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Comparative mineralogy and magnetic separation characteristics of Nb-Ta oxide minerals from rare-metal pegmatite and stream sediments, Eastern Desert and Sinai, Egypt

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Abstract

Ishikawaite, samarskite-Y, ferrocolumbite and fergusonite-Y are the most predominant Nb-Ta oxide minerals in granite-pegmatite and associated stream sediments of the Eastern Desert and Sinai Peninsula, Egypt. These minerals were separated and identified from Ras Baroud pegmatite and its surrounding stream sediments, Umm Lassifa, Abu Dob and Abu Rushied pegmatite in the Eastern Desert as well as from Ras Mohamed pegmatite in the Southern Sinai. Pure samples from these minerals were prepared individually and subjected to environmental scanning electron microscope (ESEM), XRD and Frantz Isodynamic magnetic separator (Model LB 1) in order to determine their chemical composition and magnetic separation characteristics of each mineral species. On the other hand, the magnetic separation reveals that the investigated Nb-Ta oxide minerals are generally moderately paramagnetic and concentrated as magnetic fraction in the range of 0.20-0.60 amps. It is quite clear that the magnetic susceptibility of the studied mineral varieties is mainly influenced by the chemistry of these minerals besides grain shape and intensity of grain color.

The present study may be helpful for concentrating and upgrading such economic and strategic Nb-Ta minerals by physical means, a mattar which will be greatly beneficial in reducing the leaching plant costs.

Keywords: Nb-Ta oxide minerals, Frantz Isodynamic magnetic separator, Eastern Desert and Sinai.

INTRODUCTION

Several Nb-Ta occurrences have been recorded in different localities of the Eastern Desert (namely, El Naga, Abu Khurg, Abu Dabbab, Abu Dob, Noweibi, Ras Baroud, Um Lassifa, Abu Rushied) and Sinai Peninsula (Ras Mohamed) of Egypt. However, these mineralization are mainly restricted to the granite pegmatite bodies associated with the younger granite that are widely distributed in the Eastern Desert and Sinai (Omer, 1995; Ibrahim et al., 1996; Abdalla et al., 1998; Ibrahim, 1999; Attawiya et al., 2000; Ammar, 2001; Abdalla and El Afandy, 2003; Abd El Wahed et al., 2005; Raslan, 2005; Abd El Wahed et al., 2006; Saleh, 2006, Bisher, 2007; Abdel Warith et al., 2007; Abu Khoziem, 2012; Raslan et al., 2010a&b; Raslan, 2015).

Samarskite-Y, with an average assay of about 43.23% Nb_2O_5 and 17.43% Y_2O_5 has been identified in the mineralized pegmatite bodies injected in Gabal Umm Lassifa monzogranite (Raslan, 2015). It is interesting in this regard to mention that author identified Samarskite-Y, columbite and zircon from Ras Baroud granite –pegmatite in the Central Eastern Desert (Raslan et al., 2010 b) and in the stream sediments surrounding the Ras Baroud granitic pluton (Raslan, 2009). Moreover, ishikawaite (uranium-rich samarskite) with an average assay of approximately 50% Nb_2O_5 and 26% UO_2 has been identified for the first time in Egypt in the mineralized Abu Rushied gneissosegranite (Raslan, 2008). In addition, Raslan and Ali (2011) identified ferrocolumbite, ishikawaite, uranopyrochlore, and fergusonite in the rare-metal pegmatites of Abu Rushied granitic gneisses. Raslan and Fawzy (in preparation) have studied various clusters of distinguishable megascopic crystals of fergusonite and zircon minerals scattered within the pegmatitic bodies of Abu Dob granite. In addition, colored varieties of ishikawaite were recorded for first time in the mineralized pegmatite of Ras Mohamed at Southern Sinai Peninsula (Raslan et al, in prep.).

Indeed, most minerals of niobium and tantalum, and all economically important species, belong to the class of oxide minerals. Accordingly, the present paper deals with the chemical composition as well as magnetic separation characteristics of ishikawaite, samarskite-Y, ferrocolumbite and fergusonite recorded in Egypt; a matter which will be greatly assist in physical upgrading of such minerals before hydrometallurgical treatment.

SAMPLING AND TECHNIQUES

Four mineralized grab samples were collected from Umm Lassifa, Abo Dob, Abu Rushied and Ras Mohamed pegmatite bodies. Beside, one sample from the stream sediments surrounding Ras Baroud mineralized pegmatite. The latter was collected from holes having about 70 cm diameter and about 1 meter depth. The oversize 1 mm was first removed by sieving and the undersize 1 mm was properly sieved before subjecting to heavy media separation. The rock sample was properly crushed, ground, and sieved before subjecting the liberated size fractions to heavy-mineral separation. For the purpose of the present study, a considerable amount of the total bulk heavy mineral fraction was obtained by heavy liquid separation using bromoform. (Specific gravity = 2.85 gm/cm^3) and methylene iodide (specific gravity= 3.3 gm/cm3) respectively. From the obtained heavy fractions, pure monomineralic mineral grains were handpicked and investigated under a binocular microscope. The heavy mineral grains were manually picked from each of the obtained heavy fractions under binouclear microscope. Some of these selectively picked grains were analyzed by X-ray diffraction technique (XRD) using Philips X-ray generator model PW 3710/31 a diffractometer with automatic sample changer model PW1775 (21 position). The X-ray radiation used is Cu-target tube and Ni filter at 40 kV and 30 mA. This instrument is connected to a computer system using X-40 diffraction program and ASTM cards for mineral identification. Some of the separated grains were examined by Environmental Scanning Electron Microscope (ESEM). This instrument includes a Philips XL 30 energy-dispersive spectrometer (EDS) unit. The applied analytical conditions were an accelerating voltage of 30 kV with a beam diameter of 1 µm for a counting time of 60-120 s and a minimum detectable weight concentration ranging from 0.1 wt% to 1 wt%. All these analyses were carried out at the laboratories of the Egyptian Nuclear Materials Authority (NMA).

