Elemental Distribution in Rock Samples, Wadi Baba, Southern Sinai, Egypt

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Abstract: Scanning electron microscope (SEM) with energy dispersive X ray Energy Dispersive X ray (EDX) was applied to search for Uranium, Thorium, as well as other elemental constituents of the studied rock samples. Ten samples of West of Wadi (valley) Baba, Southern Sinai, Egypt were collected for this study. These Samples varied in composition between calcareous sandy, dolomite, argillaceous and stone and ferruginous shale and clay stone. Spot analysis from these samples showed that the major elemental constituents matches with the types of the collected samples. The identified trace elements include Uranium (U), Th, Mo, Zn, In, and Tl.
[Afaf A. Fakeha. **Elemental Distribution in Rock Samples, Wadi Baba, Southern Sinai, Egypt.** *Life Sci J* 2015;12(10):70-76]. (ISSN:1097-8135). http://www.lifesciencesite.com. 8

Key Words: Sinai, SEM, EDX, Uranium, Thorium, rare earth.

Introduction:

Several locations in Southern Sinai, Egypt were studied; as the area contains minerals belonging to Manganese, Zin, Iron, REE's, Copper and Uranium (1-7). Some of these localities are not productive zones for Mn-Fe ores, while others are productive on Um Bogma and Wadi Baba. The section in the study area is represented by lower dolostone with shale interbeds, middle member of siltstone and sandy dolostone (6).

In some parts on the lower and middle members are noticed emplaced with soil sediments (laterites).

Most of the studies were about Manganese mineralization (2). The lower shaley and sandy dolo stone member was carstified and filled with sediments contain fossil wood carbonareous materials which adsorbed some Zinc, Manganese, Lead and Uranium.

This study will concern with the samples from the lower and middle members, by applying SEM and EDX (8).

The used SEM and EDX is JEOL-JSM-6S10 LV. This procedure is fast and reliable.

This analysis was carried on the Safeguard Laboratories and, Nuclear and Radiation Regulatory Authority NRRA, Egypt.

Sampling Area:

Samples were collected from Um Bogma Formation of South West Sinai, Egypt (Fig.1), the studied sedimentary rocks (Fig.1) are belonging to the lower Carboniferous Um Bogma Formation (360 m.y., Tucker, 2003). This formation contains several rock types (Table 1). These different rock types show variation in radioactivity according to variation in lithology and alteration processes that carried out. The sandy dolostone is formed by dolomitization process, while the gibbsite and gibbsite- bearing shale were formed by lateritization process. The first process (dolomitization) is accompanied by introduction of uranium, while the secondary process (lateritization) is followed by escape of uranium (El Aassy et al. 2011)[7], although the produced sediments (gibbsite) is favorable to adsorb uranium from any passing ferous solution scarier. The most important rock types are dolo stone, gibbsite, ferruginous, siltstone and clay.

Samples' preparation:

In order to increase their homogeneity for analysis by SEM and EDX, the assayed were ground to 200 mesh and sieved, small quantities with reasonable size and shapes were taken to get representative specimen, which should not exceed holder diameter for the measurements. The specimen was fixed on Carbon tap, pasted on the holder-using spatula**.**

Instrumental

The measures were carried by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX). The SEM used in this study is a JEOL JSM-650LV model with resolution of 1pÅ to 1µÅ. Scanning Electron Microscope (SEM) and Energy Dispersive X ray (EDX) spectrometer is employed for the measurement. The major parts of SEM/EDX are: vacuum specimen **chamber, electron column** with electron source, electromagnetic lenses, scanning coils and signal detectors (SE,BSE, EDS).

A number of signals such as Secondary Electrons (SE), backscattered electrons (BSE, and X rays result from interactions of a focused scanning electron beam with the atoms of a specimen thus providing different information about the sample, Sameh E. Shaaban, et al (2013)[9].

The specimen was placed inside the holder and it was fixed inside the instrument at a working distance 10 mm (WD10mm), Voltage=30KV, magnification value=500 and spot size Quantum=50. Quant method was used for analysis.

