Research Paper

Extraction of Lanthanides Concentrate from Abu Rusheid mineralized rocks, south Eastern Desert, Egypt

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Abstract: *Abu Rusheid area lies in the southern part of the Eastern Desert of Egypt at the boundary between the Central Eastern Desert and the Southern Eastern Desert. It is limited by latitudes 24° $35`$ $00``$ and $24^o$ $41`$ $22``N$ and longitude 34° $44`$ $13``$ and $34^o$ $49`$ $30``E$ at about 90 km southwest of MarsaAlam City on the Red Sea coast, is one of the most promising occurrences, the present work dealt with the extraction of lanthanides from its bearing ore. This was carried out through both lanthanides leaching and extraction processes where their optimum leaching conditions were studied concluding that 89% leaching efficiency was obtained under the following leaching conditions: 150 g/L hydrochloric acid concentration, at temperature 100 $^\square C$, -0.125+0.075 mm grain size, 4 hours contact time and ¼ solid/liquid ratio. The positive values of entropy change, ΔS, for the lanthanide ions, suggested an increase in the randomness during lanthanides leaching process and the increase in negative charge of ΔG° value with increasing the temperature implies that the reaction is favorable at higher temperature. Lanthanides extraction process was performed upon the prepared Abu Rushieid leach liquor where iron was precipitated at pH 4 by sodium hydroxide solution. Lanthanides cake was also precipitated from the filtrate at pH 11 which characterized by the presence of Y, Dy, Gd, Nd, Pr and Ho elements. Such precious elements are very important in the nuclear industry.*
Key words: *Abu Rusheid, lanthanides concentrate, leaching, extraction.*

I. INTRODUCTION

Lanthanides constitute about one seventh of the total elements of the Periodic Table. If the rare-earth-like metals (Sc, Y, and the actinide metals Ac which also resemble the REE) are added, the mentioned group would account for 30% of the known elements. They can be found in two mineral groups: minerals containing ytterium include gadolinite, xenotime, samarskite, euxenite, fergusonite, yttrotantalite, yttrofluorite (variety of fluorite), thalenite, yttrialite, small amount occurs in zircon. Besides, minerals containing cerium and light lanthanides include bastnaesite, monazite, allanite, loparite, ancylite, parasite, lanthanite, chevkinite, cerite, stillwellite, britholite, fluocerite. About 95% of the rare earths occur in only three minerals; namely bastnasite, monazite and xenotime [1] Table 1.Bastnasite and monazite are sources of LREEs.

Xenotime and minerals such as allanite are sources of the HREEs and yttrium. The lanthanides become important elements; they are now used in a great variety of applications such as in petroleum cracking crystals in its refining, in flint stones, ignition device, superconductor, hydrogen storage, polishing powder, laser materials and special optical glasses. Also, lanthanides compounds containing lanthanum are extensively used in carbon lighting applications, especially by the motion picture industry for studio light and projection and as electron dense tracer in molecular biology [2].

Table 1: Some of the important minerals of lanthanides

Mineral	Composition
Bastnasite	$CeFCO_3$
Monazite	$(Ce,La,Th,Y)PO_4$
Xenotime	YPO_4

Euxenite	$(Y,Ca,Ce,U,Th)(Nb,Ta,Ti)_2O_6$
Apatite	$Ca_5(po_4)_3F$
Gadolinite	$Be_2FeY_2Si_2O_{10}$
Loparite	$(Ce,Na,Ca)(Ti,Nb)_2O_6$
Uraninite	$(U,Th,Ce,Y,Pb)O_2$
Brannerite	$(U,Ca,Fe,Th,Y)_3Ti_5O_{16}$
Doverite	$CaY(CO_3)2F$
Pyrochlore	$(Na,Ca,RE)_2Nb_2O_6(F,OH)$
Allanite	$(Ca,RE,Th)_2(Al,Fe,Mg)_3.SiO_{12}(OH)$
Perovskite	$CaTiO_3$
Zircon	$(Zr,Y,Si,P)O_4$
Fluorite	$(Ca,Ce)F_2$ or $(Ca,Y)F_2$

In addition, lanthanides have a variety of nuclear application. Because they absorb neutrons, they have been employed in control rods used to regulate nuclear reactors. They have also used as shielding materials and as structural components in reactors. Some lanthanides have unusual magnetic properties. For instance, cobalt-samarium magnets are very strong permanent magnets [3]. New uses for lanthanides, as metals and as compounds, in various technologies are under development by many companies:

1. in Glass - as a melting aid to reduce the temperature required for melt homogeneity;
2. in Metal - as dispersion strengtheners in high performance alloys;
3. in Ceramics - as sintering aids in the production of new ceramics;
4. in Catalysts - for the control of sulfur dioxide emissions.

Extraction of lanthanides started by leaching process which can be summarized in Table2.

Table 2: Summary of leaching technologies in primary REE production

Mineral	Process	REE yield	Remarks	Status	Ref.
Bastnaesite	1-Cl leach to remove non REE carbonate. 2-alcination of residue to form REO	85- 90%	The oldest way to process bastnaesite concentrates	outdated	[4]
	Digestion with HNO_3 or H_2SO_4	98%	Acid choice depends on further processing: solvent extraction HNO_3 precipitation H_2SO_4	outdated	[4]
	1-Roast at 620 °C to drive of CO_2 2-30% HCl leach	- -	Ce+III oxidises to Ce+IV during roasting will not LeachREE fluorides willnot leach, residue is marketable	outdated	[4]
	1-Sulphuric acid roast 2-NaCl solution leach 3-Precipitation as Na double sulphates	- -	Precipitates are converted tochlorides for further purification with solvent extraction	In use	[4]
Monazite	1-Digestion in hot 60- 70% NaOH 2-Washing residue with hot water 3-Leach with mineral acid of choice	98%	Ce cannot be leached if Mn is present Th is leached together with REENa$_3$PO$_4$ is marketable by-product		[4-5]
Ion clay	Salt leach with $(NH_4)_2SO_4$	80- 90%	Targets physiosorbed REEthrough cation exchange	In use	[6]
	Leach with eawater	40%	Inefficient but cheap process	R&D	[7]
	Acid leach with strong acid (pH<1)	All	Dissolves entire clay, incurs significant additional costs	Not used	[6]

