

Leaching and Separation of Some Strategic Metals from Abu Furad Area, Central Eastern Desert, Egypt

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Abstract:

leaching and extraction of U (VI) and lanthanides from radioactive pegmatite pockets found in Gabal Abu Furad area, southwest Safaga city, Central Eastern Desert, Egypt. Optimum conditions for leaching was investigated using different acids, solid/liquid ratio, contact times, and temperature. Separation of U (VI) as sodium diuranate followed by application of N-octanoyl-3-methylpiperidine, a novel extractant, was carried out and used for the extraction and recovery of Nd (III) from its pregnant sulfuric acid leaching solution. Kerosene was found to be the most suitable diluent for the extractant compared with the other diluents tested. Extraction percent of Nd (III) has been studied as a function of extractant concentration, pH, diluents, and equilibration time. In addition, studying the principal factors affecting the stripping efficiency. Finally, an application of the extraction of Nd (III) from the pregnant leach solution using this extractant Nd_2O_3 85.55 % is obtained from lanthanides concentrate of 3.00 % Nd_2O_3 .

Keywords: Liquid-liquid extraction; uranium; neodymium; n-octanoyl-3-methylpiperidine

1. Introduction

Uranium has become a widely used source for nuclear energy especially for the nuclear electrical generation plants and nuclear weapons (1). Lanthanides are a unique group of metals regarded as being the most critical elements that are highly valued for their specialized application in up-to-date technologies. In recent years, lanthanides are significant topic of interest in the industrial metals due to global supply shortage and highly demand (2, 3). This has led many companies to develop new processing techniques for extracting lanthanides from less common lanthanides bearing deposits(4) and reclaiming lanthanides from end-of-life products by recycling spent batteries, phosphors and permanent magnets (5, 6). Lanthanide element play a critical role on the global economy due to numerous applications for those elements. For instance, the global demand for Nd (III) is projected to grow by 700% in the next 25 years (7). Neodymium which is one of the most abundant rare earths, is of current commercial interest as it is the raw material for high-strength permanent magnets (Nd-B-Fe), which cost less than samarium-cobalt permanent magnets(8).

Recovery and purification of U(VI) and lanthanides is a complex process which involves mining the ore bearing uranium and lanthanides minerals like monazite and bastnaesite, followed by mineral dressing, chemical upgrading and refining procedures(9). Physical dressing techniques such as froth flotation, magnetic separation and gravity separation are not suitable methods for obtaining high grade concentrate (10). These techniques must be followed by processes like leaching using in most cases acidic solutions (11-13) and solvent extraction, which have subsequently shown a higher percentage recovery of lanthanides from the pretreated ore (14). Leaching is a widely used extractive metallurgical technique, for the extraction of the metal of interest from its ore or concentrate. Generally, strong acids were used for the highest recovery of

lanthanides which dissolves the desired elements present in the ore into the solution (15). The major advantage of the leaching process is that it selectively dissolves the metals inherent to the ore, leaving behind most of the impurities. This reduces the processing difficulties and hence makes the extraction step simpler. A range of separation technologies can be used to effect transfer of the desired metal from the aqueous pregnant leach solution. For recovery of metals the preferred water-immiscible phase is a high boiling hydrocarbon on grounds of both cost and safety. The use of such non-polar solvents facilitates assembly reactions based on hydrogen-bonding and on ion-pair and ion dipole interactions of solutes. An essential criterion for any commercial exploitation of organic complexing agent in metal recovery is that they are inexpensive (16).

The most commonly used are based on equilibria involving liquid-liquid extraction and liquid-solid (ion exchange and chelating resins) systems. Solvent extraction has the advantages of being easily engineered for continuous operations and has proved remarkably promising since its introduction for uranium production (17). In recent years, amides have been proposed as alternatives for phosphorous-containing compounds as extractants in the nuclear fuel reprocessing because of many advantages, such as complete incinerability, high chemical and radiolytic stability, simplicity of synthesis and easy removal of the degradation products (18-20). The structures of extractants have been a major important in the development of new extractants with a potential high extracting power for metal ions. Yuan, Xu et al (21, 22) addresses that electron density at the active site, steric effects and lipophilicity of an extractant are the main factors affecting the extracting power for metal ions.

2. Aim of the work

According to the previous mineralogical studies (23) and chemical analysis of El Galy (24) which revealed the presence of high content of

both U(VI) and lanthanides and its leaching study of Kandil et. al., (25) Which provided an enriched REEs concentrate. This previous work makes it very important to perform a purification study using solvent extraction study on a high lanthanides concentration representative sample after U(VI) precipitation as sodium diuranate. The main objective of the present work is the U(VI) separation and individual separation of Nd(III) from crude lanthanides, which was precipitated and identified by chemical analysis.

