SEPARATION OF TANTALUM AND NIOBIUM FROM THE TREATED SULFATE LIQUOR USING ISODECANOL

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Abstract

A technological sample of polymetallic sedimentary rocks collected from southwestern Sinai (2.61% RE_2O_3, 1.14% Ta_2O_5 and 2.16% Nb_2O_5) is subjected to processing via sulfuric acid agitation leaching under different conditions. The produced sulfate liquor is chemically treated involving precipitation of REEs as oxalates. The treated sulfate solution was obtained and used for recovery of Nb and Ta using solvent extraction as isodecanol. The relevant factors for the extraction and stripping processes of Nb and Ta have been optimized. Highly pure products of Nb and Ta have properly been prepared and analyzed using ICP-MS.

Keywords: Niobium; Tantalum; Sulfate liquor; Isodecanol; Solvent extraction.

Introduction

The application of niobium and tantalum over the past two decades is increasing as a result of their importance in the production of modern industrial materials, high tech consumer products and strategic energy technologies such as super alloys, electronic devices, solar, wind and nuclear equipment, in conjunction with other elements such as tellurium, indium, silver, dysprosium, neodymium and molybdenum **(He et al., 1998) (Hayes and Burge, 2003) (Cunningham, 2000) (Moss et al., 2011) (Lowder, 2012) (Pell and Hora, 1990)**. Nb and Ta are presented from a variety of minerals and concentrates. The major source of these two metals is the columbite-tantalite mineral, also called coltan, $(Fe,Mn)(Nb,Ta)_2O_6$ which contains (5 – 30 % Ta_2O_5 in columbite and 42 – 84 % Ta_2O_5 in tantalite) and (55 – 78 % Nb_2O_5 in columbite and 2 – 40 % Nb_2O_5 in tantalite). Ta and Nb exist in other minerals (more than 150) as complex oxides and hydroxides with the exception of the borate mineral behierite $(Ta,Nb)(BO_4)$ and the only known non-oxide mineral containing tantalum carbide TaC **(Nete et al., 2016)**.These two metals (Nb & Ta) are presented in the present study for the polymetallic sedimentary rocks of Um Tomyem area, southwestern Sinai.

Extraction of tantalum and niobium from their ores has several studies which investigated the development of the successful dissolution of Ta/Nb sources followed by their separation and purification. The former included fusion digestion using different fluxes such as $KHSO_4$, $Na_2S_2O_7$, KHF_2, Na_2CO_3, borax and alkali hydroxides **(El-Hazek et al., 2009) (Borchers and Korinek, 1981) (Gupta and Suri, 1994) (Eckert, 1995) (Wang et al., 2009) (El Hussaini and Mahdy, 1998) (Zhou et al., 2005)** as well as acid leaching techniques such as H_2SO_4, HNO_3, HF and HCl **(Ayanda and Adekola, 2011) (El Hussaini and Mahdy, 2002) (Htwe and Lwin, 2008) (Irfan and Ahmad, 2014) (Compderros and Marchese, 2001)**. The latter included several procedures namely, precipitation, distillation, solvent extraction and ion exchange resin technique. Solvent extraction can be considered the most common separation method and has a number of important advantages such as the reduction of the number of production steps and cleaner tantalum and niobium products. Several extraction systems have been studied and some of them have proved to be highly successful to the extent that they have already been adopted for industrial productions. The studied exctractants include MIBK, TBP, cyclohexanone, DEHPA, DIPK, Alamine 336 and octanol **(Compderros and Marchese, 2001) (IAEA, 2003) (Ritcey and Ashbrook, 1979) (Zhu and Cheng, 2011) (El Hussaini, 2009) (El-Hazek et al., 2012) (Agulyansky et al., 2004) (Mayorov and Nikolaev, 2002) (Maiorov et al., 2001) (El Hussaini and Mahdy, 2001) (El Hussaini and Rice, 2004)**.

