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Mineralogical and Geochemichal aspects of Nb-Ta oxide minerals from Wadi El Sheih mineralized pegmatite, Central Eastern Desert, Egypt

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ABSTRACT

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1. Introduction

The Egyptian granitic rocks grouped into two major suites. 1- The older granitoids (OG other names: Grey, syn-orogenic or Shaitian granites) are of wide spectrum compositional Quartz-diorite, of tonalite, granodiorite and are of wide distribution in the Egyptian Eastern Desert (EED), representing ~ 26.7% of the basement outcrops (Stern, 1979). 2- The younger granites (other names: pink-, and late to post-orogenic) range from calc-alkaline to per alkaline rocks (El Gaby, 1975) and have been considered as transitional phases from calcalkaline I-type magmatism to normal alkaline and alkaline A-type granites (Noweir et al., 1990). In the last decennium, several significant works were published on the geology and tectonics of Egyptian ANS (e.g. Azzaz et al., 2015; Blasy et al., 2001; Kharbish 2010, 2013, and 2020; Kharbish and El-Awady 2018 and 2019; Kharbish et al., 2021; Saada and Kharbish 2019).

Rare metal mineralization that recorded in EED are mainly restricted to the younger granite bearing pegmatite bodies (e.g. Abd El Wahed et al., 2006; Abdel Warith et al., 2007, Raslan et al., 2010a and b; Raslan and Ali, 2011; Raslan, 2015 and Raslan and Fawzy, 2018).

Pegmatites are important because they often host a diversity of mineral species of great economic interest, and are a source of rare metals such as Be, Sn, Li, Rb, Cs, Nb, Ta and Ti.

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Rare-metals and rare earth mineralization has been observed in Wadi El Sheih older granitoids bearing uncommon mineralized pegmatite. This type of mineralization was recorded as distinguishable mega-crystals scattered within the pegmatite. Detailed mineralogical investigation was carried out using different analytical techniques as: microscopic examinations, x-ray diffraction (XRD), and scanning electron microscope (SEM) providing with energy dispersive spectrometer (EDS) unite. These studies revealed the presence of several valuable minerals like Euxenite and Fergusonite as well as; gives a clear comparative idea on the mineralogical and geochemical characteristics of the Nb-Ta oxides.

In addition, they have been considered a source of rare earth elements (REE) and radioactive elements, such as U and Th. Pegmatites also are a source for high-quality industrial minerals, such as feldspar, kaolinite, quartz, mica, and spodumene that used in ceramic industry. Pegmatites are a source of large variety of highly priced colored gem minerals as topaz, tourmaline, aquamarine, and beryl (Linnen, 2012; London, 2008).

According to Cerny (1990) pegmatite classification, the rare-earth element (REE) subclass is characterized by the niobium-yttrium-fluorine (NYF) and zirconium-niobium-fluorine (ZNF) family signatures. NYF pegmatites are distinguished by the signature Y, Nb> Ta, HREE, U, Th, and F, whereas ZNF pegmatites are distinguished by the signature Zr, Nb>> Ta, Y, Th, P, and F. From the viewpoint of exploration, post-orogenic, A2-type granites are the most favorable localities for rare-metal pegmatitic mineralization of NYF affinity (Abdalla and El Afandy, 2003).

Granite pegmatite bodies associated with the OG in Egypt are very rare, therefore this work is carried out to identify and characterize the mineralogical and chemical characteristics of the rare metals mineralization of the mineralized pegmatite of Wadi El Sheih (WSh) older granitoids at Central EED.

2. Geologic Setting

The investigated WSh area is located within Pan-African basement complex in the central EED. It nearly covers an area about 1.2 km² (Fig.1-A,B&C).The studied

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mineralized pegmatites are located in southern of the mapped area. They occur in two separated areas, along the both eastern and western banks of Qena –Safaga asphaltic road.