The magnetic behavior of the studied Nb-Ta oxide minerals was investigated individually using a Frantz Isodynamic Separator (Model LB 1).All the analyses were carried out at the laboratories of the Egyptian Nuclear Materials Authority (NMA).

RESULTS AND DISCUSSION

Microscopic investigation and Scanning electron microscope study

1- Ishikawaite: [Fe, U, Y (Nb, Ta) O_4]

Under the binocular microscope, the investigated ishikawaite crystals from Abu Rushied pegmatite occurs as black translucent massive grains of anhedral to subhedral and granular form. These grains are generally characterized by a dark brown streak and by a resinous to vitreous luster. The mineral grains are usually hard, compact, and metamict (Fig.2A). On the other hand, the studied ishikawaite separated from Ras Mohamed pegmatite exhibits a wide range of colors. Some of the grains are dark brown (Fig.2C) while others are dark oily brown or dark greenish brown (Fig.2E) with different gradations.

Environmental Scanning Electron Microscope (ESEM) data of the studied ishikawaite grains show that the mineral is enriched in niobium, uranium and thorium. The obtained ESEM analyses of the investigated ishikawaite of black color from Abu Rushied pegmatite (Fig. 2B), Ras Mohamed dark brown ishikawaite (Figs.2D) and dark greenish brown ishikawaite from Ras Mohamed pegmatite (Fig.2F) are presented in Table (1).

2- Samarskite-Y: Y, Fe, U, REE (Nb, Ta, Ti) O_4

Umm Lassifa samarskite-Y is generally massive with a granular form and having a characteristic velvet-yellow brown to bloody red in color (Fig.2G).On the other hand, samarskite–Y crystals of Ras Baroud stream sediments are generally massive with a rod-like form (Fig.3A). Although most crystals often possess rod-like in shape, however, some crystals are present as massive granular and tabular crystals (Fig. 3C). Environmental Scanning Electron Microscope (ESEM) data of the studied samarskite-Y crystals show that the mineral is enriched in niobium and yttrium. The obtained EDAX data for samarskite-Y from Umm Lassifa pegmatite (Fig. 2H), samarskite-Y separated from Ras Baroud stream sediments of a rod-like shape (Fig.3B) and samarskite-Y separated from Ras Baroud stream sediments of a massive granular and tabular shape (Fig. 3D)are tabulated in Table (1).

Samarskite belongs to a group of Nb-Ta mineral varieties that occur in granite pegmatite and have the general formula Am Bn O_2 (m+n), where A represents Fe, Ca, REE, Y, U and Th whereas B represents Nb, Ta and Ti. According to Hanson et al., (1999), samarskite have Nb>Ta and Ti in the B-site. Additionally, this group of minerals contains at least three species based on A-site chemistry. If REE+Y are dominant, the name samarskite-(REE+Y) should be used with the dominant of these cations as a suffix. If U+Th is dominant, the mineral is properly named ishikawaite whereas if Ca is dominant, the mineral should be named calciosamarskite. However, the exact nature of these minerals cannot be determined due to inability to quantify the valence state of iron present and dominant at the A-site in these minerals. Ishikawaite and calciosamarskite are light rare-earth element (LREE) depleted and heavy rare earth element (HREE) enriched with Y dominant. Recently, samarskite-Yb has been identified as a new species of the samarskite group (William et al, 2006). Nickel and Mandrino (1987) described samarskite-Y as a mineral with Y+REE dominant at A-site.

3- Fergusonite: [(Y, REE>Ca, U, Th) (Nb, Ta) O_4]

Under the binocular microscope, the defined fergusonite grains separated from Abu Dob pegmatite are generally massive grains of anhedral to subhedral and granular form and having a characteristic vitreous or resinous luster. Also, the investigated mineral crystals are generally translucent, compact, metamict and hard. The fergusonite crystals are mainly velvet-yellow brown to honey yellow in color (Fig.3E). Environmental Scanning Electron Microscope (ESEM) data of the studied fergusonite crystals (Fig. 3F) show that the mineral is enriched in niobium, yttrium and REE elements (Table.1).
The fergusonite group consists of REE-bearing Nb and Ta oxides, many of which are metamict and therefore commonly poorly characterized. The structure of fergusonite group is comparable to that of samarskite group but with large A-sites. Most of these minerals are monoclinic, although orthorhombic and tetragonal unit cells arise from cation ordering. Similar to other (Y, REE, U, Th)-(Nb, Ta, Ti) oxides, fergusonite (ideal formula: $YNbO_4$), occurs typically as an accessory component in granites (Poitrasson et al., 1998) and granitic pegmatites (Ercit, 2005) Due to its actinide content of several weight percent, fergusonite is commonly found in a highly radiation-damaged state (Ervanne, 2004) which is accompanied by major changes of physical properties and generally lowered chemical resistance. Correspondingly, fergusonite and other Nb-Ta-Ti oxide minerals are often affected by post-growth chemical alteration (Ewing, 1975; Ercit, 2005).

4- Ferrocolumbite [Fe Nb_2 O_6]

Abu Rushied ferrocolumbite grains are generally black in colour and possess a brilliant metallic luster and belong to two shape varieties. The first is manifested in the form of massive sub rounded and pitted particles containing surface cavities (Fig. 3G) filled with iron (Raslan, 2005). The second variety occurs in form of irregular grains. The obtained ESEM analyses of the investigated ferrocolumbite (Fig.3H) show that the mineral is enriched in niobium and iron (Table.1).

Minerals of the columbite-tantalite group have the general formula AB_2O_6, with the A site occupied by Fe, Mn and a smaller quantity of Mg, Na and trivalent ions, and the B site occupied by Nb, Ta and small amounts of Ti and W. The main trends known from the literature are the isovalent substitutions Fe ↔ Mn in the A site, and members ferrocolumbite, manganocolumbite, ferrotantalite and manganotantalite (Ercit, 1994; Ercit et al., 1995).