The study area lies in the southern part of the Eastern Desert of Egypt at the boundary between the Central Eastern Desert (CED) and the Southern Eastern Desert (SED). It is limited by latitudes 24° 35` 00`` and 24° 41` 22``N and longitude 34° 44` 13`` and 34° 49` 30``E at about 90 km southwest of MarsaAlam City on the Red Sea coast Figure 1.The mineralized cataclastic rocks of the study area occur thrusting under the ophiolitic mélange and intruded from east by biotite granites. The rocks are highly altered and featured by the frequent presence of pegmatite lenses extending parallel to the foliation planes. The cataclastic rocks were affected by many alteration processes as a result of hydrothermal solutions. The cataclastic rocks are mineralized by polymineralization (U, Th, Pb, Nb,-Ta, and Zr) [8] and classified into protomylonite, mylonite, ultramylonite and silicified ultramylonite with graditional contacts.
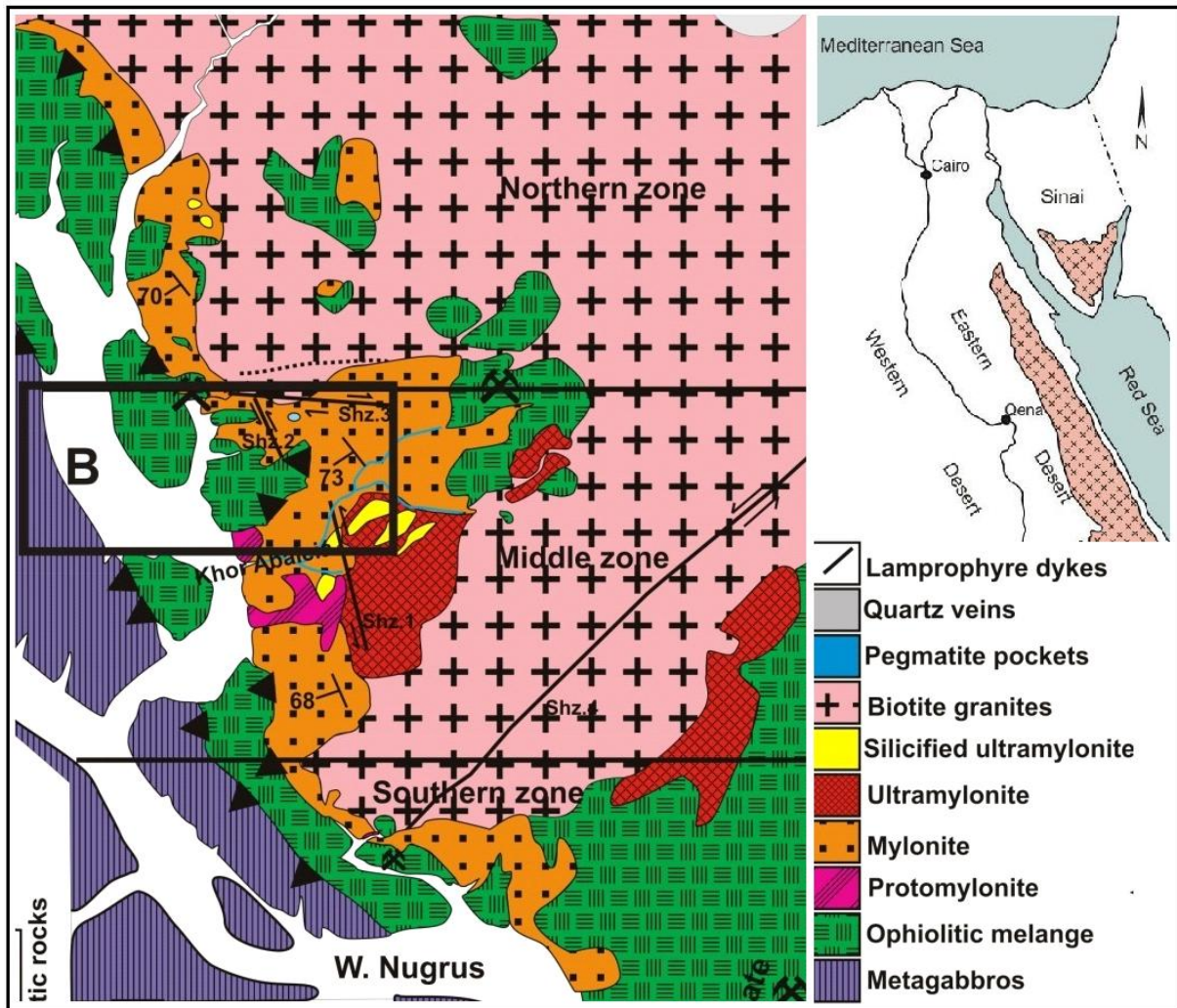

Figure 1: Geological map showing shear zone II at middle zone of W. Abu Rusheid area, SED, Egypt; the rectangle represents location of the shaft (B) drilled and studied by [8]

In mylonite, accessory minerals occupy about 2.2% of the rock as xenotime and zircon. Xenotime present as minute crystals associating or included in the enlarged crystals of zircon. Zircon found as fine crystals included in kyanite or biotite and as enlarged crystals associating the essential minerals. Radioactive minerals are mainly kasolite and meta-zeunerite. Kasolite mostly present as prismatic crystals and as relics assembled with quartz and meta-zenurite. Meta-zeunerite present as green tabular crystals of tetragonal system; characterized by green color. It is also present on the periphery of altered crystals assembled of kasolite (as relics), quartz and opaque. It is well-recognized by XRD technique [8]. In Abu Rusheid area, south Eastern Desert of Egypt, polymineralized (H REEs, U, Zn and Pb) lamprophyre dykes cutting cataclastic rocks along share zones in N-S direction are recently recorded. The secondary U-minerals are essentially represented by secondary uranophane, kasolite, torbernite and autunite [9]. Experimental work was carried out for the recovery of uranium and other valuable elements (e.g. Zn, Al and Pb) from a lamprophyre dykes technological sample. The optimum conditions were (100 g /l sulfuric acid concentration, 1:5 S/l and soaking for 15 days at a room

temperature. The leaching efficiency for recovery of uranium and other elements like Al, Zn and Pb were 40, 33.6, 98.8 and 78 respectively, from the lamprophyre dykes southeastern Desert of Egypt [10]. Leaching of thorium from representative samples collected from Abu Rusheid area, using sulphuric acid has been studied. Factors affecting the leaching of thorium were investigated such as, acid type, S/L ratio, H_2SO_4 concentration, leaching time, temperature and grain size. The best conditions for leaching of thorium were, H_2SO_4 acid as the best acid, S/L ratio of 1:5, 3.0 M H_2SO_4, 2.0 hour of leaching time, -200 mesh size at 90°C. After leaching, the mixture was filtrated then divided into two parts the leach liquor and the residue. The leach liquor was adjusted at pH 1.0 by ammonia solution and addition 10 % oxalic acid. The precipitate obtained was filtrated and dried at 120 °C for 1 hour. The obtained precipitate showed 91.60 % of thorium content when using 3.0 M H_2SO_4 acid. The quality of the precipitate was confirmed by XRF [11]. The aim of the present work is to study the leachability and extraction of total lanthanides from Abu Rusheid mineralized mylonite sample by applying acid agitation leaching technique and obtain its end product lanthanides concentrate. This product will be available for further economic applications.

