

PREPARATION OF HIGHLY PURE Y_2O_3 FROM REES CONCENTRATE OF GABAL EL-FALIQ GRANITOID ROCKS, SOUTH EASTERN DESERT, EGYPT.

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ABSTRACT

The present work aims to study the processing characteristics of the geologic ore material under consideration to prepare leach liquor suitable for the recovery of highly pure Y oxide concentrate. Ammonium sulfate roasting method was applied upon the study geologic ore material and selectively recovers 88.6% of total REEs. Several roasting parameters were investigated to optimize the selective dissolution of REEs. Since, from the foregoing results, it can be concluded that, the maximum dissolution efficiency, % of REEs from Gabal (G.) El-Faliq ore material by roasting with $(NH_4)_2SO_4$ was conducted at Solid/Reagent ratio: 1/2, Roasting time: 2 hrs. and Roasting temperature: 600°C.

From the latter, 91% of Y content from prepared $RE(NO_3)_3$ solution using 2M TBP, has been recovered at pH of 1.5 by using 2M of TBP / Kerosene for 9 min. as contact time and O/A ratio of 1/1. More than 98% of the loaded Y was regenerated using 3M H_2SO_4 at A/O ratio of 2/1 and contact time of 25 min. From the latter, highly pure Y_2O_3 was prepared after oxalate precipitation.

1. INTRODUCTION

Gabal (G.) El-Faliq area (250 Km²) is located between latitude 24° 35' and 24° 42' N and longitude 34° 28' and 34° 35'E. It is mainly composed of ophiolitic melange, gneisses, older and younger granitoids and post granitic dykes and veins (figure 1).

According to (Saleh, M. G.; Mostafa, A. D.; Darwish, E. M. and Salem, A. I., 2014)^[1], the mineralization of G. El-Faliq can be classified on the basis of mode of occurrence and lithological associations into: a) secondary uranium minerals (uranophane), b) niobium-tantalum minerals, c) sulphide minerals and d) accessory minerals.

The corresponding valuable metals of this mineralization include U, Th, REEs, Nb, Ta and Zr. The Rare earths, Th, and U mineralization of G. El-Faliq were found to be included in altered granites.

The recovery of REE is generally accomplished by a variety of methods including the mineral acids or alkali fusion at high temperature (El-sheikh, 2017)^[2]. For selective dissolution of REEs ammonium sulfate roasting process has been applied upon a variety of ore materials. In this process, the ore was roasted with $(NH_4)_2SO_4$ at high temperature (600°C) for some time. After roasting, valuable REEs were converted into corresponding sulfates that are highly soluble in water. (Liu et al, 2012^[3]; El-Hazik 2005^[4]).