X-Ray Diffraction

Pure monomineralic sample from ishikawaite grains of various colors, samarskite-Y, and fergusonite were prepared by hand picking and subjected to XRD analyses. The obtained XRD data for these after annealing (heat treatment) are presented in (Figs. 4,5&6). The metamict state is characterized by structure disorder (amorphous to X-rays) while the crystal habit is frequently well developed. The essential features of this state were discussed by Pabst (1952). The metamictic state can be changed by appropriate heating of such mineral at temperatures higher than 400 C leading to their recrystallization. The data conforms to the ASTM card index No. 10-398 for Abu Rushied ishikawaite (Fig.4A) and ASTM cards index No.10-398 and 4-0617 (Fig.4B) for Ras Mohamed ishikawaite. XRD diffractograme of samarskite-Y from Ras Baroud pegmatite is illustrated in Figure (4C). The data conforms to the ASTM card index No. 2-0690 for samarskite. The obtained XRD data for samarskite –Y from Ras Baroud stream sediment after annealing (heat treatment) are presented in (Fig. 5A). The data conforms to the ASTM card index No. 2-0690 for samarskite. XRD diffractograme of fergusonite-Y of Abu Dob pegmatite (Fig.5B) conforms to the ASTM card index No. 9-0433.

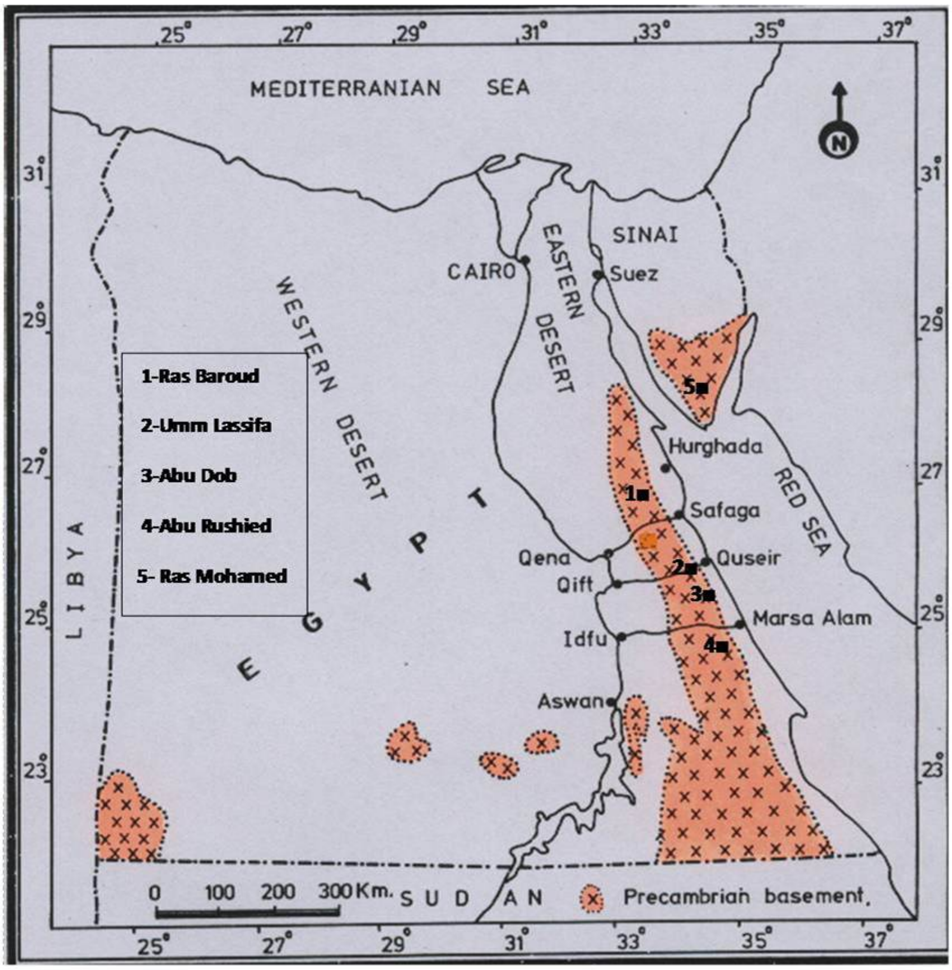

Fig.1: Location map of the studied pegmatite bodies in Egypt.

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Table (1): SEM chemical analyses of different colors of the studied Nb –Ta oxides mineral varieties.

Element oxide	Black ishikawaite	Dark brown ishikawaite	Dark greenish brown ishikawaite	Umm Lassifasam arskite-Y	Samarskite -Y rod-like	Samarskite -Y massive	Abu Dob fergusonite	Ferro-columbite
Nb_2O_5	50.31	48.60	25.90	44.81	43.39	41.61	41.49	67.35
Ta_2O_5	2.34	2.60	1.81	4.35	2.17	4.66	4.10	2.85
TiO_2	2.10	1.30	2.00	0.53	0.71	0.74	0.78	0.57
UO_2	24.19	19.30	42.72	3.01	2.26	2.27	3.30	1.54
ThO_2	4.50	4.70	13.54	2.39	2.71	1.86	4.04	0.21
FeO	11.45	4.10	3.04	1.43	-	2.74	-	16.07
Y_2O_5	-	5.00	2.31	25.07	20.82	18.65	15.60	-
CaO	1.54	4.20	3.30	1.01	2.02	1.44	0.80	-
K_2O	-	-	-	-	0.56	0.27	-	-
PbO_2	-	-	4.60	-	-	-	-	-
Ce_2O_3	-	-	-	-	-	0.82	-	-
Nd_2O_3	-	-	-	2.02	1.26	1.17	3.50	-
Er_2O_3	2.07	-	-	4.09	3.11	4.37	5.50	-
Sm_2O_3	-	-	-	1.70	1.33	1.26	2.40	-
Gd_2O_3	-	-	-	-	2.47	-	2.41	-
Dy_2O_3	-	-	-	-	4.95	-	6.30	-
Yb_2O_3	-	-	-	5.62	2.70	6.74	8.30	-
Al_2O_3	1.50	2.10	-	1.48	3.53	4.72	1.48	2.32
MnO	-	0.70	0.78	-	-	-	-	2.91
SiO_2	-	5.70	-	2.49	6.01	6.68	-	6.18
Sc_2O_3	-	1.70	-	-	-	-	-	-
Total	100	100	100	100	100	100	100	100