II. EXPERIMENTAL

2.1. Characteristics of the study mineralized sample

The representative composite sample used in this study was collected from Abu Rusheidmylonitemineralized sample. This sample was mixed, quartered, crushed, grinded and then sieved. Chemical analysis for most major, trace and lanthanide elements of the study sample were analyzed in the Bureau Veritas, Mineral Laboratories of Canada are shown in Table 3. The chemical analysis of the individual lanthanides elements was done in the Egyptian Mineral Resources Authority, Central Laboratories Sector. Most of the used reagents and solutions are as follow: Hydrochloric acid (37%, POCH, Poland), Nitric acid (65 % Merck, Germany), Sulfuric acid (95.97%, Sigma, Germany), Oxalic acid, Sodium hydroxide (98%, Winlab, U.K.). Arsenazo (III) (pure, Sigma chem. C. O. USA). Ferrous sulphate. Salfosalysilic acid (pure, Kochlight labs. Ltd., England). Diphenylamine-4- sulfonic acid sodium salt. Boric acid (99%, Hungary). Acids of laboratory grade were used in the leaching and chemical analysis experimental work.

2. 2. Spectrophotometer analysis

Concentration of lanthanides was analyzed using a double beam of UV-Vis spectrophotometer model Jasco V-530 with a wavelength (650 nm). A standard calibration was constructed Figure 2 to estimate the lanthanides content in each studied solution using Arsenazo (III) method. In this method, a 25 ml measuring flask 0.25 mL.

Table 3: Chemical analysis of major, trace and lanthanides of Abu Rusheid mineralized study sample

Element	Method Analyte Unit MDL	Rock Pulp	Element	Method Analyte Unit MDL	Rock Pulp	Element	Method Analyte Unit MDL	Rock Pulp
Mo	0.05ppm	0.63	Cr	1ppm	8	Tb	0.1ppm	9.2
Cu	0.1ppm	65.9	Mg	0.01%	0.02	Dy	0.1ppm	82.4
Pb	0.02ppm	498.76	Ba	1ppm	10	Ho	0.1ppm	23.8
Zn	0.2ppm	279.9	Ti	0.001%	0.007	Er	0.1ppm	95.5
Ag	20ppb	*	Al	0.01%	6.03	Tm	0.1ppm	22.4
Ni	0.1ppm	6.5	Na	0.001%	4.812	Yb	0.1ppm	167.4
Co	0.2ppm	0.4	K	0.01%	0.46	Lu	0.1ppm	27.0
Mn	1ppm	140	W	0.1ppm	5.7	Hf	0.02ppm	22.60
Fe	0.01%	2.06	Zr	0.2ppm	430.5	Li	0.1ppm	50.3
As	0.2ppm	1.8	Sn	0.1ppm	7.4	Rb	0.1ppm	90.3
U	0.1ppm	188.8	Be	1ppm	5	Ta	0.1ppm	153.8
Th	0.1ppm	997.2	Sc	0.1ppm	0.5	Nb	0.04ppm	1029.55
Sr	1ppm	35	S	0.04%	0.29	Cs	0.1ppm	0.4
Cd	0.02ppm	0.89	Y	0.1ppm	478.5	Ga	0.02ppm	60.82
Sb	0.02ppm	0.17	Ce	0.02ppm	135.57	In	0.01ppm	1.00
Bi	0.04ppm	36.11	Pr	0.1ppm	17.9	Re	0.002ppm	0.005
V	1ppm	2	Nd	0.1ppm	42.8	Se	0.3ppm	2.2
Ca	0.01%	0.25	Sm	0.1ppm	18.1	Te	0.05ppm	<0.05
P	0.001%	0.001	Eu	0.1ppm	0.3	Ti	0.05ppm	0.32
La	0.1ppm	34.0	Gd	0.1ppm	19.1			

lanthanides solution sample, 1 mL 1 % ascorbic acid, 0.5 ml format buffer, 2ml Arsenazo-III aqueous solution and adjust pH at 2.6 then up the volume to 25 ml deionized water to the mark [12].
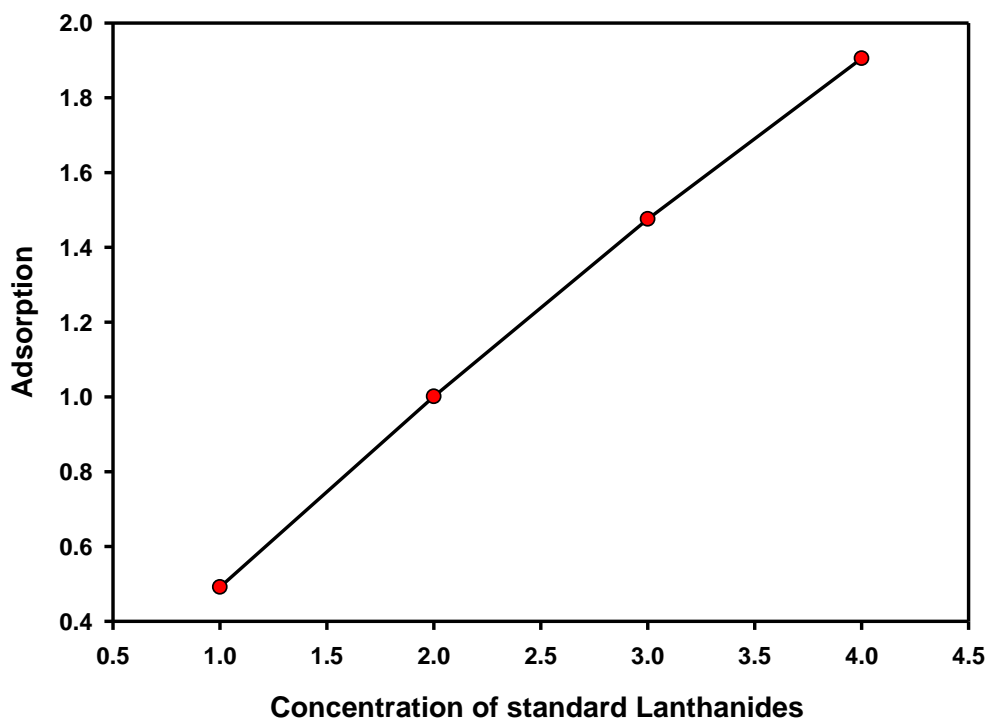
Figure 2: Standard calibration curve for lanthanides

The total lanthanides content in the study sample analyzed by this method was 3200 ppm. Environmental Scanning Electron Microscope (ESEM) model XL30. The visible spectra of metal complex of Ln and Arsenazo (III) were measured at 650 nm using a double beam spectrophotometer model Shimadzu 160A.