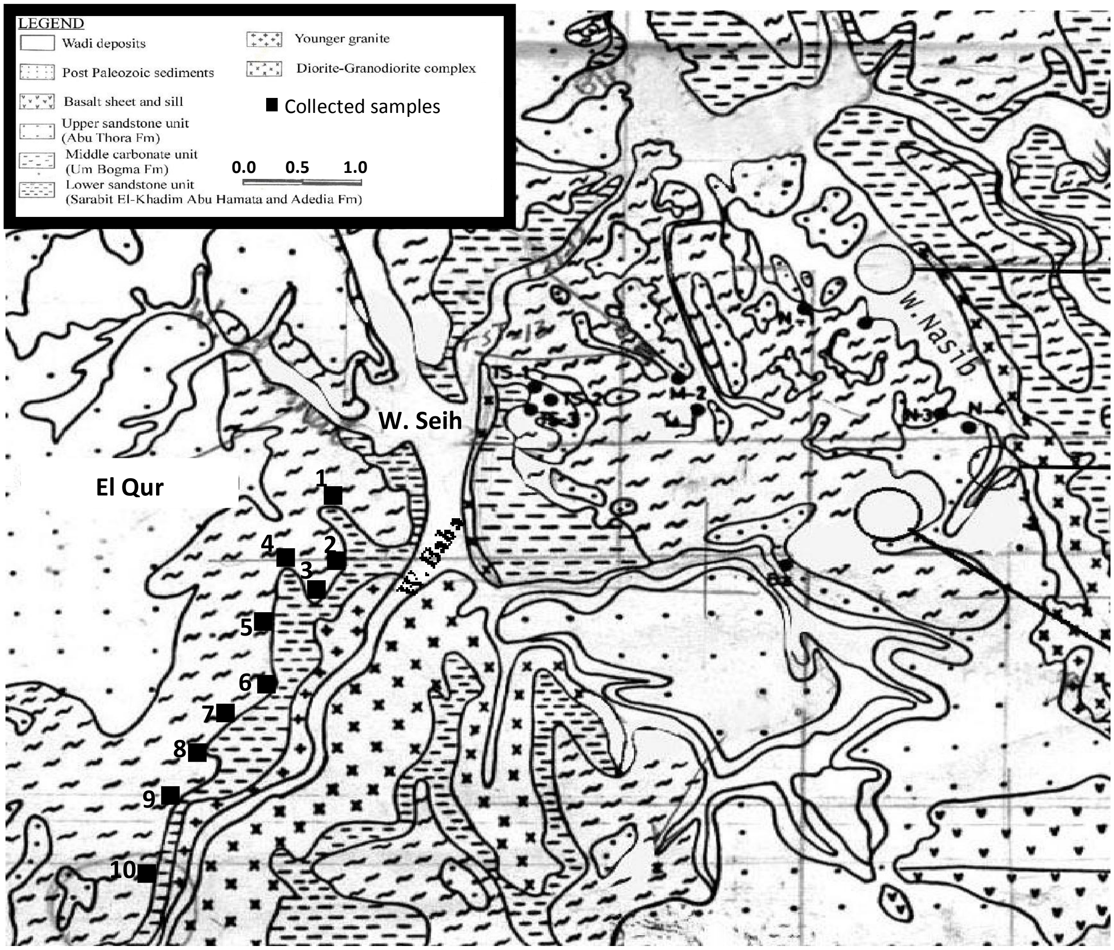


Fig. 1: Geologic and Sample Location Map

Table (1):Description of Collected Samples

Sample Code	Description
1	Sandy dolostone, medium hard to hard, grey.
2	Iron lateritic soil brown to red and grey, soft.
3	Calcareous ferruginous, red, medium hard with pebbles.
4	Sandy Ferruicrete, soft, brownish yellow.
5	Ochre, soft to medium hard, yellowish brown.
6	Ferruginous limestone, soft to medium hard, brown.
7	Ferri and silcrate, soft, brown
8	Ferruginous siltstone, soft to medium hard.
9	Conglomeratic, ferruginous, pale brown, medium hard with evaporites.
10	Ferruginous siltstone, creamy, soft to medium hard.