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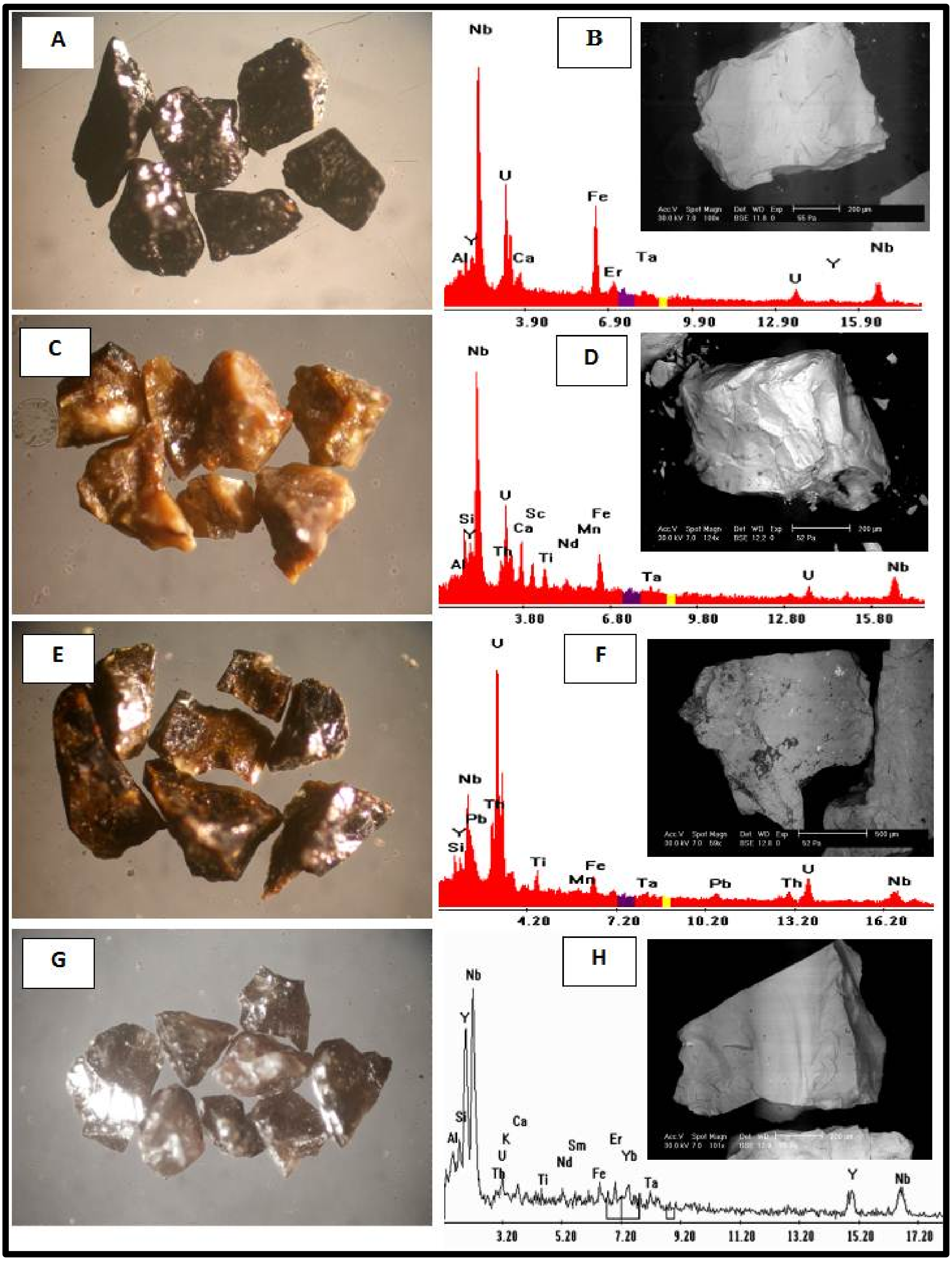

Fig.2:

A- Black massive granular ishikawaite grains, Abu Rushied pegmatite, Binocular microscope. B- Backscattered electron image and corresponding EDX spectrum. C- Yellowish brown massive granular ishikawaite, Ras Mohamed pegmatite, Binocular microscope. D- Backscattered electron image and corresponding EDX spectrum. E- Dark brown massive granular ishikawaite, Ras Mohamed pegmatite, Binocular microscope. F- Backscattered electron image and corresponding EDX spectrum. G-Reddish orange massive granular samarskite-Y, Umm Lassifa pegmatite, Binocular microscope. H-Backscattered electron image and corresponding EDX spectrum.