2. 3. Acid agitation leaching
To study and determine the optimizing factors affecting the agitation leaching of lanthanides, different series of agitation leaching experiments were performed. Certain weight of the study material, ground and sieved to different sizes, was mixed well and agitated with a suitable volume of different acids concentrations. The obtained slurry was filtered and the residue left behind was thoroughly washed with distilled water and both filtrate and washings were made up to volume before analysis of the leached lanthanides. The studied factors were: type of acid, acid concentration, grain size, agitation time, and solid / liquid ratio.

III. RESULTS AND DISCUSSION
3.1. Relevant factors affecting lanthanides leaching
3.1.1. Effect of acid type:
Different acids were studied such as sulfuric, nitric and hydrochloric acid for maximum leaching of REEs from the study Abu Rusheid mineralized sample. The experimental conditions were fixed at 50 g/L acid concentration, 2 hrs agitating time, 1/3 solid / liquid ratio at room temperature 25 $^\square$C and (- 0.075mm) grain size. The obtained REEs analysis revealed that hydrochloric acid is the most effective leaching agent Figure 3.
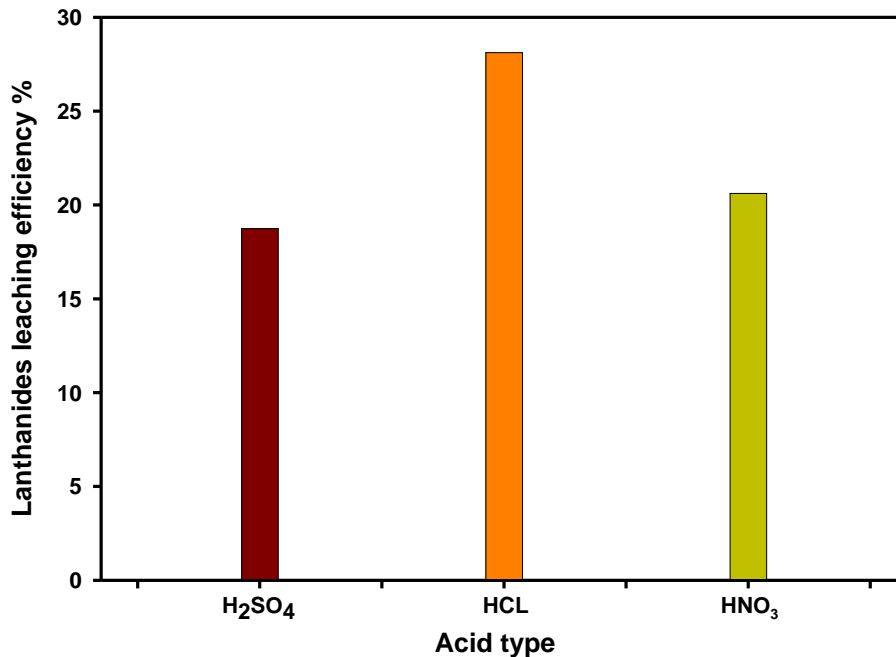
Figure 3: Effect of acid type upon Abu Rusheid REEs leaching efficiency %

3.1.2. Effect of different hydrochloric acid concentration

Different concentrations of the hydrochloric acid were used (50, 75, 100, 125 and 150 g/L) as a leaching agent of lanthanides with fixing the other leaching conditions at 2 hrs agitating time, 1/3 solid / liquid ratio at room temperature (25 $^\square$C) and - 0.075mm grain size. The obtained results indicated that a concentration of 150 g/L was the optimum one (43% leaching efficiency) Figure 4. From economic point of view, no need to use acid concentration above 150 g/L.
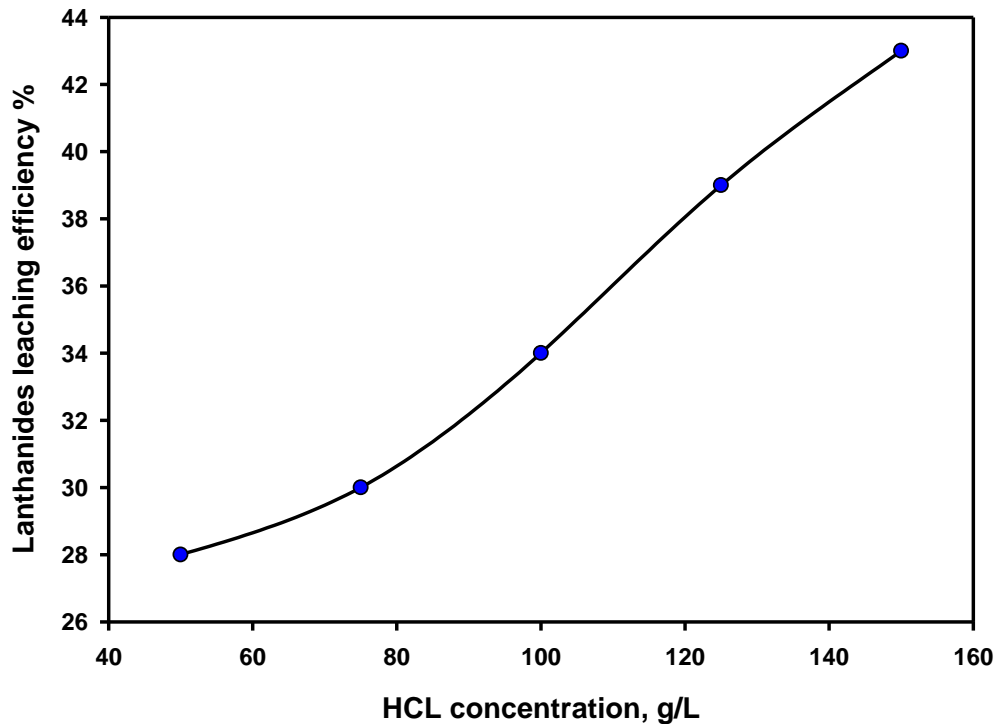

