	98.233190
<sup>234m</sup> Pa	2043.9	1705.76	318.47	15.581486	99.037624
<b>Average</b>	<b>2095.92</b>	<b>1749.01</b>	<b>318.1</b>	<b>15.177106</b>	<b>98.62542463</b>
<sup>234</sup> U	<b>2066.39</b>	<b>1649.8</b>	<b>363.14</b>	<b>17.573642</b>	<b>97.41336</b>
<b><sup>226</sup>Ra subseries</b>					
<sup>226</sup> Ra	<b>4075.39</b>	<b>3643.668</b>	<b>30.62</b>	<b>0.7513391</b>	<b>90.157948</b>
<sup>214</sup> Pb	1478.58	1385.4	17.19	1.1626019	94.860609
<sup>214</sup> Bi	1517.92	1447.37	15.79	1.0402392	96.392431
<sup>235</sup> U	<b>103.34</b>	<b>82.68</b>	<b>15.56</b>	<b>15.057093</b>	<b>95.064834</b>
<b><sup>232</sup>Th series</b>					
<sup>228</sup> Ac	61.4	51.5	7.81	12.719869	96.596091
<sup>208</sup> Ti	64.5	53.8	7.99	12.387596	95.798449
<b>Average</b>	<b>62.95</b>	<b>52.65</b>	<b>7.9</b>	<b>12.549642</b>	<b>96.187450</b>
<sup>40</sup> K	<b>493.6</b>	<b>378.58</b>	<b>81.4</b>	<b>16.4910859</b>	<b>93.18881686</b>
<sup>238</sup> U/ <sup>235</sup> U	20.28178827	21.1539671	20.44344473		
<sup>234</sup> U/ <sup>238</sup> U	0.985910722	0.943276482	1.141590695		
<sup>226</sup> Ra/ <sup>238</sup> U	1.944439673	2.083274538	0.096259038		

Sample FAZ 7					
Radionuclide	Original(Bq/kg)	Residual(Bq/kg)	Solution(Bq/l)	Leachability (%)	Summation(%) (Residual + Solution)/ (Original) (%)
<b><sup>238</sup>U series</b>					
<sup>234</sup> Th	724.8	485.9	186.6	25.745033	92.78421634
<sup>234m</sup> Pa	709.4	536.1	173.7	24.485481	100.0563857
<b>Average</b>	<b>717.1</b>	<b>511</b>	<b>180.15</b>	<b>25.122019</b>	<b>96.38125784</b>
<sup>234</sup> U	955.9	546.37	<b>282.6</b>	29.563762	86.72141437
<b><sup>226</sup>Ra subseries</b>					
<sup>226</sup> Ra	842.46	<b>641.6</b>	<b>124.31</b>	14.755597	90.91351518
<sup>214</sup> Pb	538.5	478.6	15.44	2.8672238	91.74373259
<sup>214</sup> Bi	695.1	612.8	28.6	4.1145159	92.27449288
<sup>235</sup> U	33.9	23.8	<b>8.2</b>	24.188791	94.39528024
<b><sup>232</sup>Th series</b>					
<sup>228</sup> Ac	63.6	51.49	11.41	17.940252	98.89937107
<sup>208</sup> Ti	66.1	48.72	13.33	20.166415	93.87291982
<b>Average</b>	<b>64.85</b>	<b>50.105</b>	<b>12.37</b>	<b>19.074788</b>	<b>96.33770239</b>
<sup>40</sup> K	<b>612.9</b>	<b>437.74</b>	<b>143.1</b>	<b>23.348018</b>	<b>94.76913036</b>
<sup>238</sup> U/ <sup>235</sup> U	21.15339233	21.47058824	21.969512		
<sup>234</sup> U/ <sup>238</sup> U	1.333007949	1.069217221	1.5686928		
<sup>226</sup> Ra/ <sup>238</sup> U	1.174815228	1.255577299	0.6900361		

#### IV. Conclusions

It is noticed that, there is a difference in  $\gamma$ -activity between the summation of activities of residual and pregnant solution with the activity of the original sample. This phenomenon is varied in magnitude within the different radionuclides. The type of sample plays its role in these variations. Sometimes, there is an attenuation of the gamma activities during the leaching process, due to the presence of high Pb concentration.

The behavior of radionuclides (<sup>238</sup>U, <sup>235</sup>U, <sup>234</sup>U, <sup>226</sup>Ra, <sup>214</sup>Pb, <sup>214</sup>Bi, <sup>232</sup>Th and <sup>40</sup>K) was studied during the acid leaching process; On the other hand the behavior of <sup>226</sup>Ra in the acidic leaching is completely different from <sup>238</sup>U and its isotopes. It is almost lower than the leachability of <sup>238</sup>U and <sup>234</sup>U under the same conditions in all samples.

The nuclides before <sup>226</sup>Ra in the <sup>238</sup>U decay series are easily released in the pregnant solutions than the <sup>226</sup>Ra itself and the nuclides after it (<sup>214</sup>Pb and <sup>214</sup>Bi), these are concentrated in the residuals. The <sup>235</sup>U has nearly the same leachability as <sup>238</sup>U, while <sup>232</sup>Th is immobile except in the presence of organic matters.

In the present work, the sedimentary rock samples from Farsh El Azraq area in Sinai are characterized by a high concentration of Zn and Ba , accordingly we can separate in order to used in different important industries.

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