3. Materials and Methods

3.1. Ore Composition

Radioactive mineralized pegmatites was found at Gabal Abu Furd area which is located 40 Km south west of Safaga city on the Red sea coast, limited by latitudes 26o 37' and 26o 43' and longitudes 33o 36' and 33o 43' E. Central Eastern Desert. The chemical analysis of the studied ore material is given in Table 1 for the major oxides. ICP-OES was used for analysis of the lanthanides content in the radioactive pegmatite sample as shown in Table 2. U.V. spectroscopy instrument was used to identify U (VI) concentration which was about 2020 ppm.

Major oxides	Average (wt.%)	Trace Element (ppm)	Average
SiO ₂	74.45	Zr	112
TiO ₂	0.11	Y	34
Al ₂ O ₃	13.47	Sr	37
Fe ₂ O ₃	1.12	Ba	38
FeO	0.29	Rb	63
MnO	0.40	Nb	31
MgO	0.30	Cu	32
CaO	0.84	Ni	6
Na ₂ O	3.64	Cr	11
K ₂ O	4.42	Zn	26
P ₂ O ₅	0.98	Ga	17
LOI	0.7	Pb	5
Total	99.51	S	4
		V	11

Table 1: Major oxides and trace elements analysis for the radioactive pegmatite (24)

Element	L	Ce	Pr	Nd	Sm	Gd	Dy	Er	Yb	Lu	S	Y	Th
Conc. (ppm)	5	13	2	3	3	3	2	1	1	3	5	3	1
	1	92	7	0	1	3	5	8	0	0	9	8	0
	6	1	8	0	5	0		0	5		5	5	4
	0		0	5				0					5

Table 2: ICP-OES analysis of the lanthanides content in the radioactive pegmatite Sample.

3.2. Preparation of radioactive pegmatite leach liquor

Different leaching conditions were studied to obtain the maximum leaching efficiency of U (VI) and lanthanides. These conditions include different acid leaching agents, contact time, different concentration from the used acid, solid/liquid ratio and temperature. The leaching percent of the U (VI) and lanthanides was calculated from the analysis of the pregnant leach solution after filtering and washing the slurry.

3.3. Solvent preparation

3.3.1. Synthesis of N-octanoyl-3-methylpiperidine.

These amides were prepared by acylation of cyclic amines (piperidine) with long-chain acid halides in a two-phase system of CHCl₃ and H₂O: K₂CO₃ (26).

The N-octanoyl-3-methylpiperidine used for investigation was synthesis in

two steps, as it was not commercially available. The piperidine based amides, was prepared according to a modified literature procedure (27). The relevant acid chloride was first prepared by reaction of the parent acid with thionyl chloride, and then reacted with 3-methylpiperidine (10g, 0.100827 mol) in chloroform (50 ml) was added to a solution of potassium carbonate (10g) in distilled water (50 ml) and the mixture stirred at room temperature for 3 hrs. Octanoyl chloride (5.7g mol) added to the reaction mixture and stirred for 2 hrs. The reaction mixture was cooled, the organic layer separated, dried over magnesium sulfate and the solvent evaporated in vacuo to yield a yellow oil. (Figure1).

The ligand was characterized by ¹H, ¹³C {¹H} NMR, mass spectrometry and elemental analysis. Yield: 12.55g, (0.056 mol, 79.94 %). This ligand has been observed as an extractant subduing the above-mentioned shortcomings of other extractants. It has low aqueous solubility, complete miscibility with all the commonly employed organic diluents and is reasonably resistant to hydrolysis. Also the new extractant is free of any objectionable odor and offers a quicker phase separation. The product was characterized using ¹H, ¹³C {¹H} NMR, mass spectrometry and elemental analysis (28).

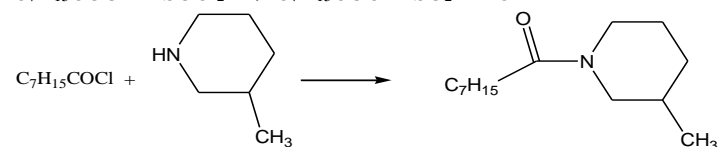
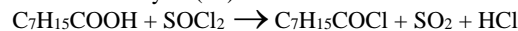


Figure1: Synthesis of N-octanoyl-3-methylpiperidine

Room temperature ¹H and ¹³C {¹H} spectra show the presence of both isomers, and a partial assignment can be made using ¹H COSY and ¹³C-¹H correlations experiments(28).

3.3.2. Regents and solutions

N-octanoyl-3-methylpiperidine is not commercially available and was synthesized as shown above. Odorless kerosene (non-aromatic) was obtained from Misr Petroleum Company, Egypt. Stock solutions of neodymium were prepared from its oxide (Nd₂O₃) Fluka in concentrated sulfuric acid and diluted with distilled water. All other chemicals were Prolabo products and were used as received.