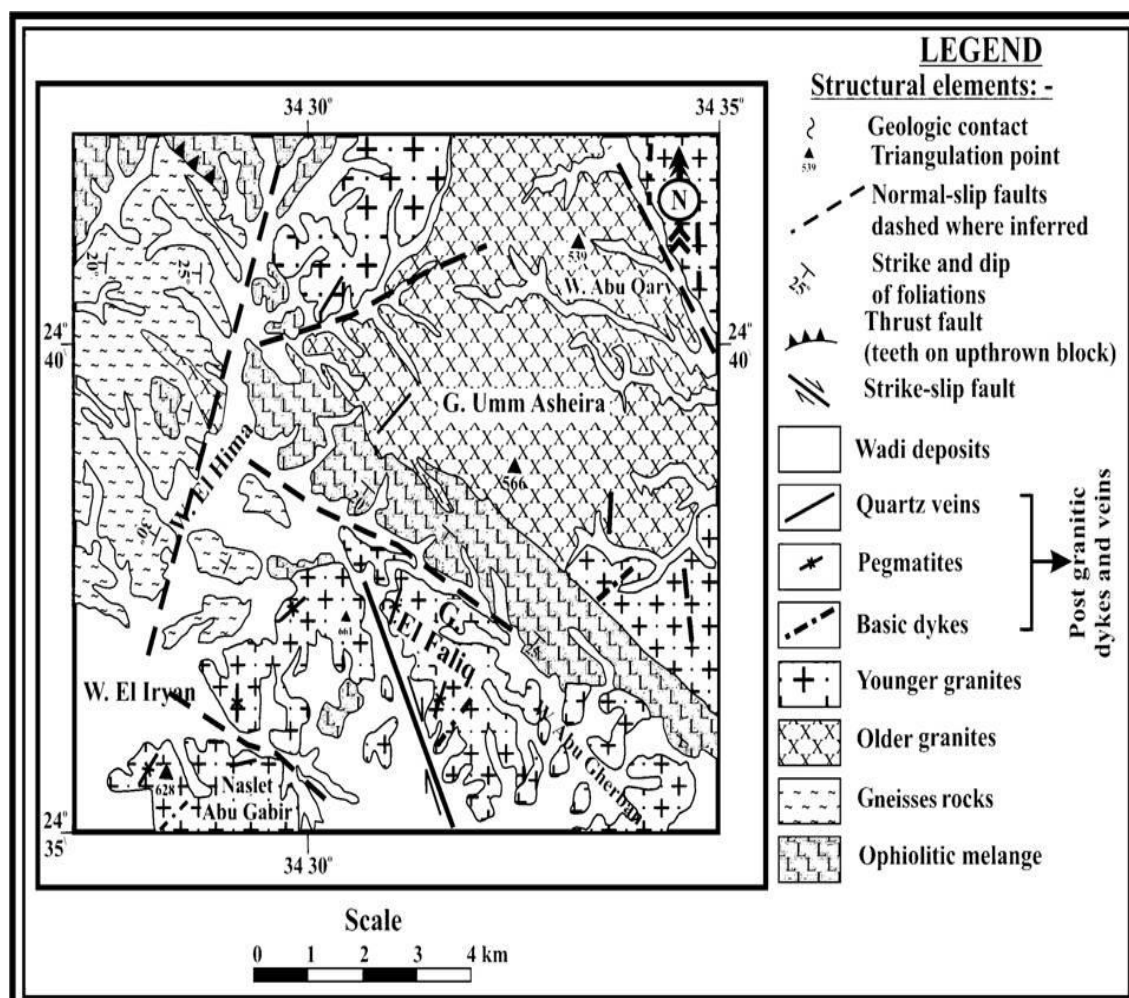


Fig. 1: Geologic map of G. El-Faliq area, South Eastern Desert, Egypt, after Saleh, et al. (2014)^[1].

Various processes have indeed been developed for the individual separation of the REEs from naturally occurring REEs mixtures include fractional crystallization, fractional precipitation as well as solvent extraction and ion exchange (Gupta & Krishnamurthy, 1992)^[5]. Yttrium (Y) is one of the important elements of great demand in astronavigation, luminescence, nuclear energy and metallurgical industries. For a variety of uses in these specialized areas, high purity Y is often required. Accordingly, in Egypt, a procedure involving HNO₃ acid leach/undiluted TBP system is successfully developed to prepare a pure Y₂O₃ more than 95% from the ore residue originally collected from Gabal Abu Dob multiple-oxide ore material (Amer, 2004)^[6].

The present work is concerned with the processing of REEs mineralization from G. El-Faliq area altered granites, which would be considered as the source of heavy REEs elements. Therefore, a representative technological sample assaying 2.1% REEs has been properly collected. The purpose was to prepare highly pure Y₂O₃ after the dissolution of total REEs by roasting with ammonium sulfate followed by proper treatment by tri-butyl phosphate.

2. EXPERIMENTAL

2.1. Characterization of the Study Sample

In order to determine the suitable leaching method and / or technique for the working ore material under consideration, it was decided to study its chemical and mineralogical composition.

2.1.1. Chemical composition

A representative sample portion of the collected technological sample was properly prepared for complete chemical analysis beside the tenor of the economic metal values. The major element oxides were analyzed using Shapiro and Brannock (1962)^[7] rapid silicate analytical procedure. While trace elements analyses were carried out by using XRF technique REEs were analyzed by using UV-Visible Recorder Spectrophotometer model Shimadzu UV-160A at λ 650 nm using 1% ascorbic acid, formate buffer, 0.05% arsenazo-III at pH 2.6, (Marczenko, 2000)^[8].

2.1.2. Mineralogical composition

To investigate the mineralogical composition of the study sample, heavy liquid separation procedure was applied using bromoform (sp.gr. 2.84). The obtained heavy mineral fractions were investigated under the binuclear microscope and some picked mineral grains were analyzed by X-ray diffraction technique (XRD). A PHILIPS X-ray generator model PW 1140/90 fitted with a diffractometer model PW1050/80 was used. The X-ray tube used was a Cu- target model PW 2233/30 fitted with a Ni-filter and was operated at 40 Kv and 20 mA.

In the meantime, the environmental scanning electron microscope (ESEM-EXL30 Philips type) coupled with an energy dispersive X-ray analyzer (EDX unit system) was also used to define the elemental composition of the investigated grains. This technique has also been quite beneficial in identifying the individual composition of the REEs content of the working ore material.