**Figure 4: Effect of acid concentration upon Abu Rusheid lanthanides
Leaching efficiency %**

3.1.3. Effect of contact time

This factor was studied where different contact times (2, 4, 6, 8 and 12 hours) and the other leaching conditions were fixed at 150 g/L hydrochloric acid, 1/3 solid /liquid ratio, at room temperature 25 $\square$C and (-0.075mm) grain size. It was concluded that 4 hours contact time was the optimum for maximum lanthanides leaching Figure 5, as the nature of the study ore (hard and massive granite) needs strong acid and long contact time.
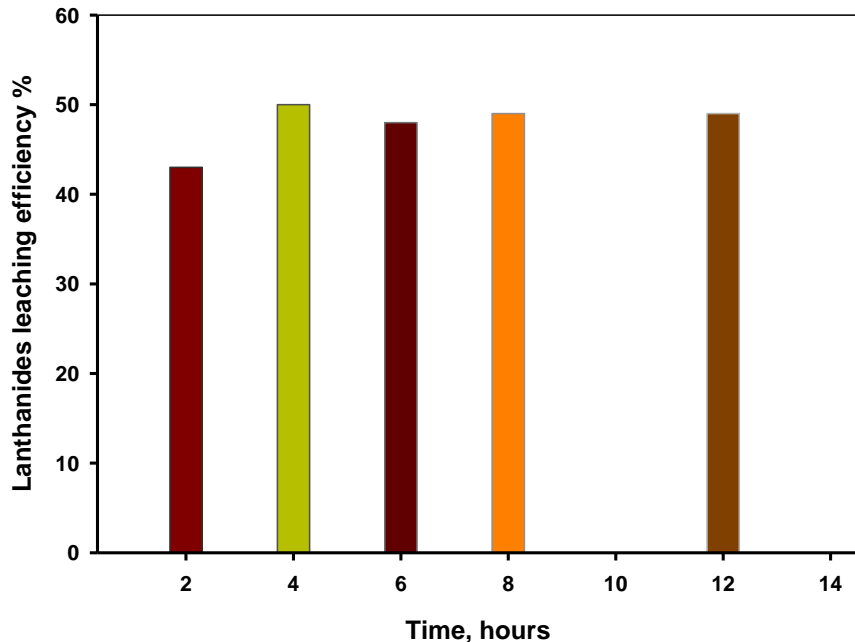

Figure 5: Effect of contact time upon Abu Rusheid lanthanides leaching efficiency %

3.1.4. Effect of temperature

It was necessary to study temperature effect upon lanthanides leaching where different temperatures were studied from 25 $\square$C, 40 $\square$C, 60 $\square$C, 80 $\square$C and 100 $\square$C. These experiments were performed under the following conditions: 150 g/L hydrochloric acid, 1/3 solid /liquid ratio, 4 hours contact time and (-0.075mm) grain size. It was observed from data illustrated in Figure 6 that 100 $\square$C is the highest leaching efficiency reached (82 %).
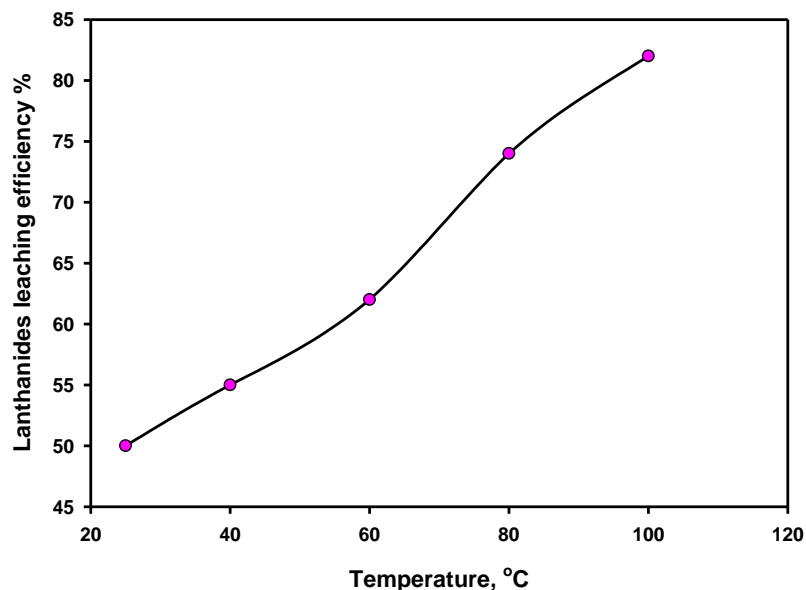

Figure 6: Effect of Temperature upon Abu Rusheid lanthanides leaching efficiency %

3.1.5. Effect of grain size

Different grain size was used such as (-0.25 +0.125 mm), (-0.125 +0.075 mm) and (-0.075mm). The obtained results revealed that the grain size of (-0.125+0.075mm) was the effective size for lanthanides leaching efficiency 86 % Figure 7 under the following conditions: 150 g/L hydrochloric acid, at temperature 100 °C, 4 hours contact time and 1/3 solid/liquid ratio.
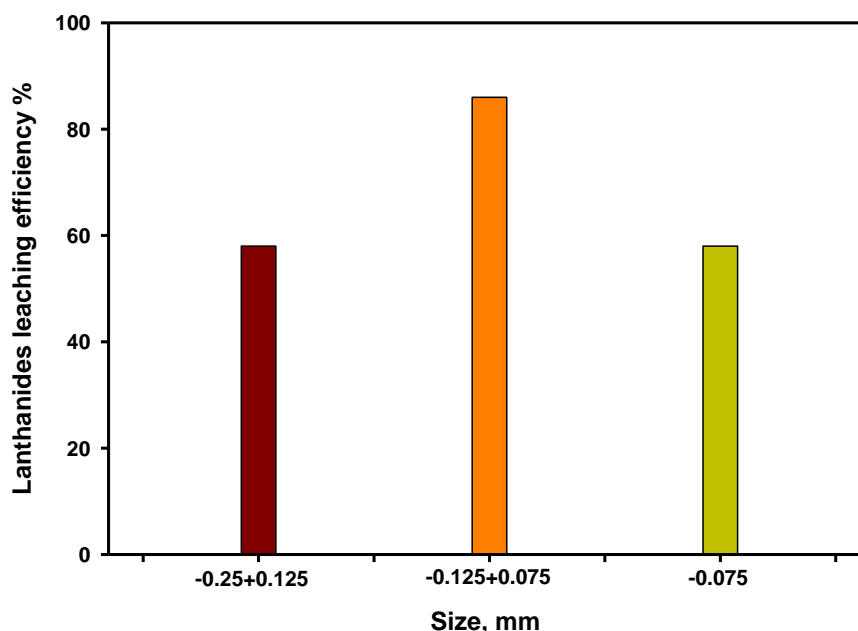