3.3.3. Instrumentations and analytical procedure

NMR spectra of N-octanoyl-3-methylpiperidine were recorded by the author using either a Bruker ARX 250 spectrometer or a Bruker DPX 300 spectrometer. Microanalyses were obtained at the University of Leeds Microanalytical Service. Infra-Red spectra were recorded as KBr discs or nujol mull by the author on a Didac FTIR spectrometer in the Department of Chemistry Inorganic Teaching Laboratory. U(VI) was determined in the pregnant leach solution and the crude uranium concentrate using the colorimetric determination spectrometer using Arsenazo III. The other lanthanides concentration was determined by A ICP-OES optical emission spectrometry. All measurements were carried out at laboratory temperature.

3.3.4. Extraction procedure

Batch experiments were carried out by equilibrating equal volumes of aqueous phase 10ml and organic phase for 10 minutes under shaking in a magnetic stirring. After phase separation, known aliquots of the aqueous phase were sampled for analysis. The distribution coefficient (D) was measured as the ratio between the concentration of Nd(III) ions in the organic phase to the aqueous phase.

3.3.5. Back-extraction studies

For the back-extraction studies the organic phase was loaded with a certain amounts of Nd(III). A certain volume of the loaded organic

solution was mixed with equal volume of aqueous solution containing the strippant using magnetic shaker for 5 minutes.

4. Results and discussion

4.1. Factors affecting the dissolution of lanthanides from radioactive pegmatites ore

4.1.1. Effect of different acid on U (VI) and lanthanides dissolution

All mineral acid can dissolve U (VI) and the lanthanides element under reasonable conditions with different efficiencies. The influence of different kind of acid has concentration significant effect of U (VI) and lanthanides leaching and was carried out using H₂SO₄, HCl, HNO₃, and HClO₄. The S/L ratio was maintained at 1:10, mixing time 4 hrs, at room temperature, the acid concentration are 3M. The obtained results are graphically represented in Figure (2). From the obtained results, it is clear that all mineral acid can dissolve the U (VI) and lanthanides from the studied sample with different efficiency, HCl was the best acid gives highest leaching efficiency for U (VI) and lanthanides. But according to the corrosion problem due to chlorine anion, we chose H₂SO₄ for the cheapest availability and less industrially problematic.

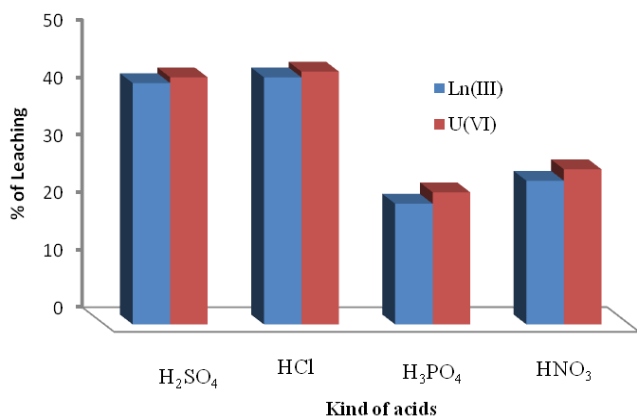


Figure 2: Effect of different acids on U(VI) and lanthanides leaching efficiency

4.1.2. Different concentration of H₂SO₄

It is clear that H₂SO₄ is the most suitable acid for leaching U(VI) and lanthanides, different concentration of H₂SO₄ and their effect on the leaching process were studied. We carried out the experiments having concentration ranging from 1-6M was added to samples at S/L phase ratio 1:10, contact time 2hr and constant temperature 500C. The results are presented in Figure (3), it was noticed that the concentration of U(VI) and lanthanide dissolution was gradually increase from 1-3M and decreased after that, 3M H₂SO₄ was the best concentration for leaching U(VI) and lanthanides. The lowest efficiencies after 3M probably due to the formation of unleachable complex species.

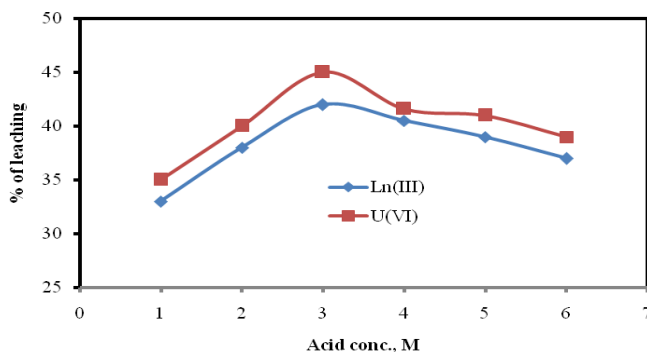


Figure 3: Effect of different H₂SO₄ concentration on U(VI) and lanthanides leaching

4.1.3. Effect of different mixing time

Contact time required for maximum leaching efficiency of U(VI) and lanthanides was studied, in which the leaching of these elements using 3M H₂SO₄ was investigated at contact time interval from 0.5-10 hr. The other factors were kept constant at 3M H₂SO₄, S/L phase ration 1:10 at room temperature. The obtained results are shown in Figure (4). From the experimental data it was observed that the concentration of U (VI) and lanthanides was gradually increased from 0.5 hr to 4 hr, after that it was decreased, excess time probably resulted in slight increase, consequently, 4hr equilibration time was chosen the most effective for U (VI) and lanthanides leaching.