In EDX, with decreasing concentration, statistical errors and uncertainities in background corrections become dominant. For concentrations in the region of 100 ppm the intensity measured on the peak consists mainly of background. The smallest detectable peak may be defined as three times standard deviation of the background count. An order of magnitude detection limit estimate can be obtained. If the count rate for a pure element is 1000 s^{-1} and the peak to background ratio is 500/1, the background count rate is 2 counts s^{-1}, in 100 s a total of 200 background count will be accumulated, giving a relative standard deviation of (200/200) or. 0.07. Since the background intensity in this case is equivalent to the peak count rate. For concentration of1000ppm, three standard deviations is thus equivalent to a concentration of 0.07 x 3x1000=212 ppm. Reducing the detection limit requires more counts which can be obtained by increasing, the counting time and/or the beam current. In EDX analysis, detection limits are typically about 0.1 wt. 100%, although reduction can be achieved by using long counting times or better count rate using SSD detectors (3).

The results were obtained from two or three different points on the specimen, as the sample is not perfectly homogenized; the error is calculated by the least square method. This procedure is fast and reliable, so it can be applied in contains organic materials specially the carbonaceous matter which play an important role in the distribution of nuclides within soils and sedimentary rock.

Results and Discussion

The distributions of major elements were identified through SEM with EDX for one area of the sample or noticed in sample(1) which is sandy dolostone. This sample composition (Fig.2) is 37% ca, 6.2 % Mg and 12 % Fe. The well noticed thing is the Mo which shows abnormal content (12.8 %) although it is known as trace element, but her is not. The same thing is noticed in sample No. 2 (Fig.3) which is iron lateritic soil (55.8% Fe.) and contain the trace element Thalium (Tl) is 2.3 %, which represents a major constituent in this sample.

The previous abnormalities was not noticed either in sample No.3(calcareous sandy ferricrete) which consist mainly 67.3 %, Fe., 9.3 % Si and3.11% Ca, or in sample No.4 (sandy ferricrete) which contains 38% Fe., 3.11 Ca, or in sample No.4 (sandy ferricrete) which contains 38% Fe.

Table 2: The percentage average concentration for the different elements

Average concentrations percent ± error								
Sample Code / Element	Mg.	Al.	Ca.	Fe.	Zn.	Mo.	Th.	U.
1	5.72 ± 0.012	0.75 ± 0.001	27.40 ± 0.207	1.30 ± 0.035	-	-	0.74 ± 0.060	2.60 ± 0.065
2	3.79 ±0.021	4.58 ± 0.069	2.17 ± 0.065	51.57 ±0.342	-	-	0.77 ± 0.023	2.05 ± 0.036
3	-	7.96 ± 0.098	6.33 ±0.040	29.41 ±0.029	-	-	2.45 ± 0.091	2.05 ± 0.037
4	7.86 ±0.095	10.15 ± 0.033	6.37 ± 0.184	18.09 ± 0.316	-	-	-	1.90 ± 0.069
5	1.01 ±0.007	3.47 ± 0.012	1.54 ± 0.003	24.0 ± 0.211	1.40 ± 0.015	-	0.31 ± 0.006	-
6	2.32 ±0.004	1.84 ± 0.003	13.92 ± 0.073	7.70 ± 0.079	1.27 ± 0.005	13.25 ± 0.182	0.52 ± 0.011	-
7	1.92 ± 0.008	9.21 ± 0.028	1.28 ± 0.003	21.96 ± 0.081	4.35 ± 0.037		-	0.72 ± 0.006
8	1.14 ± 0.004	6.96 ± 0. 021	-	5.19 ± 0.023	0.21 ± 0.001	14.18 ± 0.223	-	0.98 ± 0.089
9	0.98 ± 0.025	2.85 ± 0.028	3.64 ± 0.004	3.81 ± 0.026	0.49 ± 0.002	16.12 ± 0.274	2.72 ± 0.010	-
10	-	9.77 ± 0.028	1.32 ± 0.003	5.01 v 0.024	-	9.45 ± 0.256	-	0.88 ± 0.006

The trace elements were identified by the spot analysis of some grains of each samples11.7% Si and 5.8 % Mg (Fig,4 and 5). Mo represents a major constituent (12.4 %), and Zn.(3.8%) in sample No.5 (Fig.6).