Figure 7: Effect of grain sizeupon Abu Rusheid lanthanides leaching efficiency %

3.1.6. Effect of Solid / liquid ratio

A group of different solid/liquid ratios such as 1/2,1/3 and 1/4 was performed upon the study Abu Rusheid mineralized sample at150 g/L hydrochloric acid, 100 °C, 4 hours contact time and (-0.125+0.075 mm) grain size. The obtained results revealed that a solid/liquid ratio of 1/4 was the effective ratio for maximum lanthanides leaching efficiency 89% Figure 8.
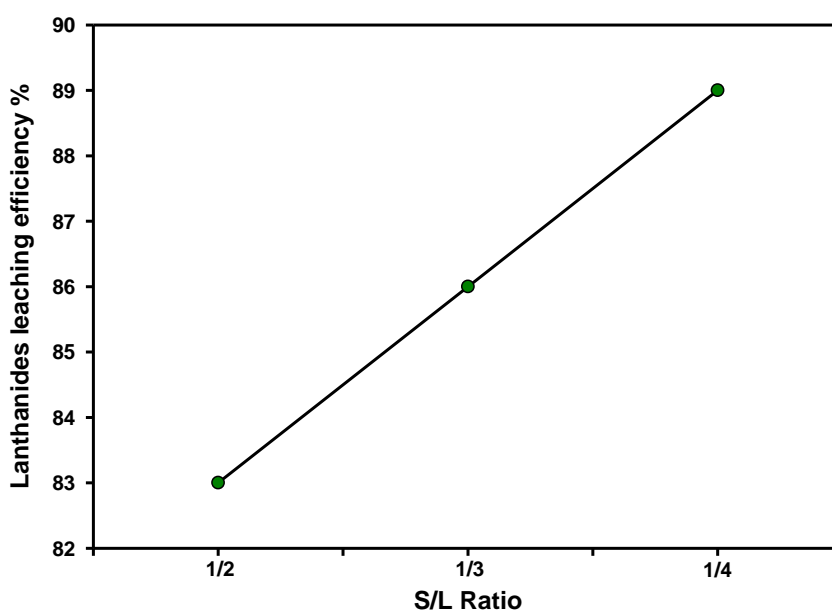

Figure 8: Effect of solid /liquid ratio upon Abu Rusheid lanthanides
Leaching efficiency %

3.2. Leaching efficiency at different temperatures and different time

Plotting of lanthanides leaching efficiency at different temperatures and time intervals measured at 150 g/L hydrochloric acid, temperature 100 □C, (-0.125+0.075 mm) grain size, 4 hours contact time and 1/4 Figure 9, indicates its increase with increasing temperature and time.
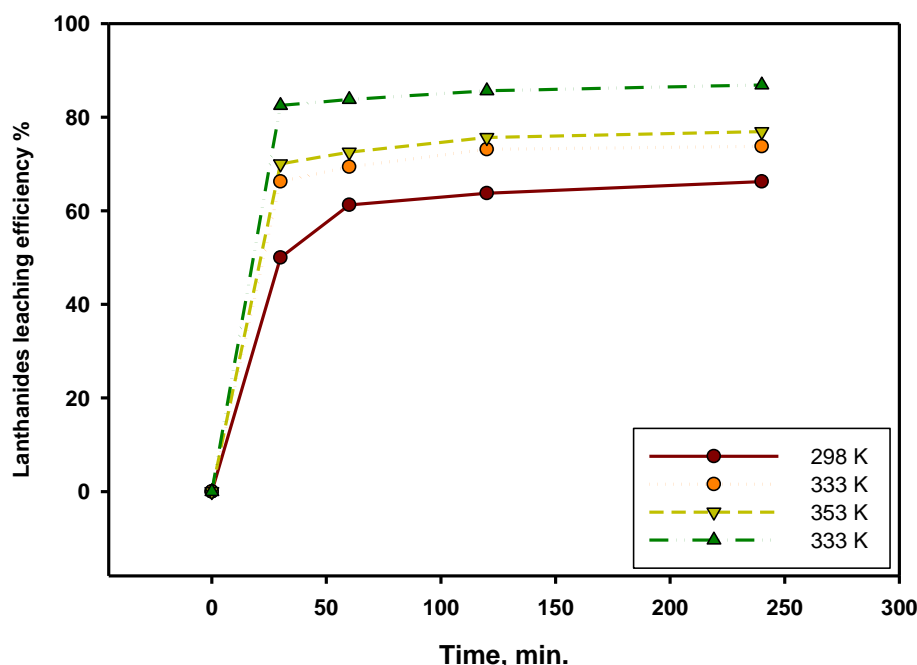

Figure 9: Plotting of lanthanides leaching efficiency % at different Temperatures and time

3.3. Thermodynamic characteristics

The thermodynamic parameters for leaching process including Gibbs [13] free energy of adsorption $\Delta G\square$ (kJ/mol), enthalpy change $\Delta H\square$ (kJ/mol), and entropy change $\Delta S^{\square}$ (J/mol.K), were calculated using Van't Hoff equations using the following formula:

$$lnk = {\Delta S^{\square}}/_R - {\Delta H^{\square}}/_{RT}$$

Where k is the equilibrium constant= 8.3145 J mol-1K^{-1} and T = absolute temperature in Kelvin. ΔH^o and ΔS^o were determined from the slope and intercept of lnK versus 1/T Figure 10, where K^{-1} gives a straight line. Values of ΔH^o and ΔS^o have then been used to obtain the corresponding free energy ($\Delta G^{\square}$) at different temperatures using the free Gibbs energy of sorption ($\Delta G^{\square}$, kJ mol^{-1}) according to the following equation:

$$\Delta G^{\square} = \Delta H^{\square} - T\Delta S^{\square}$$

Table 4 summarizes the thermodynamic parameters for lanthanides leaching. The positive values of enthalpy change, $\Delta H^{\square}$, for lanthanides confirmed the endothermic nature of the hydrochloric acid leaching upon the study sample. The positive values of entropy change, $\Delta S^{\square}$, for the lanthanide ions, suggested an increase in the randomness during lanthanides leaching process. The negative values of Gibbs free energy of lanthanides expecting the spontaneous reaction at any temperature and the increase in negative charge of ΔG° value with increasing the temperature implies that the reaction is favorable at higher temperature.
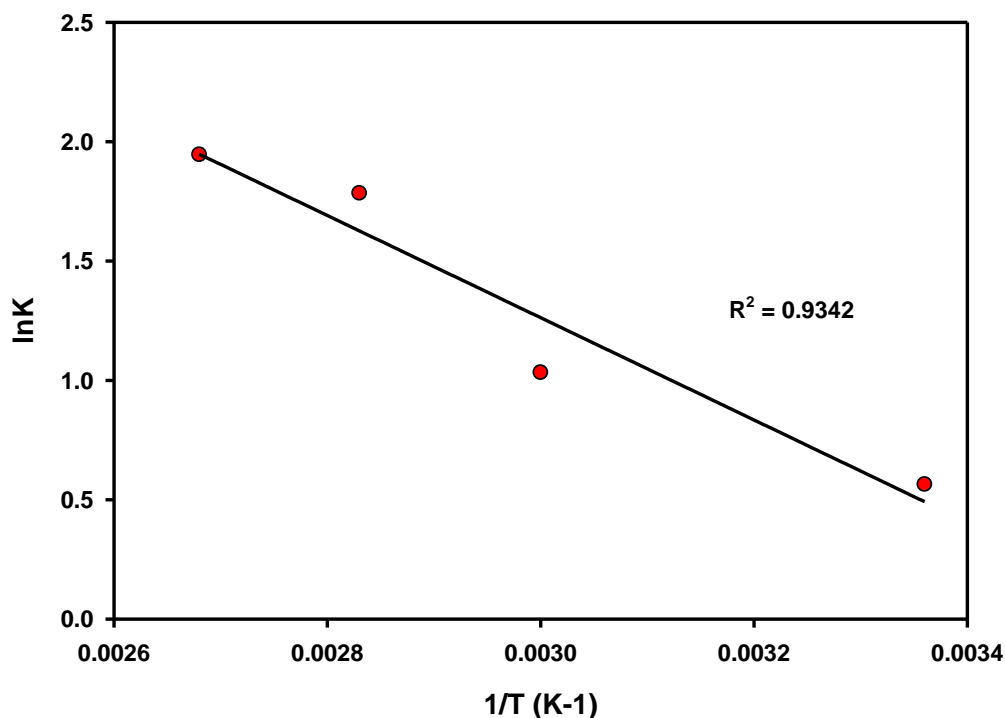
Figure 10: Plot of lnk versus 1/T for lanthanides in leaching from Abu Rusheid mineralized sample