2.2. Recovery Procedures

2.2.1. Ammonium sulfate roasting process of the working ore material

Ammonium Sulfate roasting experiments were applied under different conditions to study the dissolution efficiency of REEs from the study ore material. In each experiment, a ground ore sample portion of 5 g was mixed with ammonium sulfate of different solid/ reagent (S/R) ratio, different time and different temperature. At the end of each leaching experiment, the slurry was filtered and washed; both the filtrate and washings were made up to volume. Proper aliquots of the latter were then analyzed for the leached REEs metal values to calculate their leaching efficiencies.

For the preparation of pure products, proper leach liquor was prepared after applying the obtained optimum roasting conditions upon 1 kg of technological sample, which was found to assay 2.79 g/L REEs. From the latter, REEs were recovered through the oxalate precipitation.

2.2.2. Selective separation of Y

After precipitation of REEs cake as oxalate at pH 1.3 using 20 % of oxalic acid solution, the obtained precipitate was filtered, washed and re-dissolved in Conc. nitric acid solution. The latter was subjected to selective yttrium extraction using TBP in kerosene at the obtained optimum extraction conditions and recovered as oxalate.

3. RESULTS AND DISCUSSION

3.1. Characteristics of the Working Ore Material

3.1.1. Chemical composition

Chemical composition of the study G. El-Faliq ore sample is given in table 1. From the latter, it is evident that the G. El-Faliq ore material is mainly composed of SiO₂, Al₂O₃. This may reflect as its high content of aluminum-silicate gangue minerals and lower content of Fe₂O₃, TiO₂. In addition, the MgO & CaO not exceeded than 3.5%, which would favor using acid leaching. On the other hand, the economic trace and rare earth elements represent economic interesting values, beside some trace elements, e.g. Zr, Nb, and U. The average REEs content represents about 2.1%. From the latter, the present work was oriented to recover of REEs and hence Y.

Table (1): Chemical composition of the study G. El-Faliq ore material.

Major contents	Conc., (%)	Trace elements	Conc. (%)
SiO ₂	69.22	U	0.0120
TiO ₂	0.011	Cd	0.0450
Al ₂ O ₃	13.87	Nb	0.757
Fe ₂ O ₃	1.33	V	0.002
CaO	2.55	Th	0.01
MgO	0.86	*REEs	2.122
Na ₂ O	4.91	Zr	4
K ₂ O	2.89	Zn	0.014
P ₂ O ₅	1.031	Cu	0.0003
MnO	0.010		
**L.O.I.	1.13		
Total	97.81		

* REE: measured as Y (Marchizinc, 2000)^[8]

** L.O.I. loss of ignition.

The estimated error for major constituents is not more than ± 5%

3.1.2. Mineralogical composition of the working ore material

From the detailed mineralogical study of the working, G. El-Faliq mineralized sample the following mineral assemblage recognized:

- 1- Yttrocolumbite mineral which is yttrium-rich columbite with the formula (Y,U,Fe)(Nb,Ta)O₄. (figure 2).
- 2- Xenotime and Zircon minerals that were identified by using XRD technique as shown in table 2.

This mineral assemblage reflects the refractory nature of the study ore material, a matter that requires applying severe conditions for its processing. In the present work, salt roasting technique was applied by using $(\text{NH}_4)_2\text{SO}_4$.