The present work is concerned with the chemical processing of polymetallic sedimentary rocks collected from Um Tomyem area, southwestern Sinai for the recovery of Nb and Ta metal values. For this purpose, the working ore sample is subjected to complete chemical analysis and processing via sulfuric acid agitation leaching. The relevant factors of the acid leaching step have first been studied and after water washing of the residue, both the REEs, Nb and Ta values have been dissolved. From the latter, the REEs have been precipitated as their oxalates while recovery of the Ta and Nb value left behind has properly been studied via solvent extraction technique using isodecanol.

Experimental
Material characterization

The collected technological sample representing the polymetallic sedimentary rocks of Um Tomyem area, southwestern Sinai has first been subjected to complete chemical analysis of the major oxides as well as for some interesting metal values. The major oxides were analyzed using the conventional wet procedure of Shapiro and Brannock (1962). For Ca and Mg, a titrimetic method with EDTA and Eriochrome black T as indicator was

used. The spectrometric analysis was used for SiO_2, Al_2O_3, P_2O_5 and Fe_2O_3 (total) while for Na_2O and K_2O, flame photometry was used. Weighed sample portions were used to estimate the loss of ignition at various temperature degrees. The total loss of ignition (obtained at 1000 °C) is corresponding to humidity, combined water, CO_2 as well as possible organic matter.

On the other hand, analysis of the interesting metal values; viz the REEs (in terms of La) has spectrophotometrically been determined using Arsenazo-III as the chromogenic reagent (Merczenko and Balcerzek, 2000) while Nb, Ta and Ti were determined by ICP-MS Egilant technologist 7700 serial, Japan in the labs of the Chemical Warefare.

Experimental Procedures
Sulfuric acid leaching
Each leaching experiment has been performed by agitation a weighed amount of the working Um Tomyem ore material (10 g) with H_2SO_4 acid of different concentrations with different solid/liquid ratios for different periods of time at different temperatures. In these experiments, a hot plate with magnetic stirrer was used and precautions were taken for avoid evaporation. The obtained slurry was then cooled, filtered, washed with distilled water and made up to volume for REEs, Nb and Ta analysis.

Solvent extraction of Nb and Ta
To study the recovery of both Nb and Ta, a 500 g sample portion of the working Um Tomyem ore material was subjected to sulfuric acid agitation leaching under the optimum conditions of acid leaching followed by cooling and proper washing of the slurry. From the obtained sulfate leach liquor, the REEs were relatively recovered by their direct precipitation as oxalates while Nb and Ta were left behind in the solution.

Nb and Ta were extracted from the treated sulfate liquor (free of REEs) using the organic solvent isodecanol. The solvent was diluted in kerosene to prepare different concentrations. In each extraction experiment, the organic phase was conducted with the aqueous phase in separating funnel where they shaked together for the desired time. The two phases were then allowed to settle, separate and an aliquot sample of the aqueous phase was analyzed for its remaining metal content in order to calculate the extraction efficiency.

The relevant solvent extraction factors; namely solvent concentration, the contact time and the pH of the leach liquor as well as aqueous/ organic ratio (A/O) were studied. On the other hand, a loaded solvent sample was prepared for studying the stripping behavior of the extracted metal value using the same procedures as that used for extraction.

Control analysis

For the control analysis of all the performed experiments, REEs, Nb and Ta have been analyzed in all the working stream solutions via the above mentioned procedures; viz spectrophotometric and ICP-MS respectively. On the other hand, the two highly pure products of both Nb and Ta were analyzed in the labs of the Chemical Warefare using ICP-MS.

Results and discussion

Material characteristics

Chemical composition of Um Tomyem ore material shown in **Table (1)** indicated that the ore material is mainly composed of SiO_2 beside Al_2O_3 and Fe_2O_3 that attained 3.5% and 8.8% respectively, as well as high K_2O content (3.9%). In the meantime, a total loss of ignition of about 0.86 was obtained at 1000 °C. On the other hand, analysis of the economic trace metals involved RE_2O_3 2.61%, Nb_2O_5 2.16% beside Ta_2O_5 1.14%.

Table 1. Chemical composition of Um Tomyem ore material.