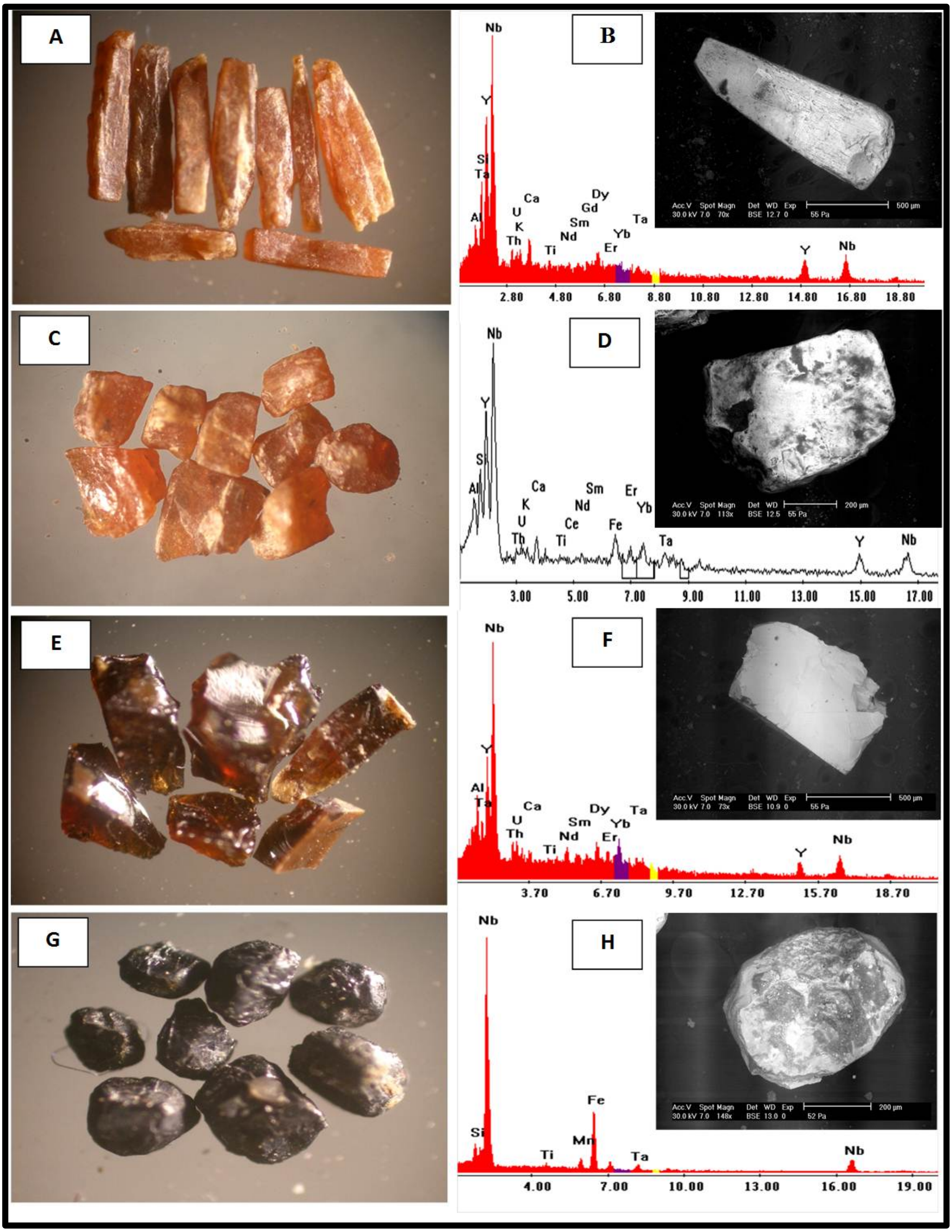

Fig.3:

A- Samarskite-Y of rod-like shape, Ras Baroud stream sediments, Binocular microscope. B- Backscattered electron image and corresponding EDX spectrum. C- Samarskite-Y of massive tabular form, Ras Baroud stream sediments, Binocular microscope. D- Backscattered electron image and corresponding EDX spectrum. E- Massive granular fergusonite crystals, Abu Dob pegmatite, Binocular microscope. F- Backscattered electron image and corresponding EDX spectrum. G- Black ferrocolumbite grains, Abu Rushied pegmatite, Binocular microscope. H- Backscattered electron image and corresponding EDX spectrum.

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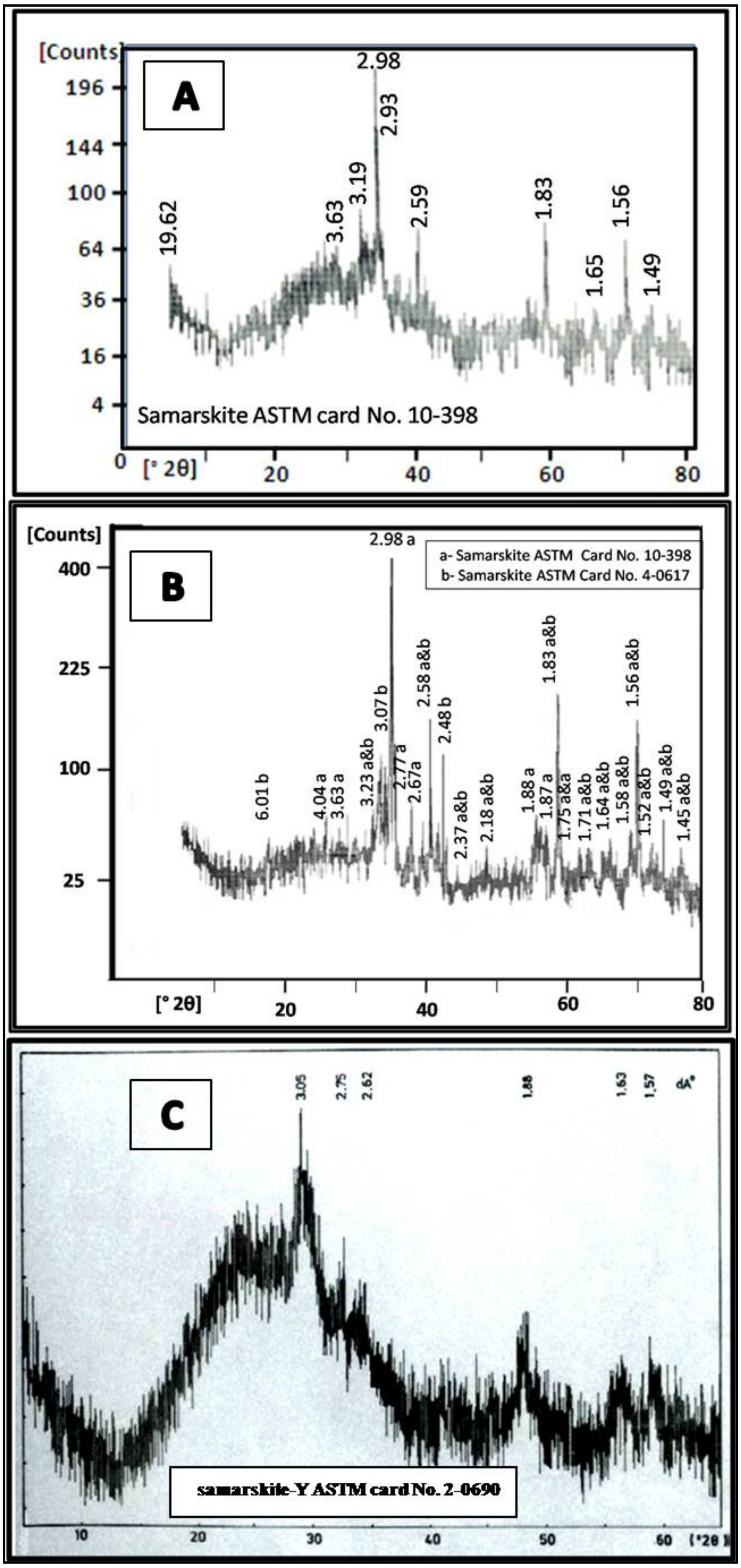