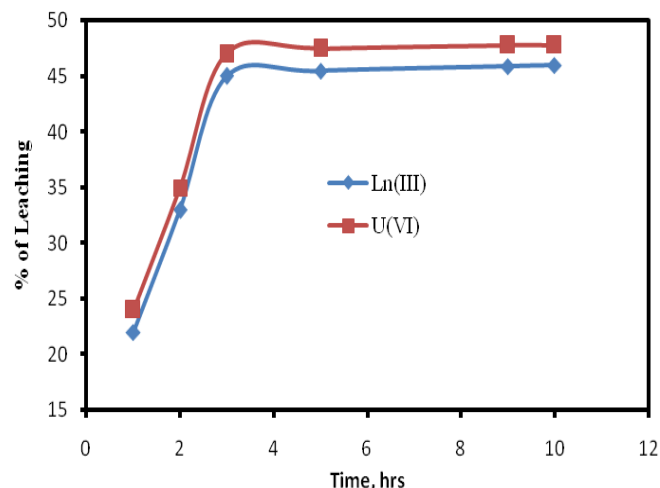


Figure 4: Effect of different equilibration time on U(VI) and lanthanides leaching

4.1.4. Effect of temperature

Effect of temperature on U (VI) and lanthanides leaching efficiency from Abu Furad radioactive pegmatite was studied in the range from 250C to 1000C. All other parameters of 3M H₂SO₄, S/L ratio 1:20 and 3hr of equilibration time were constant. The obtained results of leaching data were illustrated in Figure (5). From these results, it is clear that U (VI) and lanthanides leaching efficiency increased from 500C to 900C, but the increase of leaching efficiency after 900C was very slightly and for economic reason we chose this temperature (900C) as optimum condition. Also, increasing temperature enhances solubility of the undesirable impurities.

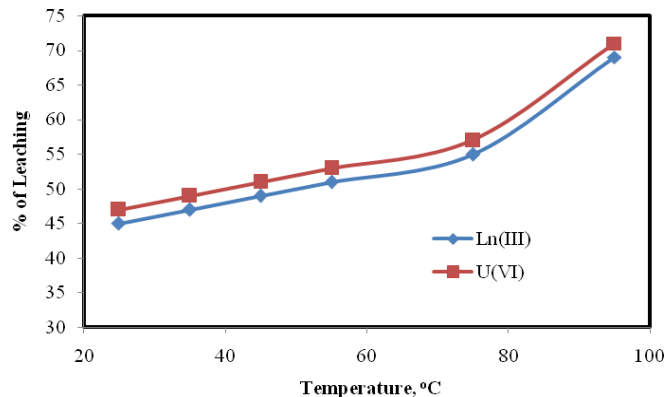


Figure 5: Effect of different temperature on U (VI) and lanthanides leaching

4.1.5. Effect of solid/liquid phase ratio

In this experiment, the effect of leach ore to acid phase ratio for the leaching of U(VI) and lanthanides was investigated with different ratios from 1:5 to 1:40 (gm/ml) with fixing the following conditions -60 mesh size, 3M H₂SO₄, 4 h of contact time at 90°C. The obtained data are graphically represented in Figure (6). From the obtained results, it was noticed that, the concentration of U (VI) and lanthanides was increased from 1:5 to 1:20 and decreased from 1:20 to 1:40, so it was clear that 1:20 S/L phase ratio was chosen the best and suitable for the leaching of lanthanides. S/L phase ratio gives highest leaching efficiency, probably large volume was not recommended due to the analysis and the following solvent extraction experiments.