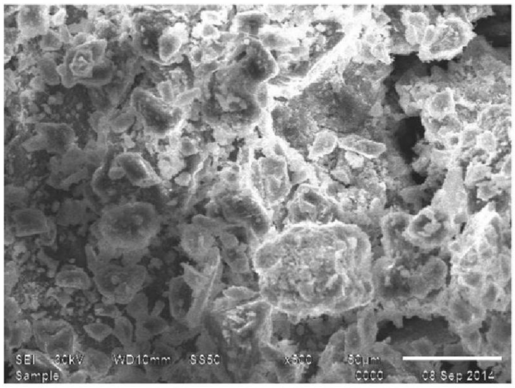
In sample No.6 which is ferruginous limestone (20.6% Ca and 15.1% Fe.) contains also 14.4 %Mo. and 4.6% Zn (Fig.7).

Sample No.7, which is known as ferricrete and silcrete consist of 29.9% Fe with 10.1% Si as main constituent. Zn reached to 8.7%, while Ti is 1.1 % (Fig.8). Again, Mo represents 13.4% in sample No.8 which is ferruginious siltstone and contains 1.5%Si (Fig.9). The higher Mo content 20.5% (Fig.10) is noticed in sample No.9 (conglomeratic, pebbly stone sand), it is ferruginous (16.1 percentage Fe, it contains 9.4% S and 7.4% Ci, with 2.2% K 2.9% Na which may be present as evaporates. Sample No.10 (ferruginous siltstone) contains 26.2% Si and 12.8% Fe (Fig.11), it also originated from either basic and/or acid basement rocks and Th with 1.8 %.

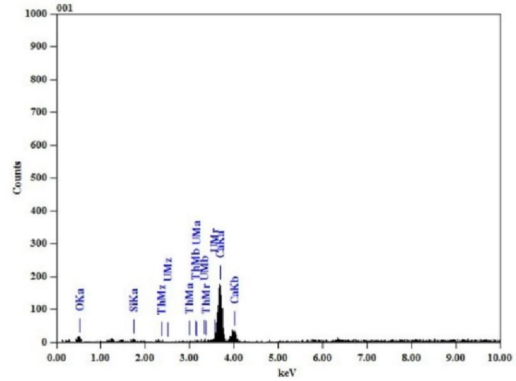
2. Trace elements distribution:

The trace elements were identified by the spot analysis of some grains on each sample. In sample No.1 (Fig.12) uranium was identified in one grain with 3.6% and Th with 1.08%, which means that uranium is 3 times Th, in sample No.2 (Fig.13) uranium was not identified, while Th has 5%, in another grain in the same sample uranium was identified with 1.5%, while Th Is not detected. The same case was noticed in sample No.3 (Fig.14) in which one grain has 0.36 % U without Th, and in other grain it has 19% Th without U. Uranium is also noticed in sample No. 4 with 2.6% U without Th (Fig.15). Both Th and U were identified in one grain of sample No.5 in which Th is very high (9.4%) relative to U (0.3%). In sample No.6 (Fig.16) Th is only noticed wit 0.6%, but Cu has 16%.Th was not noticed in one grain of sample No. 6 (Fig.17), while U has 2.7% of the same grain. The same case was noticed in sample No.8 (Fig.18) and the U has 1.4%, while it has 6.1% in sample No.9 (Fig.19), This noticed only with 2.2% in one grain, while the reverse is noticed in sample No.10 (Fig.20) in which one grain has 2.0% U without Th.

From the above-mentioned data, it is noticed that both U and Th are noticed in the whole samples in the scale of grains with size of about 20 µm.



Fig,1



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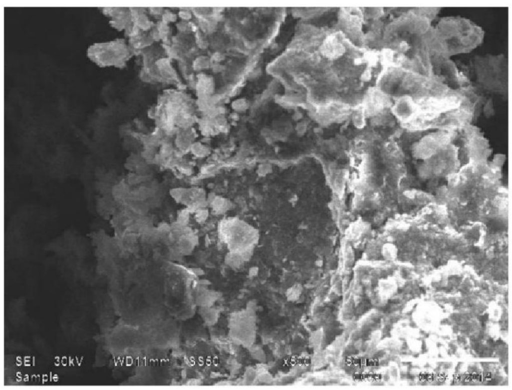