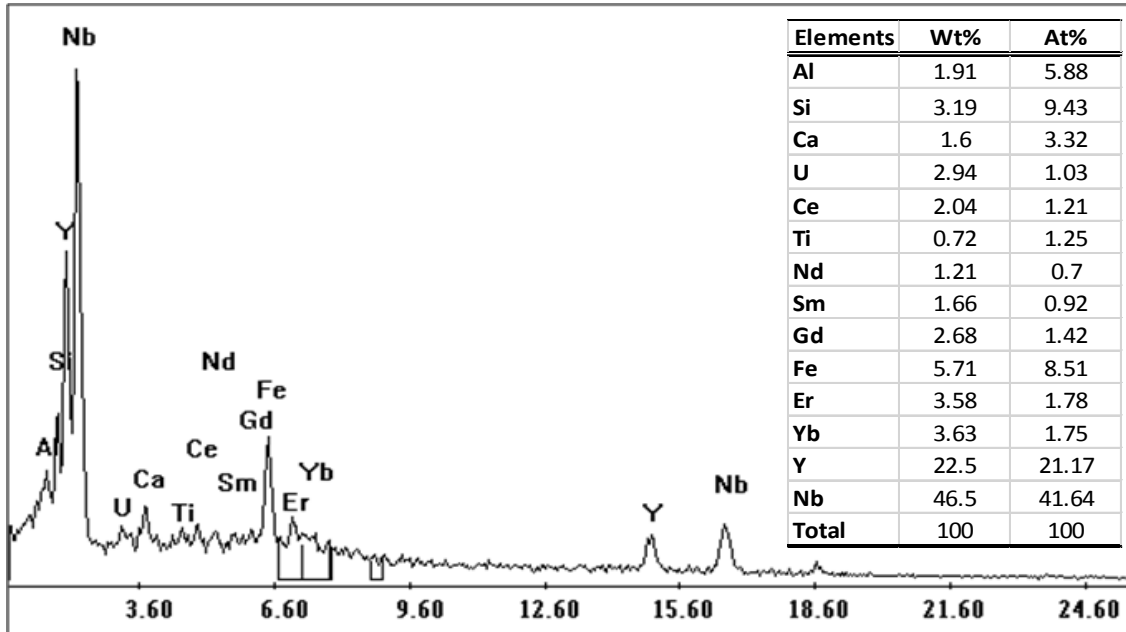


Fig. 2: EDAX analysis for the identified yttrocolumbite.

Table (2): X-Ray diffraction data of the identified Zircon & Xenotime.

Sample	ASTM #6-0266 Zircon		ASTM #11-0254 Xenotime		
	ZrSiO ₄		YPO ₄		
	dA ^o	I/I ^o	dA ^o	I/I ^o	
	4.46	47	4.43	45	
	3.45	55			3.45 100
	3.31	100	3.3	100	
	2.75	3			2.75 10
	2.67	6	2.65	8	
	2.57	18			2.56 50
	2.53	48	2.518	45	
	2.34	8	2.336	10	
	2.55	6	2.217	8	
	2.15	4			2.15 25
	2.08	17	2.066	20	
	1.92	12	1.908	14	1.929 10
	1.77	13	1.751	12	1.738 50
	1.72	39	1.712	40	
	1.66	14	1.651	14	
	1.55	2	1.547	4	1.543 10
	1.48	8	1.477	8	
	1.38	10	1.381	10	1.383 8
	1.37	7	1.362	8	1.346 6

3.2. Results of $(\text{NH}_4)_2\text{SO}_4$ Roasting Process

According to the mineralogical constitution of the working ore material, it was found necessary to roasting technique to selectively leach of its REEs content in a trial to reach to selective extraction of Y. Several roasting parameters were investigated to optimize the selective dissolution of REEs.

3.2.1. Effect of the Solid/Reagent (S/R) ratio

This factor was studied at different S/R ratios ranging from 1/1 to 1/4 at a temperature of 600°C and a roasting time of 1.5 h. The cooled roasted matrix was washed with distilled water and closed to up to 100 mL volume. REEs in the aqueous solutions were analyzed and its dissolution efficiency, (%) was calculated. The obtained data, table 3, indicates that, increasing of the S/R ratio from 1/2.5 to 1/2 has a marked effect on REEs dissolution efficiency. While decreasing S/R ratio from 1/2 to 1/4 decreases REEs dissolution efficiency, to 66.58%. This may be due to the precipitation of light REEs to their double sulfates with increasing input. **Abreu and Morais (2010)**^[9].

Table (3): Effect of S/R ratio upon REEs dissolution efficiency, %.

Solid/Reagent, ratio	Dissolution efficiency, %
1:1	61.79
1:1.5	68.97
1:2	75.16
1:2.5	70.09
1:3	70.31
1:4	66.58

3.2.2. Effect of roasting time

The effect of roasting time upon REEs dissolution efficiency from the study ore material is represented in table 4. It was conducted by roasting the ore sample at different time ranging from one hour to three hours. In this experiment, the other roasting conditions were fixed at solid/reagent ratio of 1/2, at 600°C as roasting temperature. The obtained data revealed that, a dissolution efficiency of 88.6% was achieved. After 2 hrs. REEs dissolution efficiency decreased due to its precipitation as double sulfate.

Table (4): Effect of roasting time upon REEs dissolution efficiency, %.

Time, h.	REEs dissolution efficiency, %
1	54.3
1.5	75.16
2	88.6
2.5	84.8
3	63.1

3.2.3. Effect of roasting temperature

The effect of the roasting temperature upon REEs dissolution efficiency was investigated within the range from 550 up to 750°C . The other roasting conditions were

fixed at solid / reagent ratio of 1/2, and 2h as roasting time. Table 5 revealed that increasing roasting temperature more than 600°C has resulted in an opposite effect up on REEs dissolution efficiency, which decreased, from 88.6 % to 72.26 % due to hydrolysis.

Table (5): Effect of temperature upon REEs dissolution efficiency, %.