Fig.4: A- XRD diffractograme of Abu Rushied ishikawaite grains of black color.
B- XRD diffractograme of Ras Mohamed ishikawaite of various colours.
C- XRD diffractograme of samarskite-Y from Ras Baroud pegmatite**.**

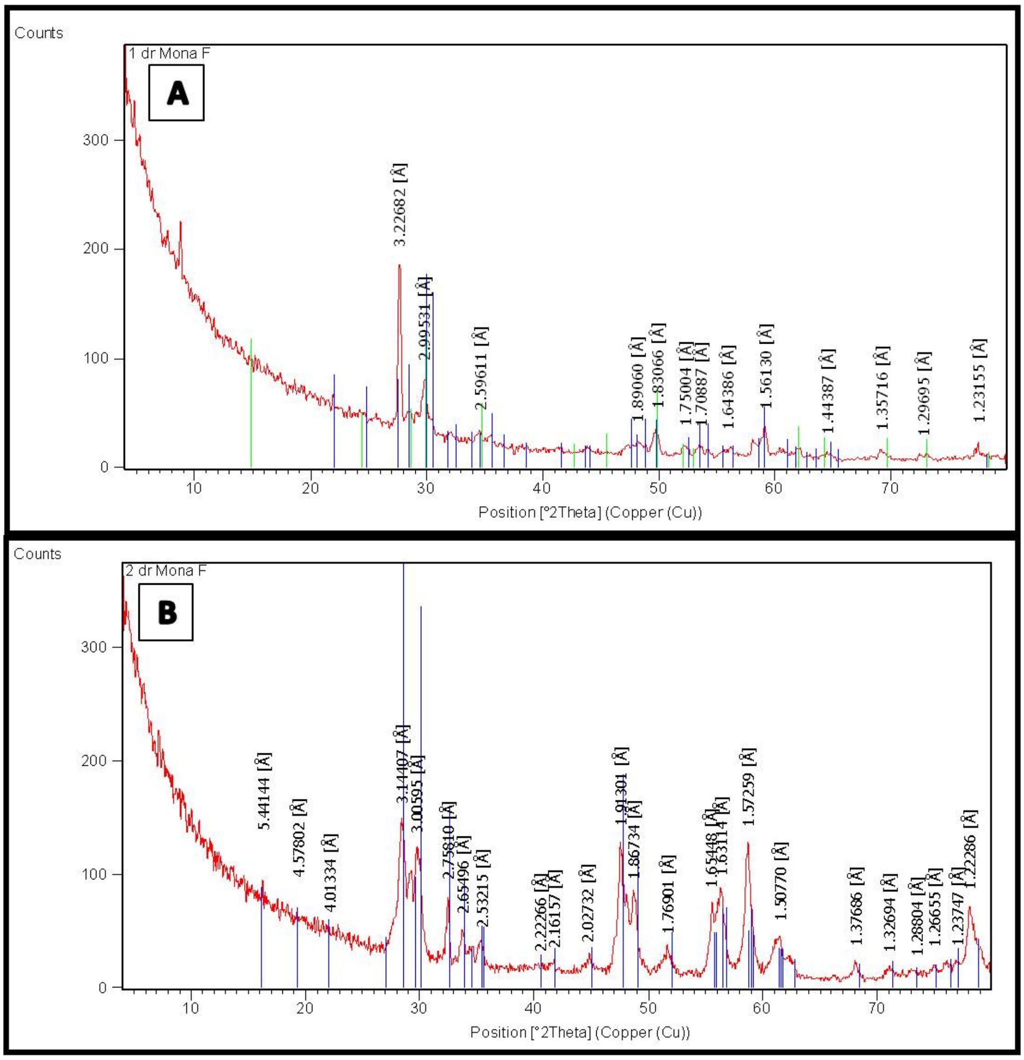


Fig.5: A- XRD diffractograme of samarskite-Y from RasBaroud stream sediment.
B- XRD diffractograme of fergusonite-Y from Abo Dob pegmatite.

Magnetic separation characteristics of niobium -tantalum oxide minerals

Preparation of a suitable feed for separation is very useful for attaining the maximum efficiency of the used equipment. The size of the separated particles is the most important factor affecting separation (Taggart 1944; Jones 1959; Pryor 1974; Gaudin 1980).Silberberg (1998) explains that magnetic properties of minerals are affected by the chemical structure of the individual molecules. In order for a substance to be paramagnetic, it must have unpaired electrons in the molecular orbitals, while diamagnetic substances contain paired electrons in the molecular orbitals. Molecular orbitals are formed when two or more atoms are bonded covalently. According to (Rosenblum and Brownfield, 1999), the minerals containing nickel, cobalt, platinum metals, and rare earth metals are also paramagnetic, and may even be ferromagnetic. This is apparently based on magnetic susceptibilities of the native metals, but in a chemical compound such as a mineral, the presumption that the same magnetic property will be found in the mineral is not often true because of the way that atomic bonding affects the magnetic susceptibility. We discovered that other metals such as manganese, copper, chromium, niobium, and tantalum, when occurring as major components of minerals, may enhance the magnetic susceptibilities of some members of a mineral series.
A considerable amount of pure monomineralic samples (about 2gm for each mineral variety) in the size range of 400-200 μm were prepared and subjected to magnetic separation using the Frantz Isodynamic separator (Model LB 1) (Fig. 6) in order to determine the behavior of each Nb-Ta mineral variety. The setting of the separator during this study was (20°) forward slope and (5°) side

slope. The mineral grains of each mineral variety were carefully prepared by hand picking under binocular microscope and generally free from inclusions or locked fragments of other minerals. After each sample run, the magnetic separator was thoroughly cleaned by a jet of compressed air to avoid contaminating following samples.