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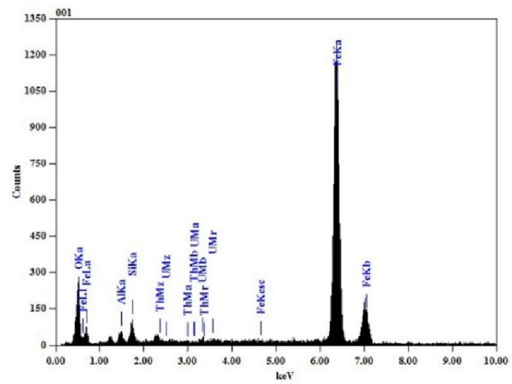

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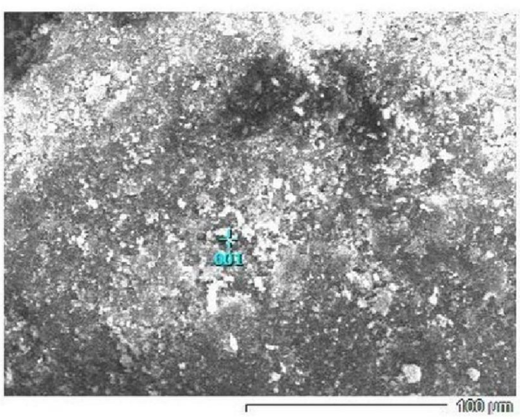
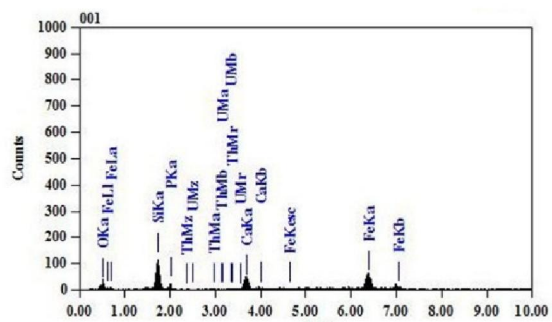


Fig 5



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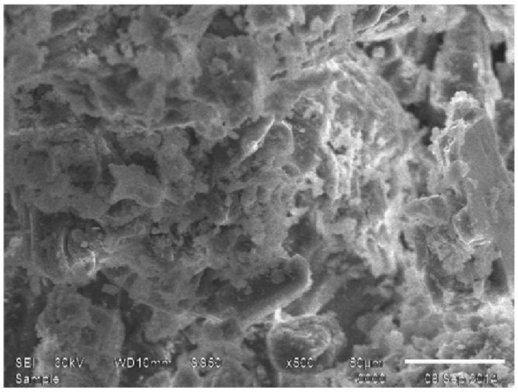
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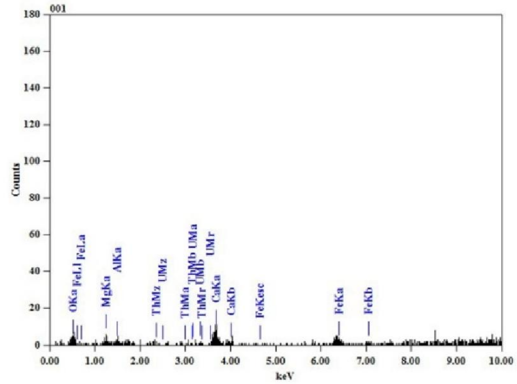

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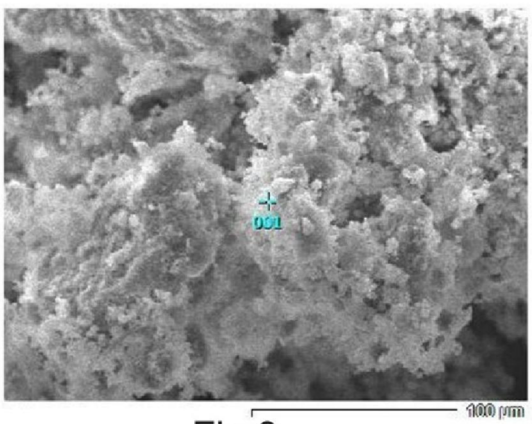