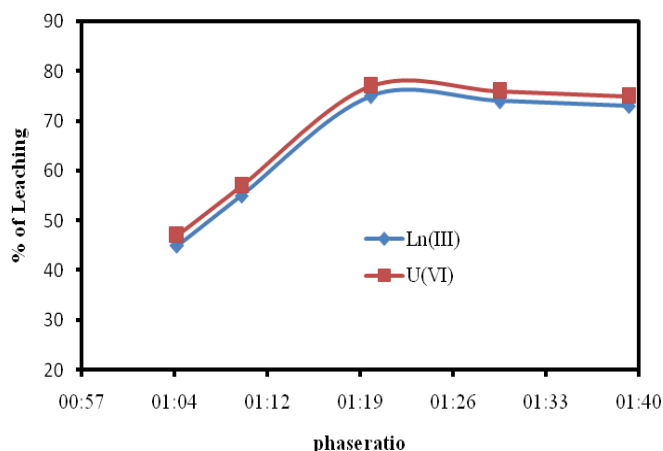


Figure 6: Effect of solid/liquid phase ratio on U (VI) and lanthanides leaching

4.1.6. Preparation of pregnant stock solution

U(VI) and lanthanides stock solution was prepared according to the nature and mineralogy of Abu Furad mineralized studied sample, the acidic agitation leaching technique was chosen, a technological sample 50gm was prepared for the purification of U(VI) and Nd(III) under the following conditions: 3M H₂SO₄ concentration, 1:20 (g/ml) solid: liquid ratio, -60 mesh size, agitation for 4h and at 90 °C temperature, using 20% NaOH the iron is precipitated and nearly completely removed from the stock solution at pH 3-3.5. After the iron separation and filtration of iron hydroxide.

4.1.6.1. Precipitation of U (VI) as yellow cake.

It is customary to regard the form of the resulting U (VI) in solution as being UO₂SO₄. In practice, however high proportions of the anionic complexes UO₂(SO₄)₂²⁻ and UO₂(SO₄)₃⁴⁻ are also present. As a matter of fact the dissolved U(VI) occurs in all three forms simultaneously, the abundance of which being controlled by complex equilibria involving the concentration of H⁺, SO₄²⁻ and HSO₄⁻ ions besides that of the various forms of dissolved U(VI). These complex anionic forms take a major role in the subsequent purification and recovery of U (VI) from the pregnant acid liquor (29).

Preliminary experiments showed that cold precipitation results in very fine highly fluctuated precipitate. With a very slow settling rate and sluggish filtration. Precipitation was affected therefore at room temperature with continuous mechanical agitation at pH 6. This pH was obtained by gradually addition of 20% NaOH from a burette. Neutralization of the solution was continued where granular deep yellow uranium compound (yellow cake), precipitated and settled rapidly. The precipitate was filtered then washed several times with distilled water and then dried at 110°C. The U (VI) concentration in the yellow cake was found to be 61.42% U₃O₈ as determined by spectrophotometry.

4.1.6.2. Precipitation of lanthanides

Finally using 20% NaOH the lanthanides is precipitated from the pregnant solution by adjusting pH between 8-9 as lanthanide hydroxide after filtration of lanthanide hydroxide was dried and dissolved in 0.2 M H₂SO₄

which consider the starting point for individual separation of Nd(III) from total lanthanides using N-octanoyl-3 methylpiperidine dissolved in kerosene. The chemical composition of the obtained pregnant sulfate liquor was shown in Table (3).

Elements	Conc.,%	Elements	Conc.,%
Al ₂ O ₃	5.33	Pr ₂ O ₃	9.24
SiO ₂	19.19	Nd ₂ O ₃	19.00
K	0.57	MnO ₂	2.19
Ca	2.70	Fe ₂ O ₃	4.53
La ₂ O ₃	17.24		
Ce ₂ O ₃	19.81		

Table 3: Chemical composition of the leaching solution after dissolving crud lanthanides in 0.2M sulfuric acid.

4.2. Effect of different factors influencing the liquid-liquid extraction of ND (III) from sulfate leach liquor of radioactive pegmatites ore using new ligand.

4.2.1. Effect of different concentrations of N-octanoyl-3-methylpiperidine

Firstly the solvent was equilibrated by contacting it with sulfuric acid solution for 10 minutes. To 10 ml of the sulfate liquor of neodymium, 10 ml of a solution of N-octanoyl-3-methylpiperidine in carbon tetrachloride was added with concentrations ranging from 0.01 to 0.15M. The solution was then shaken for 10 minutes at room temperature to attain equilibrium state. It is clear from Fig. 7 that the distribution coefficient increases from 1.5 to 8.7 by increasing the extractant from 0.01M to 0.1M in carbon tetrachloride, after it remains constant. Accordingly 0.1M solution of N-octanoyl-3-methylpiperidine in CCl₄ is considered as an optimum value. A plot of log D versus log [OMPED] gives a linear correlation with a slope 2 indicating the requirement of two moles of ligand for each mole of ND (III).

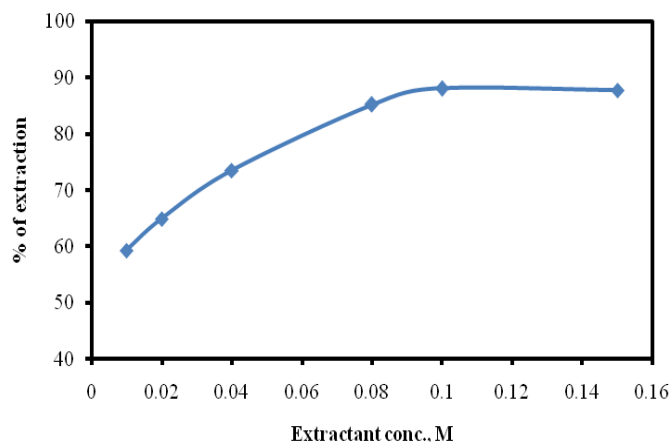


Figure 7: Effect of different ligand concentration on neodymium extraction

4.2.2. Effect of equilibration time on the distribution coefficient of neodymium

The effect of contact time on the attainment of an equilibrium state was studied at intervals between 1– 50 minutes, while the other factors were kept as previously mentioned. The results obtained are shown in Fig.8. It