Colorless or white minerals are generally diamagnetic; that is, beyond the paramagnetic range of the magnetic separators used. Dark-colored and opaque minerals are generally extracted at less than 1.00 ampere and less commonly at greater amperages. The exceptions include red tourmaline, red rutile, some corundum, microlite, cassiterite, and many sulfides. Variations in chemistry tend to affect the depth of color and the extraction range (Rosenblum and Brownfield, 1999).

1- Ishikawaite

The results shown in figure 7 reveal that the investigated black ishikawaite variety from Abu Rushied pegmatite is strongly paramagnetic and separated as the magnetic fraction in the range between 0.1-0.4 amps of the Frantz Isodynamic separator (Fig.7 A). The extraction range which contains the greatest amount of the desired mineral concentrates (the best range) between 0.2-0.3 amperes (Fig.8). On the other hand, the colored varieties of ishikawaite separated from Ras Mohamed pegmatite show low magnetic susceptibility with respect to the black variety. The yellowish brown variety separated as the magnetic fraction in the range between 0.1-0.7 amps (Fig. 7B), and the best range is 0.4-0.7 amps (Fig.8). The dark brown variety separated as the magnetic fraction in the range between 0.1-0.6 amps (Fig. 7C), and the best range is between 0.4-0.6 amps (Fig.8). The obtained results confirm the effect of color depth in the investigated ishikawaite crystals in the extraction range.

2- Samarskite-Y

The obtained results indicated that the Umm Lassifa samarskite-Y is generally moderately paramagnetic and separated in the range between 0.1-0.5 amperes (Fig.7D). The best range of concentration between 0.3-0.4 amperes (Fig.8). Generally, the mineral show strong magnetic susceptibility with respect to colored ishikawaite varieties and low with respect to black ishikawaite. On the other hand, samarskite crystals of Ras Baroud stream sediments are generally massive with a rod-like form. Although most crystals often possess rod-like in shape, however, some crystals are present as massive granular and tabular crystals. The rod-like variety is mainly separated in the range between 0.1-0.4 amperes (Fig.7E) and the best range of concentration between 0.25-0.3 amps (Fig.8). The tabular crystals of samarskite-Y show low magnetic susceptibility than those of rod-like shape and separated in the range between 0.1-0.5 amperes (Fig. 7F) and the best range of concentration is similar to the rod-like variety (0.25-0.3 amps.), (Fig.8).

3- Fergusonite

Abu Dob fergusonite is generally massive with granular form and separated in the range between 0.1-0.4 amperes (Fig.7G). The best range of concentration is between 0.2-0.25 amperes (Fig.8). The mineral is strong paramagnetic and having strong magnetic susceptibility than both ishikawaite and samarskite-Y varieties most probably due to its iron content.

4- Ferrocolumbite

Abu Rushied ferrocolumbite grains are generally black in color and possess a brilliant metallic luster. The mineral grains behave as a strong paramagnetic and concentrated in the range between 0.1- 0.4 amperes (Fig. 7H)and the best range of concentration in the zone between 0.2-0.3 amps (Fig.8). The cumulative recovery of each mineral variety versus current intensity is shown in figure (9).

Fig.6: Frantz Isodynamic Separator (Model LB 1).