Temperature	REEs dissolution efficiency, %
550°C	51.6
600°C	88.6
650°C	86.99
700°C	72.11
750°C	72.26

Finally, from the forgoing results, it can be concluded that, the maximum dissolution efficiency, % of REEs from G. El-Faliq ore material by roasting with (NH₄)₂SO₄ was conducted at Solid/Reagent ratio: 1/2, Roasting time: 2 hrs. and Roasting temperature: 600°C.

3.3. Results of Y Recovery

3.3.1 Preparation of leach liquor

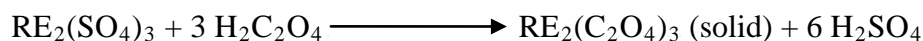
Applying the obtained optimum roasting conditions upon 150 g of the working sample and after cooling and water leaching of the obtained matrix yielding 1 L of sulfate solution of pH 1.8. The latter was chemically analyzed for REEs and the other valuable elements as given in table 6.

Table (6): Chemical composition of the prepared pregnant sulfate leach liquor.

Element's ions	g/L
R ³ E ⁺	2.790
U ⁺⁶	0.016
Th ⁺³	0.04
SO ₄ ⁻²	1.6

3.3.2. Precipitation of total REE

The total REE (2.79 g/L) in the prepared leach solution were selectively precipitated by the gradual addition of 20 % oxalic acid with continues stirring. Almost complete precipitation of REE as strong stable insoluble RE oxalate cake was attained at pH 1.3 with precipitation efficiency of 98.5%. The precipitation process is illustrated in the following equation.



After filtration, washing and dryness the obtained RE oxalate cake was then identified by means of EDAX and chemical analyzes to determine the other associated impurities and its RE concentration. From the latter, it was found that, the purity of RE oxalate concentrate achieved 93.09 % with impurities (1.63 % of Ca, 2.68 % of Si and 2.60 % of Al) as in figure 3. For more purification, the obtained RE product was dissolved in 10

% HCL and re-precipitated again using NH_4OH at pH 8.7 to obtain REEs hydroxide of purity achieved 98.25%. **Abdellah, (2009)**^[10], The latter was dried and then dissolved in Conc. HNO_3 to prepare $\text{RE}(\text{NO}_3)$ solution which subjected to solvent extraction process using TBP for individual separation of Y. So, the nitrate solution required for Y extraction was already prepared by complete dissolution of REE hydroxide in 31.5 % HNO_3 acid at S/L ratio of 1/3 and stirring for 2h at 70 °C, **Khalafalla, (2013)**^[11].