Table 4: Thermodynamic parameters of lanthanides leaching upon Abu Rusheid sample at different temperatures.

$\Delta G^\square$ At different temperatures					$\Delta S^\square$ kJ/mol	$\Delta H^\square$ kJ/mol
Temp. $^\square$C	298K	333K	353K	373K		
$\Delta G^\square$	-1.469066	-2.688196	-4.643738	-5.063807	17.9795	0.0641

Therefore, 150 g/L hydrochloric acid, at temperature 100 $^\square$C, -0.075 mm grain size, 4 hours contact time and 1/4 were the optimum conditions for maximum lanthanides leaching.

3.3. Extraction process

A batch experiment was performed where 20 g of the study Abu Rusheid mineralized sample was subjected to the previously studied optimum leaching conditions, 150g/L HCl acid, 4 hours contact time, 1/4 S / L ratio, (-0.125+0.075 mm) grain size and temperature (100 $^\square$C) using condenser system. The obtained leach liquor (200 ml) assaying 2600 ppm (81.25% leaching efficiency) total lanthanides was subjected to pH adjustment at 4 where iron was precipitated while by rising its pH to 11 lanthanides was precipitated (about 0.9 g) leaving nearly no lanthanides in the filtrate. The obtained lanthanides cake was investigated by the environmental scanning microscope (ESEM) Figure 11 where it revealed the presence of some valuable elements. Elements content of the precipitated lanthanides cake after analysis by the ICP are shown in Table 5.
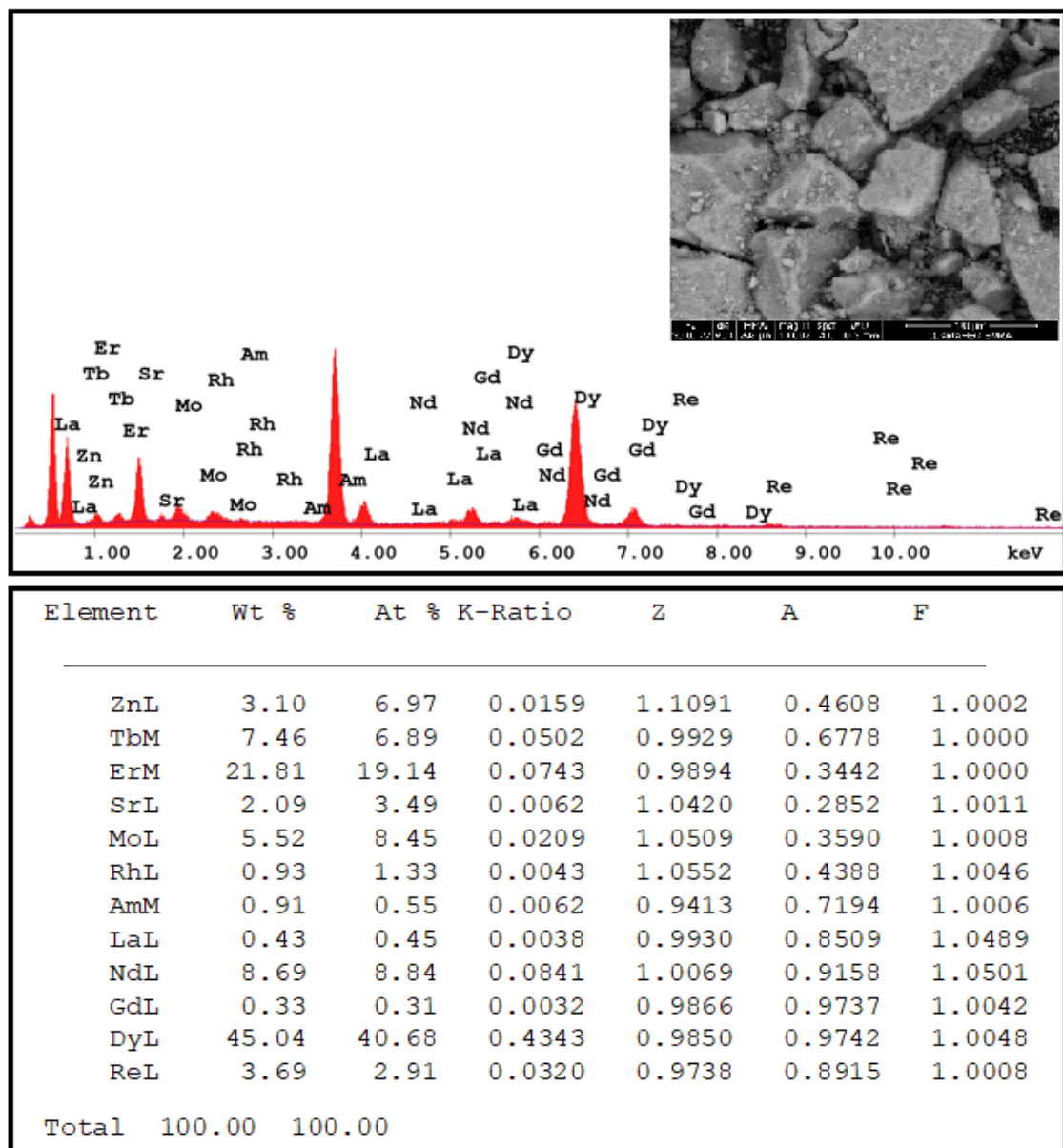
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Element      Wt %      At %  K-Ratio       Z         A         F