Major constituent	Wt.,%	trace constituent	Wt.,%
SiO_2	70.29	RE_2O_3*	2.61
TiO_2	2.15	Nb_2O_5	2.16
Al_2O_3	3.50	Ta_2O_5	1.14
Fe_2O_3	8.80	U_3O_8	0.02
CaO	1.44	ThO_2	0.04
MgO	0.90	V_2O_5	0.01
MnO	0.04	BeO	0.02
Na_2O	2.03		
K_2O	3.90		
P_2O_5	0.08		
L.O.I	0.86		
Total		99.99	

* RE_2O_3 measured as light rare earths (lanthanum).

Leaching studies

Acid agitation leaching of Nb, Ta and REEs

Effect of H_2SO_4 acid concentration

To study the effect of H_2SO_4 conc. upon leaching efficiency of the studied ore material, a set of leaching experiments was performed using different concentrations of the H_2SO_4 acid ranging from 100 to 400 g/l. The other leaching conditions were fixed at 100 °C for 1 h and S/L ratio of 1/1. From the obtained data in **Table (2)**, it is clear that the leaching efficiencies of REEs, Nb and Ta increased from 22.7 to 36.8 %, from 12.2 to 23.4 % and

from 13.9 to 25.8 % respectively by increasing the acid concentration from 100 to 300 g/l. Further, increase in H_2SO_4 conc. at 400 g/l has not improved the leaching efficiencies of REEs, Nb and Ta. From hence, the best acid concentration for leaching metal values is 300 g/l. The dissolution of Nb and Ta is low due to that elements are refractory (strong crystal lattice).

Table 2. Effect of H_2SO_4 conc. upon leaching efficiency of the studied metal values of Um Tomyem ore material.

Conc.H_2SO_4, (g/l)	Leaching efficiency, %		
	REEs	Nb	Ta
100	22.7	12.2	13.9
200	28.9	18.9	17.9
300	36.8	23.4	25.8
400	39.7	23.8	26.1

Effect of Solid/Liquid ratio (S/L)
The effect of S/L ratio upon the leaching of the interesting metal values of the ore material under consideration was studied from the ratio 1/1 to the ratio 1/2.5 when applying H_2SO_4 conc. of 300 g/l, 100 °C as leaching temperature and 1 h as leaching time. From the obtained data in **Table (3)**, it is clear that the leaching efficiencies of REE, Nb and Ta increased from 44.7 to 56.8 %, from 23.8 to 33.4 % and from 26.1 to 32.8 % respectively by increasing the S/L ratio from 1/1 to 1/2. Further, increase in the S/L ratio at 1/2.5 has not improved the leaching efficiencies of REEs, Nb and Ta. From hence, the best S/L ratio for leaching metal values is 1/2. It is obvious that 300 g/l H_2SO_4 cannot enough breakdown the Nb-Ta mineral lattice even at 1/2.5 S/L ratio.

Table 3. Effect of S/L ratio upon leaching efficiency of the studied metal values of Um Tomyem ore material.

S/L ratio	Leaching efficiency, %		
	REEs	Nb	Ta
1/1	44.7	23.8	26.1
1/1.5	48.9	28.9	28.7
1/2	56.8	33.4	32.8
1/2.5	57.7	33.9	33.1

Effect of agitation time
To study the effect of agitation time upon the leaching efficiency of the interested metals, a set of leaching experiments has been performed at different times ranging from 1 to 4 h. The other leaching conditions were fixed at H_2SO_4 conc. of 300 g/l, 1/2 S/L ratio at 100 °C. From the obtained

results in **Table (4)**, it is obvious that the refractory nature of the study ore material required long leaching periods of time under the applied leaching conditions, where the leaching efficiencies of REEs, Nb and Ta increased from 56.8 to 61.3 %, from 33.4 to 49.9 % and from 32.8 to 45.8 % respectively by increasing the time from 1 to 3 h. Further, increase in the time at 4 h has not improved the leaching efficiencies of REEs, Nb and Ta. So, the leaching time of 3 h is the optimum time for leaching of interesting metal values.

Table 4. Effect of agitation time upon leaching efficiency of the studied metal values of Um Tomyem ore material.