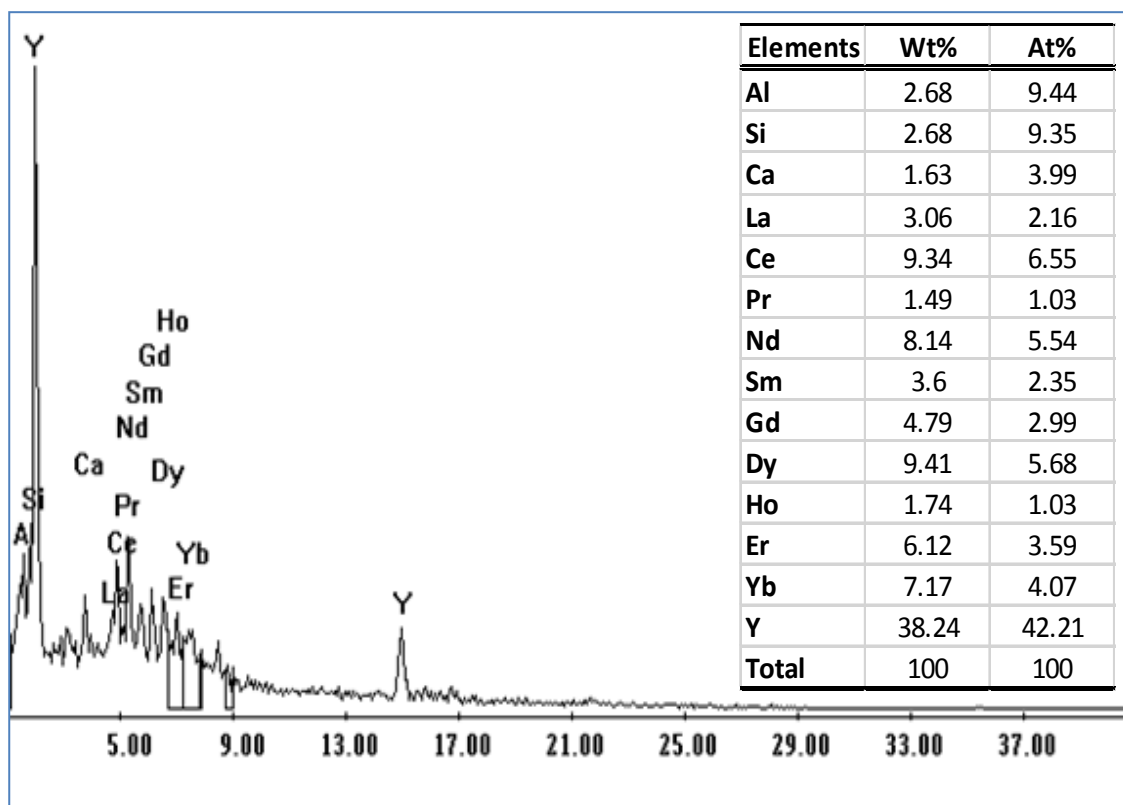


Fig. 3: The EDAX analyses of the obtained RE cake before purification.

3.3.3. Individual separation of yttrium using TBP

The prepared nitrate solution assaying 2.17 g/L of Y was used for selective yttrium extraction process by using TBP in kerosene in the presence of 0.5M of EDTA as a complexing agent for masking different impurities.

The relevant effective extraction parameters such as TBP concentration, pH value, contact time and phases volume ratios (O/A) were investigated to optimize yttrium extraction.

3.3.3.1. Effect of TBP concentration

The effect of varying TBP concentration in range from 0.5 – 2.5 M in kerosene upon yttrium extraction efficiency was studied. The pH of aqueous solution was 0.5 at O/A ratio of 1/1 and a contact time of 9 min. After separation, yttrium was analyzed in the aqueous solution. The obtained data, table 7, revealed that the maximum yttrium extraction efficiency of 59.3 % was attained at TBP concentration of 2.0 M.

Table (7): Effect of TBP concentration upon yttrium extraction efficiency.

TBP, M Conc.	Y Extraction Efficiency (%)
0.5	29.9
1.0	40.9
1.5	45.7
2.0	59.3
2.5	57.1

3.3.3.2. Effect of pH

The effect of pH values of the prepared nitrate solution upon yttrium extraction efficiency was studied in the pH range from 0.5 to 2.5. The other parameters were kept constant at TBP concentration of 2.0 M in kerosene, O/A ratio of 1/1 and contact time of 9 min. The obtained data, table 8, indicated that, Y extraction efficiency increased from 59.3 to 78 % with increasing the pH value of the nitrate solution from 0.5 to 1.5. Further increasing of pH to 2.0 and 2.5 have a reverse effect, where yttrium extraction efficiency decreased to 71.5 % at pH 2.5

Table (8): Effect of pH value upon yttrium extraction efficiency.

pH	Y Extraction Efficiency (%)
0.5	59.3
1.0	70.3
1.5	78.0
2.0	70.5
2.5	71.5

3.3.3.3. Effect of contact time

Table 9 shows the effect of contact time (shaking time) upon yttrium extraction efficiency from the nitrate solution of pH 1.5 at O/A ratio of 1/1 and shaking at different time ranging from 3 to 15 min. The data indicates that, the extraction efficiency of Y achieved 90.0 % after 9 min. Further increase in contact time to 15 min showed negligible effect.

Table (9): Effect of contact time upon yttrium extraction efficiency.

Contact Time, min.	Y Extraction Efficiency, (%)
3	72.6
6	83.5
9	90.0
12	90.1
15	90.3

3.3.3.4. Effect of O/A ratio

This effect was studied by mixing the present nitrate solution adjusted at pH 1.0 with 1.5 M of TBP in kerosene at different O/A ratios between 1/1, 1/2, 1/3 & 1/4 for a contact time of 9 min. Yttrium in the aqueous solution was analyzed after separation and its loading efficiency was calculated as given in table 10. From this table it is clear that yttrium extraction efficiency at O/A ratio of 1/1 is the optimum where it reached 91%.

Table (10): Effect of O/A ratio upon Y extraction efficiency.

O/A ratio	Y extraction efficiency, %
1/1	91
1/2	86.5
1/3	72.2
1/4	61.2

From the forgoing study, it was found that 91 % of Y was extracted from the prepared RE (NO₃)₃ solution using 2M TBP at pH 1.5 and O / A ratio of 1 / 1. The loaded solvent was regenerated by stripping process when the following stripping parameters were investigated. It is worthy to mention herein that before striping tests, the loaded solvent was subjected to water scrubbing (1:1) to remove traces of Nb and the co- extracted REE.