is obvious that the extraction is fast and contact time of 10 minutes is quite adequate for efficient neodymium distribution coefficient and by increasing the shaking time the distribution coefficient is slightly decreased, and this behavior probably due to the entrainment of some organic phase droplets into aqueous phase, which causes decrease in the extractive power of extractant.

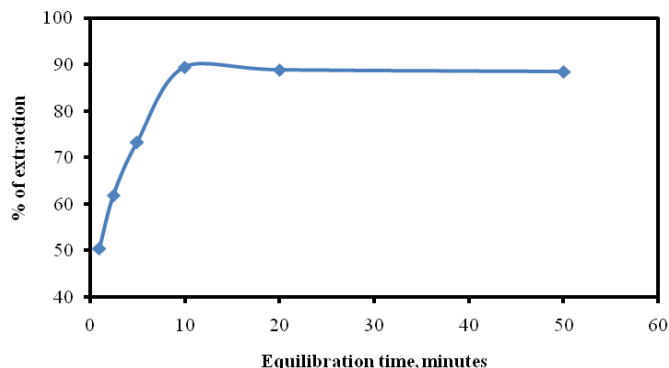


Figure 8: Effect of contact time on the percent extraction of neodymium.

4.2.3. Effect of pH on the distribution coefficient of neodymium.

The distribution coefficient has been studied by varying the pH values of the sulfate liquor from 0.2 to 2.2, using either 1M sulfuric acid or 2M sodium hydroxide. Other factors were fixed at 1:1 (v/v) organic to aqueous phase ratio, 0.1M N-octanoyl-3-methylpiperidine dissolved in CCl₄, contact time 10 minutes and the experiments were carried out at room temperature. The results obtained are shown in Fig. 9. It is clear that the pH 1.7 of the sulfate liquor can be taken as an optimum pH value. Fortunately, that is the same pH of the parent sulfate liquor under examination. The reason for the decrease in neodymium distribution coefficient may be result from the formation of non-extractable praseodymium species as a result of complication with components of aqueous phase. This occurs, in sulfate media due to equilibria between SO₄²⁻, HSO₄⁻, and H₂SO₄ resulting in the formation of neodymium complexes with these anions. At higher pH of extraction, there is a possibility of hydrolysis of ion-pair complex. This indirectly promotes competing equilibria with the formation of ion-pair complex, resulting in a decrease in extractability with an increase in pH (30, 31).

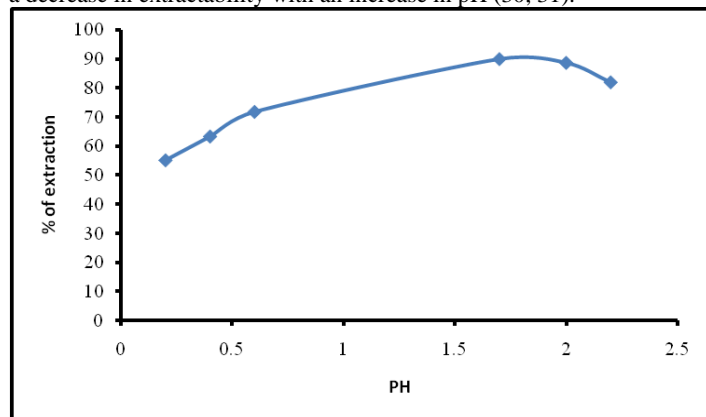


Figure 9: Effect of equilibrium pH on the distribution coefficient of neodymium.

4.2.4. Effect of diluents on the distribution coefficient of neodymium

The distribution coefficient of neodymium has been studied using different organic diluents, namely chloroform, carbon tetrachloride, toluene, benzene, o-xylene, and kerosene. The other factors were fixed at the values at which maximum distribution coefficient occurred i.e. 1:1 (v/v) organic to aqueous phase ratio, room temperature, 0.1M N-octanoyl-

3-methylpiperidine in all diluents, and a contact time of 10 minutes. Results are presented in Table 4. Short equilibrium time and good phase separation was achieved when aliphatic diluents were used compared to aromatic diluents. Considering several factors like cost, environmental and safety aspects and maximum neodymium (III) distribution coefficient, kerosene was preferred as the diluent for further neodymium (III) extraction studies.