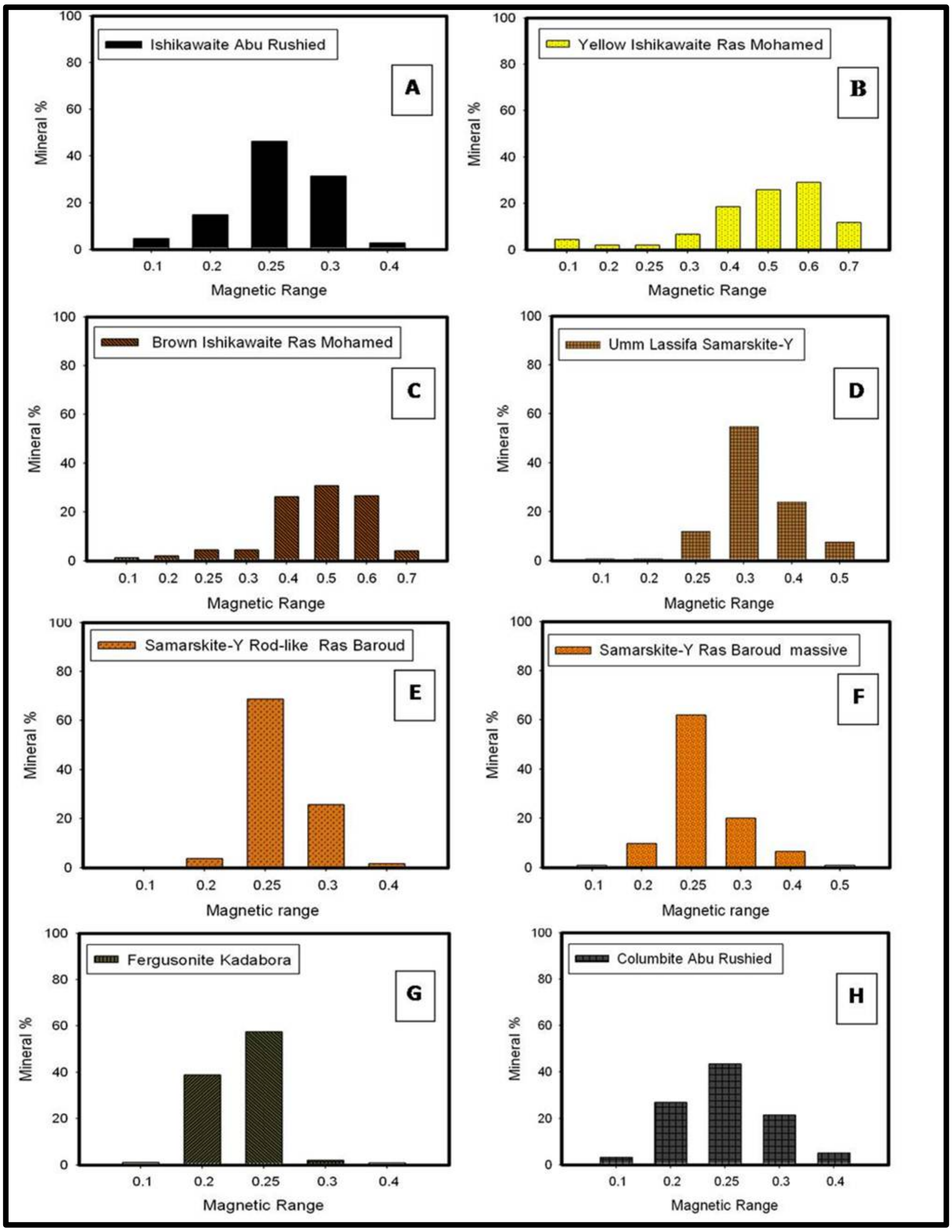
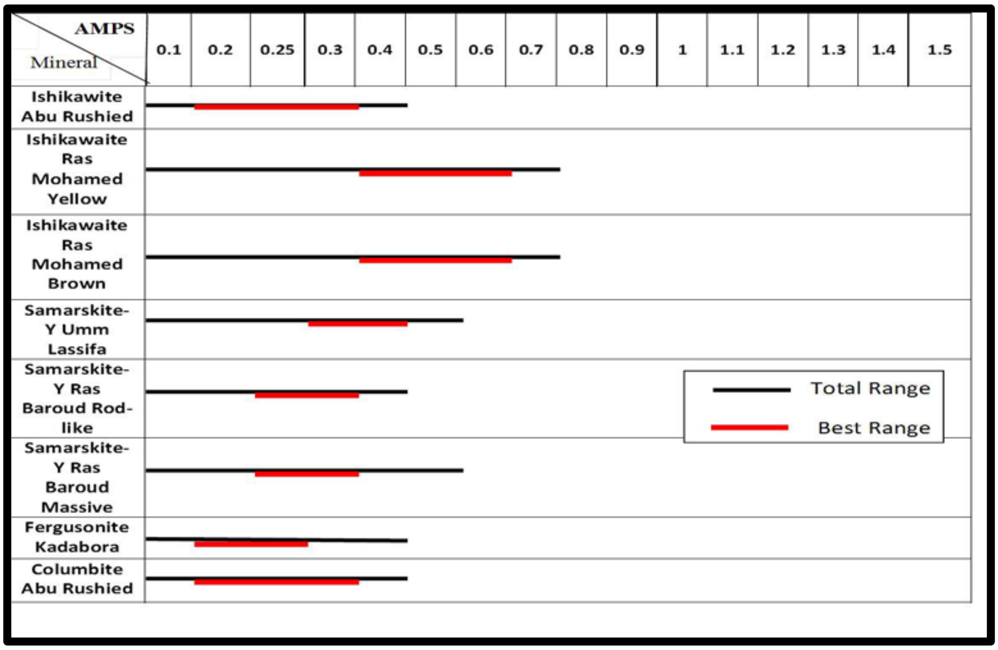


Fig.7: Histogram to show the percentage of the individual Nb-Ta oxide minerals separated as magnetic fractions at different current intensities.

Fig.8: The total range and the best range of magnetic separation for the studied Nb-Ta oxide minerals.



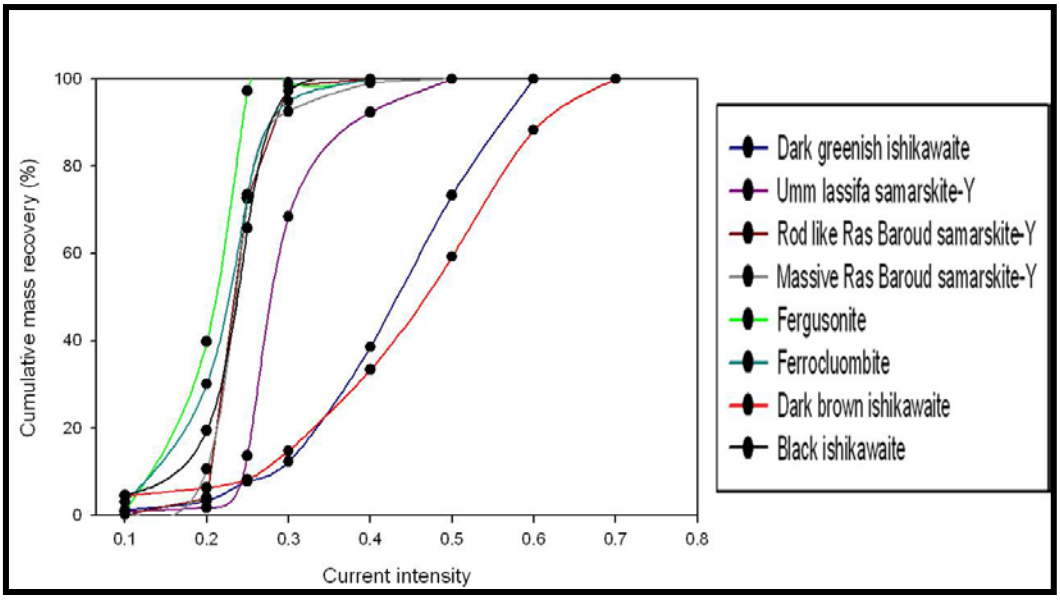


Fig.9: The cumulative recovery of each mineral variety versus current intensity

CONCLUSIONS

Magnetic separation characteristics of Nb-Ta oxide minerals species ishikawaite, samarskite-Y, fergusonite-Y and ferrocolumbite) using Frantz Isodynamic Separator (Model LB 1) proved a rather good potentialities for upgrading such economic minerals as magnetic fractions, a matter which will be of greatly beneficial in reducing leaching plant costs**.**

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