3.3.3.5. Effect of stripping agents type

The yttrium loaded TBP was stripped with different stripping agents, e.g. hot H₂O and 1.0M of HCl, HNO₃ and H₂SO₄, at A/O ratio of 1/1 and contact time of 5 min. After separation, Y in the strip solution was analyzed and its stripping efficiency was calculated. The obtained data revealed that, the maximum Y stripping efficiency of 39% was attained by using 1M H₂SO₄, table 11.

Table (11): Effect of stripping agent's type upon yttrium stripping efficiency.

Stripping Reagent	Y Stripping Efficiency, %
Hot H ₂ O	7
1 M HCL	10
1M HNO ₃	18
1M H ₂ SO ₄	39

3.3.3.6. Effect of H₂SO₄ concentrations

The loaded TBP was contacted with H₂SO₄ strip solution of different concentrations ranged from 1 to 4M. The other stripping parameters were kept constant at A/O ratio of 1/1 and contact time of 5 min. The obtained data, table 12, emphasized that, Y stripping efficiency shows improvement from 39.0 to 58.7% as the strip solution concentration increased from 1 to 3M. Further increasing in H₂SO₄ concentration to 4 M has a limited influence.

Table (12): Effect of H₂SO₄ concentration upon Y stripping efficiency.

H ₂ SO ₄ Conc. (M)	Stripping Efficiency, %
1.0	39.0
2.0	45.5
3.0	58.7
4.0	60.2

3.3.3.7. Effect of contact time

The effect of the stripping time upon Y stripping efficiency from 5 to 30 min. using 3 M H_2SO_4 solution at A/O ratio of 1/1. The obtained results are given in table 13. It was noticed that yttrium stripping efficiency is highly influenced by increasing the stripping time where it increased from 58.7 to 92.0% by increasing the contact time from 5 to 25 min.

Table (13): Effect of contact time of stripping upon yttrium efficiency.

Contact Time, (min.)	Y Stripping Efficiency, %
5	58.7
10	69.4
15	77.1
20	85.6
25	92.0
30	91.5

3.3.3.8. Effect of A/O Ratio

This factor not only represents the equilibrium state between both aqueous and organic phases but also shows the suitable applied A/O ratio. It was studied at A/O ratios ranged between 4/1, 3/1, 2/1 & 1/1 by using 3 M H_2SO_4 and contact time 25min. The obtained data, table 14, revealed that the Y stripping efficiency of 99.4 was attained at A/O ratio of 3/1. Indeed, the suitable applied A/O ratio of 2/1 because it yield more concentrated Y strip solution suitable for the subsequent precipitation process.

Table (14): Effect of A/O ratio upon Y stripping efficiency.

A/O ratio	Y Stripping Efficiency, %
4/1	99.7
3/1	99.4
2/1	98.0
1/1	92.0

Finally, it can be stated that the optimum conditions for regenerating 98% of Y content from the loaded TBP are 3 M H_2SO_4 stripping solution and shaking for 25 min at A/O ratio of 2/1. The yielded strip solution contains 1.91 g / L of Y was then adjusted to precipitation process to prepare the required Y concentrate.

3.4. Precipitation of Yttrium

Yttrium in rich sulfate strip solution was precipitated by gradual addition of 10% oxalic acid solution with continues stirring. Almost complete precipitation of Y as strong stable insoluble yttrium oxalate cake was attained at pH 1.1 and stirring for 60 min with 98.25% precipitation efficiency. The obtained Y oxalate precipitate was filtered, washed and then ignited at 950°C for 2h. The produced Y_2O_3 was investigated by means of EDX analysis to confirm its high purity as shown in figure 4.