Diluents	Dielectric constant	Extraction percent, [%]
Benzene	2.30	86.57
Toluene	2.23	85.13
O-xylene	2.28	84.93
Chloroform	5.10	87.82
Carbon tetrachloride	2.23	88.50
Kerosene	2.00	89.32

0.1M N-octanoyl-3-methylpiperidine; Phase ratio (Org:Aq) 1:1; time 10min; room temperature.

Table (4): Extraction of Nd (III) by N-octanoyl-3-methylpiperidine as a function of diluents

4.2.5. Effect of aqueous: organic phase ratio

The aqueous: organic phase ratio has a significant effect on the distribution coefficient and entrainment. This effect was investigated by changing the aqueous: organic phase ratio from 1:1 to 1:4. The results presented in Table (5) it is obvious that a phase ratio of 1:1 gives the best extraction of neodymium. At this phase ratio the rate of coalescence and re-dispersion of the dispersed phase is also enhanced.

Phase ratio (Aq:Org)	Distribution coefficient	Extraction percent
1:1	8.70	89.32
2:1	7.91	82
3:1	4.33	69
4:1	1.56	62

Table (5): Effect of aqueous: organic phase ratio on the extraction process

4.2.6. Effect of temperature

The extraction of a metal complex into an organic phase involves large changes in enthalpy (solvation processes) and in entropy (solvent orientation and restructuring), leading to considerable temperature effects. To study the effect of temperature on extraction of neodymium, experiments were carried in the range 298–333°K. The Results obtained showed that the extraction of neodymium decreased from 89.12% at 298°K to 81.9% at 333°K. This behavior is agrees with that reported by previous investigators (32).

Fig. 10 plots Log D versus 1000/T, K⁻¹ which gives a straight line for The extraction of neodymium by N-octanoyl-3-methylpiperidine from 0.3 M H₂SO₄

Solution. The ΔH value for neodymium (III) was -27 kJ/mol — as calculated

from the slope using the Van't Hoff equation (Eq. (1)).

$$\text{Log } D = -\Delta H/2.303RT + C \quad (1)$$

Where D is the distribution coefficient, ΔH the enthalpy change for the Extraction reaction, R is the universal gas constant (8.314 J/mol k) and C is a constant for the system. This value of ΔH was used to obtain the Corresponding free energy ($\Delta G = -9.4$ kJ/mol) and entropy ($\Delta S = 47$ J/mol

K) at 298°K using Eqs. (2) and (3), respectively:

$$\Delta G = -2.303RT \log D \quad (2)$$

$$\Delta G = \Delta H - T\Delta S \quad (3)$$

The negative value of ΔH indicates that the extraction of neodymium this system is an exothermic process and the reaction becomes more favourable at room temperatures.

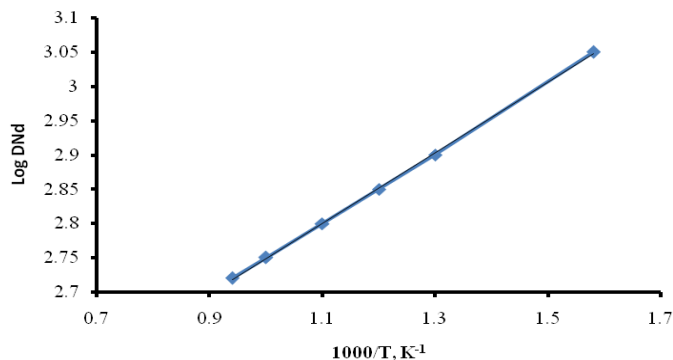


Figure 10: Plot of DNd against 1000/T, K-1 for extraction of neodymium.

4.2.7. Extraction isotherm

The number of the theoretical stages was calculated by the McCabe-Thiele diagram which was constructed by standard procedure and is given in Fig. 11. Equal volumes of the organic phase (0.1M N-octanoyl-3-methylpiperidine in kerosene) and aqueous phase were contacted for five minutes until equilibrium was obtained the phases were allowed to separate and the aqueous phase was removed and analyzed. Fresh aqueous solution was added to the organic phase to give the same phase ratio as that originally used and the procedure repeated. Care must be taken to keep the same pH value throughout the series of shake-outs. In constructing this diagram, the data was used to obtain extraction isotherm curve by plotting neodymium concentration in the organic phase against its concentration in the aqueous phase. A vertical line is then drawn from the concentration of neodymium in the feed solution on the x-axis. The operating line is next inserted, the slope of which is equal to the phase ratio. From Fig. 11 we can see neodymium extraction under these conditions needs two extraction stages for nearly complete recovery.