The Gray granite is the main rock type occupying the study area, where it covers about the whole map area. The Gray granite is dissected by felsite and pegmatite dykes and bodies. According to the field radiometric measurements, the mineralized pegmatites vary from weak, moderate and high radioactive types. According to field geological investigation and radioactivity measurement studies, the highest radioactive pegmatite type displays as huge large irregular shape pegmatite bodies, up to few tens of meters in size and it has been further distinguished and classified into three subtypes.



Fig.1. Geological map of the studied area Wadi El Sheih, Central Eastern Desert, Egypt

The first highly radioactive pegmatite subtype namely; dark patch habit rare metal mineral subtype, occur as medium body size and has lowest radioactive measurements ranging between 2500 to 5000 cps, compare to this category. It mainly contains potach feldspar, quartz and mica, and fluorite. The second pegmatite subtype is called well-developed crystal habit rare metal mineral subtype. It displays as medium to large body size and has moderately radioactive values (5000-9000) cps. It possesses well mineral habit crystal, potch feldspar, mica and quartz with accessory mineral such as fluorite and iron oxides. While the last and the highest radioactive values mineralized pegmatite subtype is called composite patch and well developed crystal habit radioactive rare metal mineral, and occasionally occurs with fluorite secondary quartz.(Eldabe 2018)

3. Samples and Methods

Four composite samples from different mineralized pegmatite bodies of WSh weighing approximately 10 kg for each were collected for mineralogical investigation. The samples were initially crushed using jaw crusher to prepare a feed about -3mm for the grinding process. Then, the grinding process was taken place using Denver pilot rod mill to reduce grain size 100% passing 1mm. After crushing and grinding processes, dry sieving analysis were take place, then each size fraction and part of the bulk sample were subjected to heavy-mineral separation for the purpose of mineralogical identification and calculation of heavy mineral content relative to gangue light mineral percent. The heavy liquid was used for the purpose of separation is bromoform with specific gravity 2.85. From the obtained heavy fractions, pure monomineralic grains were handpicked and investigated under a stereoscopic microscope. The heavy mineral grains were manually picked from each of the obtained heavy fractions under Olympus stereo binocular microscope. Some of these selective picked grains were analyzed using X-ray diffraction equipment (XRD). PAN analytical x-ray diffraction equipment model X'Pert PRO with Secondary Monochromator, Cu-radiation (λ =1.542A°) at 45 K.V., 35 M.A. and scanning speed 0.04 °/sec. were used. The diffraction peaks between $2\theta = 2^{\circ}$ and 60° . corresponding spacing (d, A°) and relative intensities (I/I⁰) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files. XRD analyses were carried out at Central Laboratories of The Egyptian Mineral Resources Authority.

Selected grains were examined by Scanning Electron Microscope (SEM) includes a Philips XL 30 energydispersive spectrometer (EDS) unit at an accelerating voltage of 30 kV with a beam diameter of 1µm for a counting time of 60-120 s and a minimum detectable weight concentration ranging from 0.1 wt % to 1wt %. Trace element contents were detected using PHILIPS X Unique-II spectrometer PW 1510 (30 positions). This instrument is connected to a computer system using X-40 program for spectrometry. All analyses were carried out at the laboratories of the Egyptian Nuclear Materials Authority (NMA).

4. Results and Discussion

The systematical and mineralogical investigation of the bulk composite sample of the WSh pegmatite sample manifested that, the essential minerals are quartz, and feldspar that represent about 93.4 wt. %. The content of the heavy valuable minerals is approximately 6.6 wt. % that includes the existence of several rare-metal, rare earth and radio minerals; rare-metal are; euxenite-(Y), fergusonite-(Y) (Table 1).