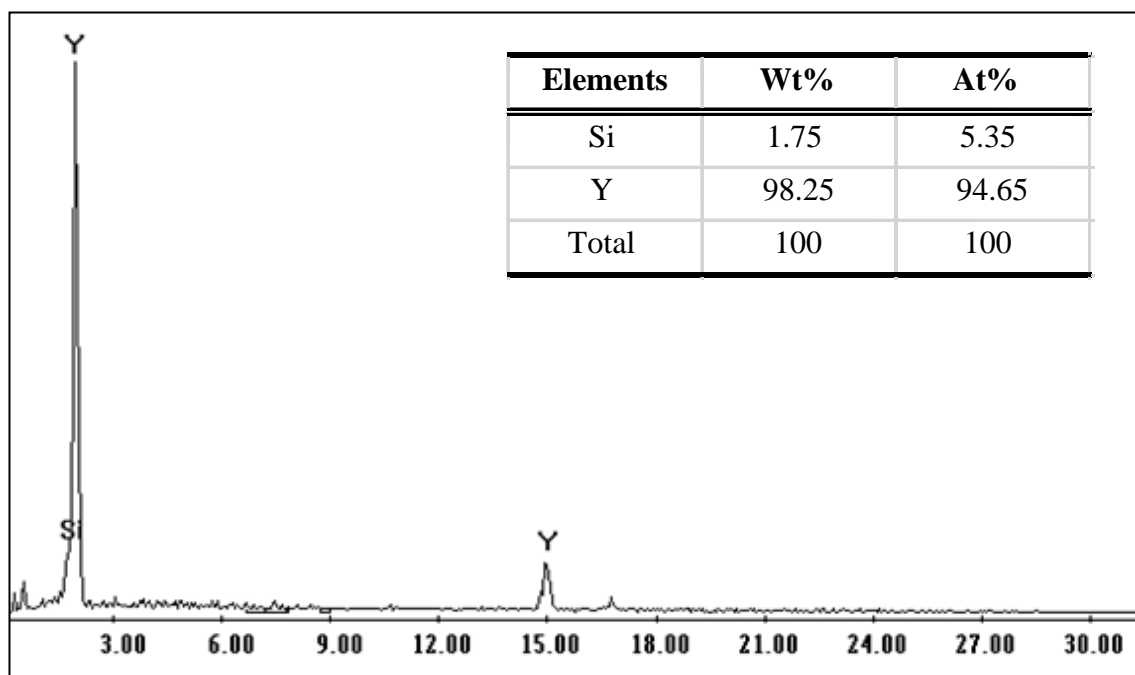


Fig. 4: Separated Y oxides from RE cake.

4. CONCLUSION

G. El-Faliq representative sample, South Eastern Desert, Egypt hosts several economic minerals such as yttracolumbite, xenotime, and zircon. The corresponding interesting valuable metals of these minerals include Y, REEs, Nb, Ta and Zr, which assayed 2.1% of REEs (1.78 %Y) and 0.76 % of Nb, beside 4% of Zr. Ammonium sulfate roasting method has been applied for selective dissolution of REEs with achieved efficiency of 88.6% under the following conditions; 1:2 S/R ratio at 600 °C for 2h.

Sulfate leach liquor was prepared assaying: 2.79 g / L of REEs, 0.016 g / L of U, and 0.040 g / L of Th. Solvent extraction techniques was applied by using TBP in kerosene for the selective recovery the Y from REEs oxalate cake in highly pure forms.

5. REFERENCES

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تحضير أكسيد إيتريوم عالي النقاوة من ركازات العناصر الأرضية النادرة من صخور جبل الفالق بجنوب الصحراء الشرقية ج.م.ع.

إعداد: دعاء إبراهيم زكي
هيئة المواد النووية

يهدف هذا العمل إلى دراسة خصائص معالجة المواد الخام الجيولوجية قيد الدراسة لإعداد المحاليل المائية المناسبة لاستعادة وإسترجاع ركازات أكسيد الإيتريوم عالي النقاوة. ولقد تم تطبيق طريقة تحميص كبريتات الأمونيوم على دراسة المواد الخام الجيولوجية لاستعادة مجموع العناصر الأرضية النادرة بنسبة 88.6% بشكل انتقائي.

ولقد تم دراسة العوامل العديدة المؤثرة على التفكك الانتقائي للعناصر الأرضية النادرة ومن النتائج المتقدمة التي حصلنا عليها ومنها تم الوصول إلى النسبة المئوية للكفاءة القصوى للتفكك للعناصر الأرضية النادرة من العينة الجيولوجية خاصة جبل الفالق بجنوب الصحراء الشرقية بتحميصها مع كبريتات الأمونيوم والتي كانت على النحو التالي: نسبة الصلب إلى المذيب 2/1، وزمن التحميص ساعتان، ودرجة حرارة التحميص 600 درجة مئوية. ومن هذا الركاز لمجموع العناصر الأرضية، في الآخر تم استعادة 91% إيتريوم من محلول نيترات العناصر الأرضية النادرة عند أس هيدروجيني 1.5 بإستخدام 1.5 مولر من TBP/kerosene لمدة تسعة دقائق كزمن التصاق ونسبة O/A تكون 1:1 وفي الخطوات الأخيرة يتم إسترجاع حوالي 98% من الإيتريوم المحمل بإستخدام حمض كبريتيك 3 مولر عند نسبة وسط مائي/ وسط عضوي 1/2 وزمن التصاق 25 دقيقة ومن ذلك أمكننا تحضير أكسيد إيتريوم عالي النقاوة بعد ترسيبه في شكل أوكسالات.