ZnL         3.10      6.97    0.0159     1.1091    0.4608    1.0002
TbM         7.46      6.89    0.0502     0.9929    0.6778    1.0000
ErM        21.81     19.14    0.0743     0.9894    0.3442    1.0000
SrL         2.09      3.49    0.0062     1.0420    0.2852    1.0011
MoL         5.52      8.45    0.0209     1.0509    0.3590    1.0008
RhL         0.93      1.33    0.0043     1.0552    0.4388    1.0046
AmM         0.91      0.55    0.0062     0.9413    0.7194    1.0006
LaL         0.43      0.45    0.0038     0.9930    0.8509    1.0489
NdL         8.69      8.84    0.0841     1.0069    0.9158    1.0501
GdL         0.33      0.31    0.0032     0.9866    0.9737    1.0042
DyL        45.04     40.68    0.4343     0.9850    0.9742    1.0048
ReL         3.69      2.91    0.0320     0.9738    0.8915    1.0008

Total     100.00    100.00
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Figure 11:ESEM micrograph and the corresponding EDX spectrum of the lanthanides oxalate precipitate

Table 5: Chemical analysis of elements content in Abu Rusheid lanthanides cake

Element	Elements content of Abu Rusheid ore (g/kg)	Abu Rusheid lanthanide cake (g/Kg)	Extraction percentage
La	2.6	0.7	27.0
Ce	13	4.0	31.0
Dy	3.5	3.1	88.6
Er	6.0	3.1	52.0
Eu	-	-	-
Gd	1.8	1.0	56.0
Ho	1.3	1.1	85.0
Lu	1.7	0.6	35.3
Nd	3.2	3.0	93.8
Pr	2.0	1.8	90.0
Sm	17	0.4	2.4
Tb	0.8	0.2	25.0
Tm	0.9	0.5	56.0
Y	36	18.0	50.0
Yb	10.6	4.7	44.4
U	15.2		
Th	72		

The obtained Abu Rusheid lanthanide cake is characterized by the presence of Y, Dy, Gd, Nd, Pr and Ho elements with a considerable percentage of leaching and precipitation. Such precious elements are very important in the nuclear industry. Summing up the lanthanides leaching study of Abu Rusheid mineralized rocks is represented in the following flowsheet:
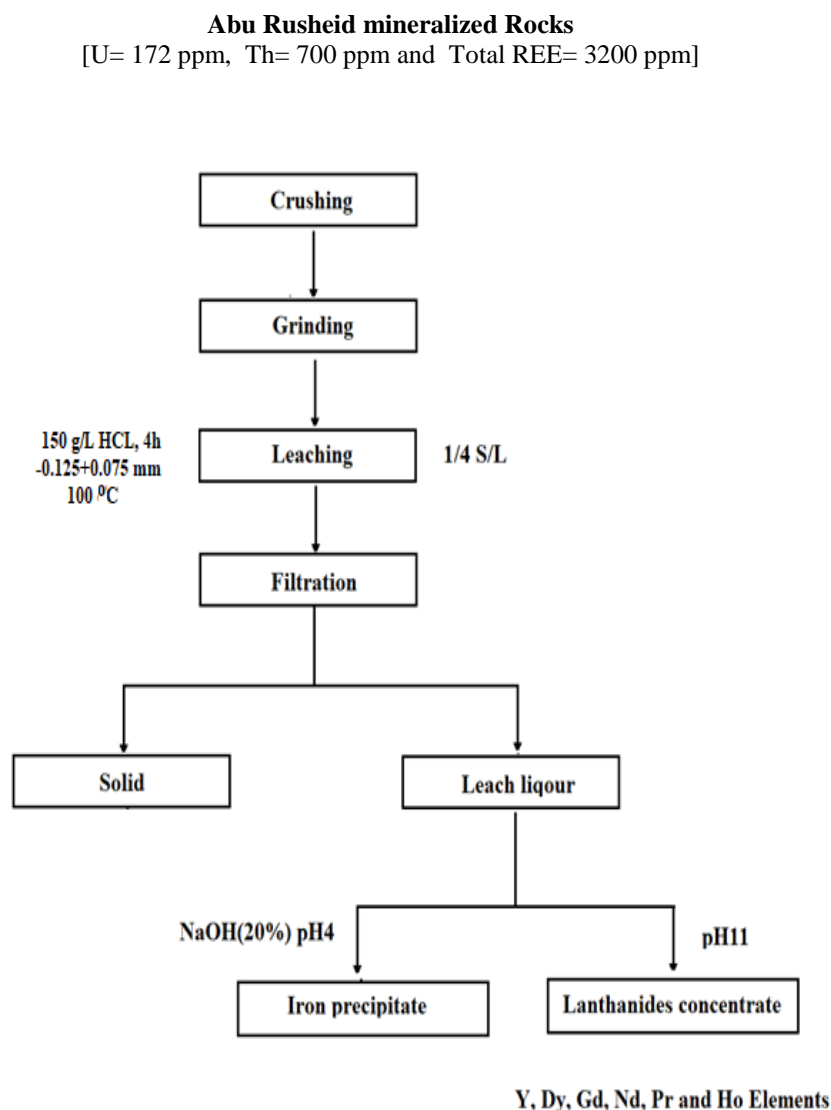
Abu Rusheid mineralized Rocks
[U= 172 ppm, Th= 700 ppm and Total REE= 3200 ppm]

Figure 12: A proposed technical flowsheet for Abu Rusheid lanthanides precipitate

IV. Conclusion

According to the policy of the Nuclear Materials Authority of Egypt, it is very important to get use of the radioactive mineralized ores. As Abu Rusheid is one of the most promising occurrences, the present work dealt with the extraction of lanthanides from its bearing ore. This was carried out through both lanthanides leaching and extraction processes where their optimum leaching conditions were studied concluding that 89% leaching efficiency was obtained under the following leaching conditions: 150 g/L hydrochloric acid concentration, at temperature 100 $^\circ$C, (- 0.075 mm) grain size, 4 hours contact time and ¼ solid/liquid ratio. The positive values of entropy change, ΔS°, for the lanthanide ions, suggested an increase in the randomness during lanthanides leaching process and the increase in negative charge of ΔG° value with increasing the temperature implies that the reaction is favorable at higher temperature. Lanthanides extraction process was performed upon the prepared Abu Rushieid leach liquor where iron was precipitated at pH 4 by sodium hydroxide solution. Lanthanides cake was also precipitated from the filtrate at pH 11 which characterized by the presence of Y, Dy, Gd, Nd, Pr and Ho elements. From this work, it is recommended to conduct extraction of the most important elements individually in further work.

Acknowledgement

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