Table 1. Mineralogical composition of Wadi El Sheih

 pegmatite granitoid

Mineral	Weight %	Mineral	Weight %		
Quartz	29.17	Zircon	0.18		
Feldspar	64.23	Xenotime	0.04		
Euxenite+fergusonite	1.56	Iron oxides	1.87		
Allanite-(Ce)	2.66	Muscovite	0.31		

4.1. Euxenite-(Y): [(Y,Ca,Ce,U,Th)(Nb,Ta,Ti)₂O₆]

Euxenite-group minerals is a group of niobiumtantalum oxides that occur in Y, REE-rich pegmatites, with a formula AM_2O_6 , where A site = Y, REE, U, and Th whereas M = Nb, Ti and Ta. Members of the euxenite group include euxenite-(Y), [Y(Nb,Ti)₂O₆], tanteuxenite-(Y), [Y(Ta,Ti,Nb)₂O₆], polycrase-(Y),[Y(Ti,Nb)₂O₆], and uranopolycrase, [UTi₂O₆] (Erict, 2005b). Polycrase-(Y) and euxenite-(Y) are most properly differentiated: if Ti exceeds (Ta + Nb), then the mineral is polycrase-(Y), and the reverse for euxenite-(Y).

Euxenite and aeschynite group minerals have the same general formula of AM_2O_6 , and they are typically metamict, altered, and can be difficult to identify on a structural basis (XRD); consequently, examination of the sample composition may be the most reliable approach to their identification. A working differentiation of the euxenite group from the aeschynite group can be done according to the preliminary calculations of Ewing (1976): the aeschynite group has LREE > 0.326 Ti – 0.060 Nb + 3.1 (oxide wt. %), whereas for the euxenite group, the converse applies. In the present study, the euxenite-(Y) variety has been well identified using microscopic investigation using stereo binocular microscope, and proper analysis by ESEM, field-emission scanning electron microscope, as well as by using XRD analyses.

Stereo microscopic examination of WSh euxenite-Y picked grains revealed that, they are generally massive grains of subhedral to anhedral and granular form with resinous, semi metallic or vitreous luster. Also, the investigated mineral crystals are generally translucent, compact, metamict and hard. Wadi El Sheik euxenite-Y crystals are commonly honey brown while others are dark brown to brownish black in color under the stereoscopic binocular microscope and distributed through all size fractions (Figure 2Aand B).

Several BSE images and EDX spectrum of discrete euxenite-(Y) grains are presented in Figure 2C, D, E, F, G and H. The obtained composition results show that the euxenite grains are enriched in niobium, titanium, yttrium and uranium. Semi quantitative chemical composition of represented ten grains of WSh euxenite-Y are displayed in Table 4.The chemical analyses of euxenite grains give mainly Nb₂O₅ ranging between 12.8 to 40 with average about 26.4 wt. %, TiO₂ ranging between 9.5 to 45 with average of 24.2 Wt. %, Y₂O₃ ranging between 4 to 24 with average of 15.6, and UO₂ ranging between 2.4 to 19.5 with average of 12.3 wt. %.. Rare earth (REE) oxides content for Wadi El Sheik euxenite-Y grains show enrichment in heavy REE (HREE) oxides (3.96 wt. %) relative to light REE (LREE) oxides (0.40 wt. %).

4.2. Fergusonite-(Y): [YNbO₄]

Anhedral to subhedral granular form of fergusonite-(Y) were detected in WSh pegmatite. Most of Fergusonite-(Y)

grains having a characteristic vitreous or resinous luster. Also, they are generally translucent, compact, metamict and hard. The fergusonite crystals are mainly pale to dark yellowish brown in color (Figure 3A and B). SEM data of the studied fergusonite grains (Figure 3C, D, E, F, G and H) reflect an enrichment in niobium, yttrium and HREE than LREE elements. The semi quantitative chemical composition of eight studied fergusonite-Y grains using scanning electron microscope are shown in Table 5.The obtained results in average are revealed that 43.65 % Nb₂O₅, 1.79 % Ta₂O₅, 24.31 % Y₂O₃, 1.11 % ΣLRE_2O_3 and 8.92 % ΣHRE_2O_3 .