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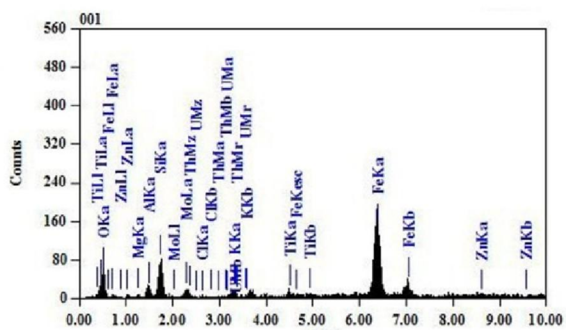
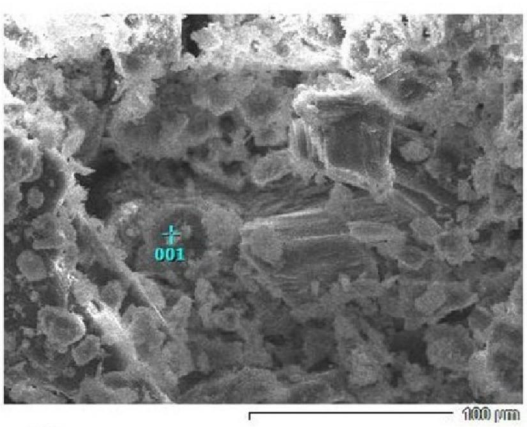
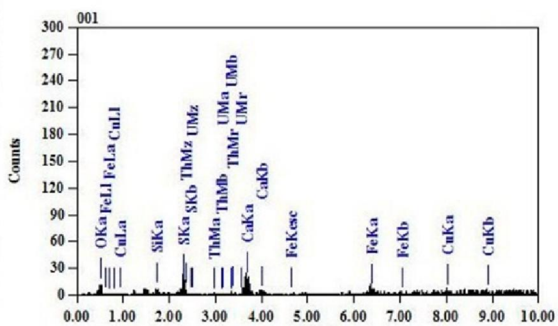