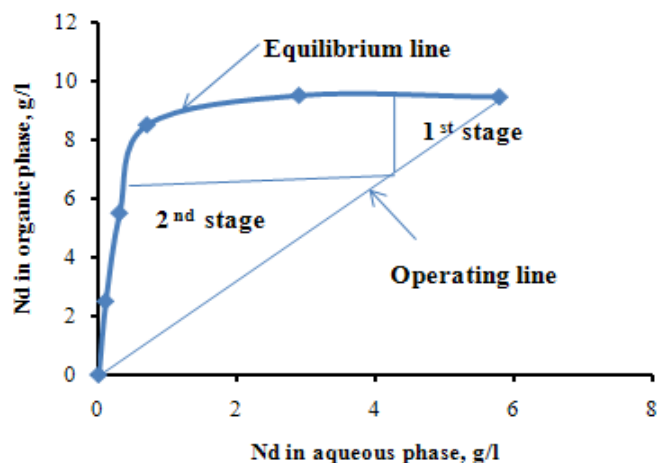


Figure 11: McCabe-Thiele diagram for neodymium extraction

4.2.8. Effect of stripping agents

Stripping is the reverse of the extraction, so it should be promoted by these factors that affect extraction negatively. Neodymium (III) stripping from loaded organic solvent, were investigated using various stripping agents,

such as HCl, H₂SO₄ and HNO₃, in the range 0.5– 6M and O: A ratio of 1:1. The results are presented in Fig.12. From the results, it is clear that 4M HNO₃ is the most effective acid for the quantitative stripping of Nd (III). Variation of the stripping period showed that 5 minutes equilibration time were sufficient for quantitative recovery of neodymium (III) from organic phase. Neodymium ions can be stripped from neodymium-loaded N-octanoyl-3-methylpiperidine solution into 4M HNO₃, where 10 % oxalic acid was added gradually to the obtained strip liquor rich in Nd(III), and was left for 24 hours where neodymium oxalates were obtained.

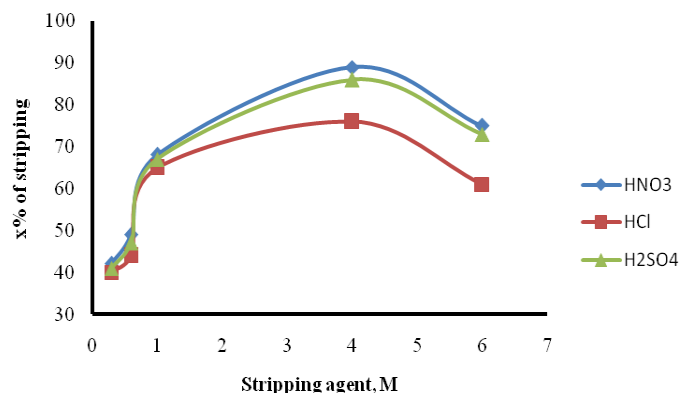


Figure 12: Effect of different stripping agents on stripping efficiency

4.2.9. Stability and regeneration capacity of N-octanoyl-3-methylpiperidine

Experiments were conducted to check the chemical stability of 0.1M ligand dissolved in kerosene towards long-term mixing with 0.3M H₂SO₄. The extraction of Nd(III) was periodically tested after 3 days. A negligible variation nearly 2% in the distribution coefficient and percent extraction of Nd(III) ion was observed even after a contact of the extractant with the acid for 30 days.

4.3. Solvent extraction/stripping of Nd(III) followed by neodymium precipitation. The obtained pregnant leach solution is contacted with 0.1M N-octanoyl-3-methylpiperidine dissolved in kerosene two stages followed by scrubbing with distilled water finally stripping in two stages using 4M HNO₃ at an aqueous: organic phase ratio of 1:1 after that the strip liquor is studying for Nd(III) precipitation. Overall recovery of neodymium was 89-90%. Neodymium was finally precipitated from the nitrate solution as oxalate (using 10% oxalic acid) and calcined to oxide. The final product contained 85.5% Nd₂O₃ together with a little amounts of 3.31% Pr₂O₃, 4.81% La₂O₃ and 5.95% Ce₂O₃.

5. Conclusions

Solvent extraction studies of neodymium from sulfuric acid solutions by N-octanoyl-3-methylpiperidine which can be easily synthesized and showed that this extractant is a promising one in the separation and purification of neodymium from rare earth elements as summarized in the following points:

1. Neodymium (III) extraction efficiency was achieved with 0.1M N-octanoyl-3-methylpiperidine in kerosene, at an organic: aqueous (O/A) phase ratio of 1:1, equilibrium pH of 1.7 and for 10 minutes contact time.
2. Kerosene is showed good results as diluent for N-octanoyl-3-methylpiperidine comparing with the other diluents used.
3. Neodymium (III) stripping is achieved using 4M HNO₃ at an O: A ratio of 1:1 and 5 minutes contact time is recommended for quantitative stripping efficiency.
4. As an application of the proposed method, a final neodymium oxide of ca. 85.55% is obtained from a neodymium rich rare earth concentrate of 19.00% Nd₂O₃ produced from Gabal Abu Farad area which is located

40 Km south west of Safaga city on the Red sea coast concentrate, Nuclear Materials Authority, Egypt.

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