The XRD data for euxenite-(Y) and fergusonite-(Y) after annealing for 1 hour at 1000° C is presented in Figure 4. The data confirms to the PDF-2 card no. 5-603 for heated euxenite-Y and PDF-2 card no. 9-443 for fergusonite.

Elemental Oxide	1	2	3	3	4	5	6	7	8	9	10	Min	Max	Avera
Al ₂ O ₃	1.88	2.45	3.36	2.52	1.90	1.90	2.03	3.06	2.58	2.15	1.53	1.53	3.36	2.31
SiO ₂	1.94	3.92	6.10	3.13	3.33	3.84	9.38	4.98	4.89	6.36	0.00	0.00	9.38	4.35
ThO ₂	0.00	2.55	2.57	2.97	0.00	0.00	3.70	5.18	3.36	0.00	3.48	0.00	5.18	2.16
UO ₂	17.84	8.34	11.15	5.42	12.97	17.99	2.42	11.35	11.19	19.48	17.24	2.42	19.48	12.31
Fe ₂ O ₃	2.24	1.01	4.59	3.02	1.20	3.55	7.49	3.33	4.44	3.20	2.59	1.01	7.49	3.33
CaO	0.73	0.57	0.88	0.95	0.65	2.91	4.32	2.42	1.54	3.83	1.96	0.57	4.32	1.89
Pb	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.47	0.00	0.00	0.00	0.00	2.47	0.22
K ₂ O	0.33	0.00	0.27	0.00	0.23	0.00	0.00	0.10	0.00	0.12	0.00	0.00	0.33	0.10
P_2O_5	0.00	0.00	0.00	0.00	0.00	0.00	2.12	0.00	2.01	0.00	0.00	0.00	2.12	0.38
MgO	0.00	1.85	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.85	0.17
Ce ₂ O ₃	0.00	0.00	0.99	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.41	0.00	0.99	0.13
Ho ₂ O ₃	0.00	0.00	0.00	1.81	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.81	0.16
Gd_2O_3	0.89	2.27	0.00	0.00	1.17	0.00	0.00	1.70	1.83	0.00	0.86	0.00	2.27	0.79
Sm ₂ O ₃	0.31	0.00	0.00	1.12	0.00	0.00	0.00	0.00	0.89	0.00	0.00	0.00	1.12	0.21
Nd_2O_3	0.00	0.00	0.00	0.66	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.66	0.06
Eu ₂ O ₃	0.00	0.00	0.00	0.00	0.32	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.32	0.03
Dy_2O_3	2.49	2.95	0.00	0.00	2.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.95	0.68
Er ₂ O ₃	0.00	2.85	0.00	2.71	1.37	0.00	0.00	0.00	2.03	0.00	0.00	0.00	2.85	0.81
Yb ₂ O ₃	3.08	2.94	1.51	0.00	2.29	0.00	0.72	2.63	3.12	0.00	0.00	0.00	3.12	1.48
Y ₂ O ₃	18.39	16.41	17.92	24.43	20.19	16.38	4.00	10.46	14.61	14.04	15.26	4.00	24.43	15.64
TiO ₂	21.89	19.79	17.93	9.51	18.34	20.38	45.09	37.53	26.28	25.45	24.11	9.51	45.09	24.21
ТаО	1.42	3.24	3.21	1.76	1.77	2.49	1.80	1.94	2.11	2.53	2.01	1.42	3.24	2.21
Nb ₂ O ₃	26.56	28.86	29.52	40.00	32.25	30.56	16.92	12.84	19.13	22.81	30.57	12.84	40.00	26.37
ΣLRE ₂ O ₃	0.31	0.00	0.99	1.78	0.00	0.00	0.00	0.00	0.89	0.00	0.41	0.00	1.78	0.40
ΣHRE ₂ O ₃	6.46	11.01	1.51	4.52	7.17	0.00	0.72	4.33	6.98	0.00	0.86	0.00	11.01	3.96



Fig.2. Stereo microscopic images of euxenite-Y grains show the color and shape (A-B). BSE images and EDX spectrum of euxenite-Y grains show their chemical composition (C, D, E, F, G and H).