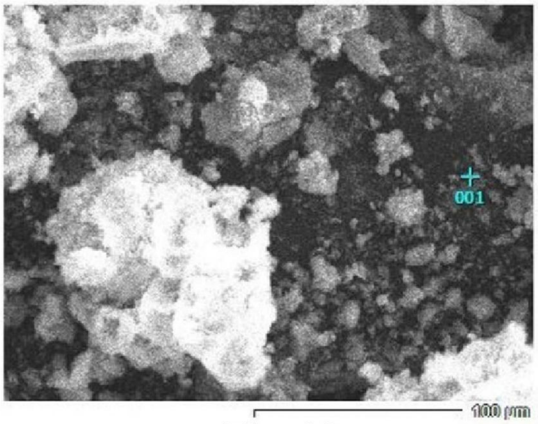
Fig,10



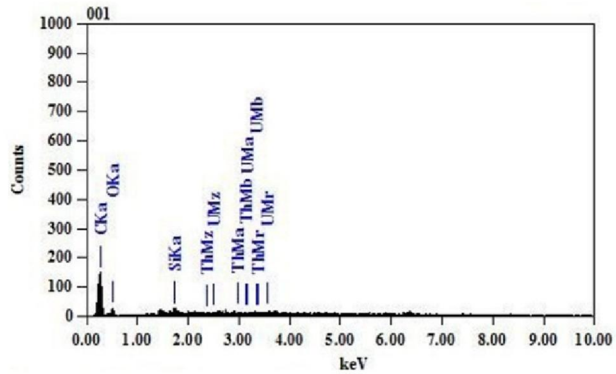
Fig,11



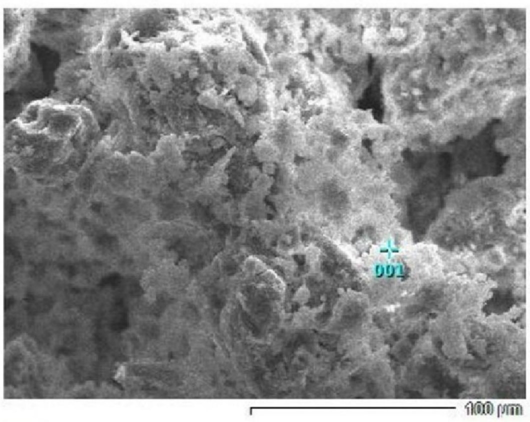
Fig,12



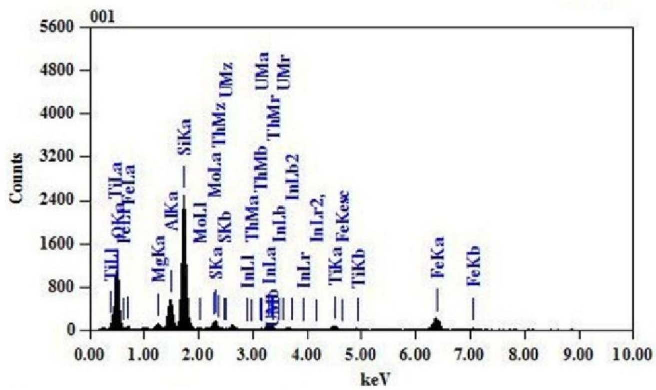
Fig,13



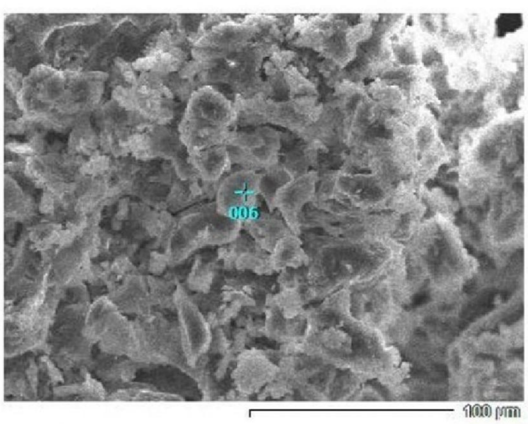
Fig,14



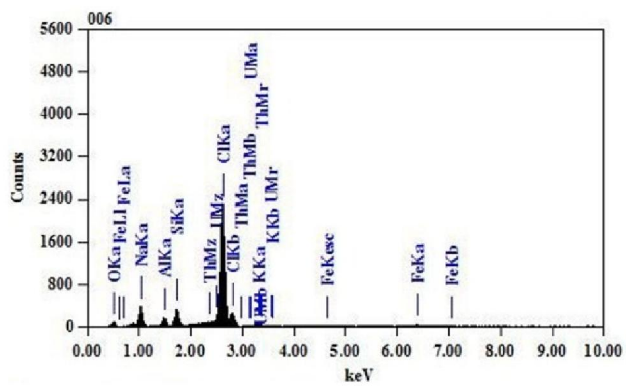
Fig,15



Fig,16



Fig,17



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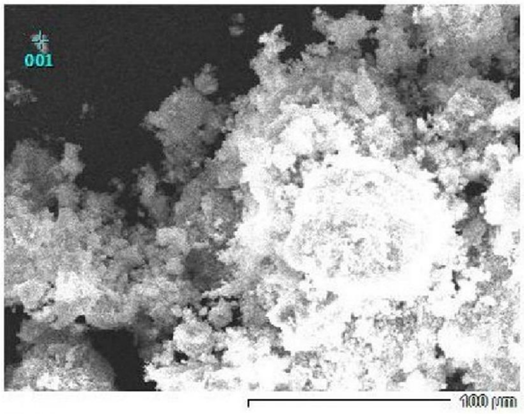
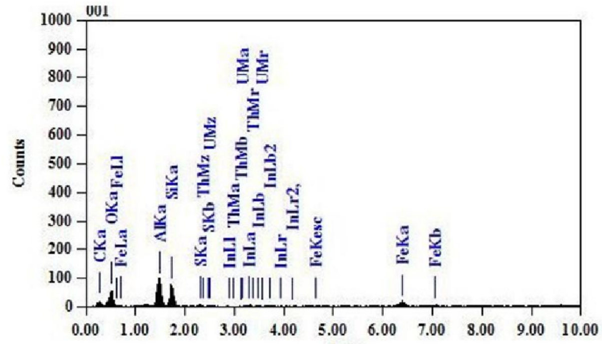


Fig,19



Fig,20

Summary and Conclusion

10 samples from the pafrozoic sedimentary section in southern Sinai were analyzed by SEM with EDX for major elements, and spots for trace elements. It was noticed that Fe, Ca and Si are the prevailed elements. Mo; is also noticed in major scale with Mn, while U and Th were presented in trace scale, Zn and Ti are recorded with minor scales.

From this study, it can be concluded that sediments were originated from either basic and/or acidic basement rocks that were subjected to several processes of weathering and alterations.

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10/2/2015