Table 3. Semi-quantitative chemical analyses of fergusonite-(Y) grains from Wadi El Sheih pegmatite

Elemental oxide	1	2	3	4	5	6	7	8	Min	Max	Average
Al ₂ O ₃	0.93	0.68	1.38	3.13	2.36	1.90	1.23	1.17	0.68	3.13	1.60
SiO ₂	3.44	1.28	3.42	4.64	5.84	2.13	2.39	2.06	1.28	5.84	3.15
ThO₂	12.11	5.63	6.69	3.43	1.74	2.68	6.88	2.81	1.74	12.11	5.25
UO₂	5.44	6.67	7.59	3.18	4.07	3.45	8.84	4.25	3.18	8.84	5.44
Fe ₂ O ₃	1.88	0.59	0.55	0.90	1.57	1.58	4.92	1.71	0.55	4.92	1.71
CaO	1.25	0.65	0.48	0.62	1.44	1.13	1.73	1.21	0.48	1.73	1.06
MgO	0.00	0.00	0.00	2.40	0.00	0.00	0.00	0.00	0.00	2.40	0.30
Pr ₂ O ₃	0.00	0.29	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.29	0.04
Pm ₂ O ₃	0.00	0.37	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.37	0.05
Ho ₂ O ₃	0.95	0.00	0.00	0.00	0.00	0.00	0.00	1.54	0.00	1.54	0.31
Gd ₂ O ₃	2.04	2.84	2.36	2.81	1.64	2.20	1.59	0.00	0.00	2.84	1.94
Sm ₂ O ₃	0.95	1.74	0.00	0.00	0.00	1.00	0.00	0.87	0.00	1.74	0.57
Nd_2O_3	0.00	1.21	1.10	0.00	0.56	0.00	0.43	0.73	0.00	1.21	0.50
Dy_2O_3	3.80	3.33	3.20	4.19	4.05	3.26	0.00	0.00	0.00	4.19	2.73
Er_2O_3	2.45	3.12	2.45	2.85	2.47	0.00	1.37	1.85	0.00	3.12	2.07
Yb ₂ O ₃	2.90	3.43	2.81	2.77	3.08	0.00	0.00	0.00	0.00	3.43	1.87
Y_2O_3	21.26	23.09	22.09	25.13	25.35	29.95	18.35	29.26	18.35	29.95	24.31
TiO ₂	0.94	2.10	2.93	0.89	1.09	1.42	1.92	1.39	0.89	2.93	1.59
TaO	1.46	3.39	2.30	2.10	1.56	1.37	1.17	1.00	1.00	3.39	1.79
Nb ₂ O ₃	38.18	38.20	40.65	40.97	43.19	47.94	49.19	50.15	38.18	50.15	43.56
ΣLRE ₂ O ₃	0.95	3.24	1.10	0.00	0.56	1.00	0.43	1.60	0.00	3.24	1.11
ΣHRE_2O_3	12.14	12.72	10.82	12.62	11.24	5.46	2.96	3.39	2.96	12.72	8.92



Fig. 3. Stereo microscopic images of fergusonite-Y grains show the color and shape (A&B). BSE images and EDX spectrum of fergusonite-Y grains show their chemical composition (C, D, E, F, G and H).



Fig. 4. XRD pattern for euxenite and fergusonite of Wadi El Sheih pegmatite sample

Conclusion

According to the mineralogical investigation of the pegmatitic bodies of WSh older granitoid, they considered important source for several rare-metal, rare earth and radio mineralization. Microscopic examination, XRD and SEM confirmed the presence of economic and steratigic minerals as euxenite-(Y), fergusonite-(Y). WSh pegmatite granitoid is considered to be a promising locality as a source of Nb, Ta, Ti, Y, REE, Zr, U and Th.

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