Time, h.	Leaching efficiency, %		
	REEs	**Nb**	**Ta**
1	56.8	33.4	32.8
2	57.6	38.8	39.9
3	61.3	49.9	45.8
4	61.8	50.2	46.2

3.2.1.4. Effect of leaching temperature

The effect of leaching temperature upon the leaching efficiencies of the interested metal values was investigated in the range from 100 up to 300 °C under the fixed experimental conditions of 300 g/l H_2SO_4 conc., S/L ratio of 1/2 and leaching time of 3 h. From the obtained data in **Table (5)**, it is clear that the refractory nature of the polymetallic ore material has required high temp. up to 300 °C to be broken, where the REEs, Nb and Ta leaching efficiencies increased from 61.3 to 99.9 %, from 49.9 to 98.2 % and from 45.8 to 99.2 % respectively with increasing the temperature from 100 to 300 °C under the applied leaching conditions. So, the leaching temperature of 300 °C is the optimum temperature for leaching of interesting metal values.

Table 5. Effect of leaching temperature upon leaching efficiency of the studied metal values of Um Tomyem ore material.

Temp., °C.	Leaching efficiency, %		
	REEs	**Nb**	**Ta**
100	61.3	49.9	45.8
150	74.5	59.8	61.2
200	88.6	73.5	77.7
250	91.2	88.4	90.2
300	99.9	98.2	99.2

From the above studied acid agitation leaching factors of Um Tomyem ore material, it can be concluded that the optimum conditions for almost complete dissolution the interesting metal values can be summarized as follows:

H_2SO_4 acid concentration   :   300 g/l
S/L ratio   :   1/2
Leaching time   :   3 h.
Leaching temperature   :   300 °C

Preparation of the sulfate leach liquor

The sulfate solution of polymetallic ore sample of Um Tomyem area, southwestern Sinai was prepared by applying the above optimum factors followed by filtration and washing the residue with distillated water to attain up to volume 2 L. Analysis of the latter is shown in **Table (6)**. From the latter, the produced pregnant solution pH is 1.8 and assays 8.5 g/l REEs, 5.32 g/l Nb and 2.74 g/l Ta with almost complete dissolution of Nb, Ta and REEs.

Table 6. Chemical composition of the prepared sulfate solution.

Constituent	Concentration, g/l
Fe_2O_3	6.24
Al_2O_3	3.34
RE_2O_3*	8.50
Nb_2O_5	5.32
Ta_2O_5	2.74
pH	1.8

Treatment of the sulfate leach liquor of the polymetallic ore sample

The prepared sulfate leach liquor is used to extract the interesting metal values Nb, Ta and REE. Thus, REE is recovered using direct precipitation technique. The latter carried out using 10 % oxalic acid. At pH 0.8, almost complete REEs precipitation occurred where their precipitation efficiencies attained 97.4 %. The REEs product can be calcined at 800 °C and analyzed using UV-Visible spectrophotometer which indicated a REE purity of 94.7 %. The produced solution free of REEs presented the treated sulfate liquor for separation of Nb and Ta.

Recovery of tantalum and niobium

Solvent isodecanol ($C_{10}H_{22}O$) was chosen for extracting both Nb and Ta from the treated sulfate solution because of its availability and low cost. Generally, the good solvating character of isodecanol may be due to its high dipole moment and the presence of electronegative O-H bond. The molecular formula of isodecanol is {CH_3 (CH_3) CH (CH_2)$_6$ CH_3 (OH)} and density = 0.841. Alcohols of C_8-C_{12} chain length could have particular interest as prospective extractants for LLE (Liquid-Liquid extraction) where 1-octanol provides efficiency comparable with both MIBK and TBP in LLE of tantalum

and niobium. Isodecanol main advantages are insoluble in water, sufficiently less volatile and less dangerous.

antalum and niobium extraction

In practice, the principal solvent extraction factors including solvent concentration in the organic phase, the optimum contact time, the pH of the leach liquor and the aqueous / organic ratio (A/O) were studied for each of the two interesting metal values namely; Nb and Ta.

Effect of isodecanol concentration

This effect was studied upon the Ta and Nb extraction efficiencies by contacting equal volumes of the treated sulfate solution with solvent of different concentrations ranging from 25 to 100 % at the fixed conditions of pH 0.9, contact time of 12 min. From the obtained data in **Table (7)**, it is noticed that extraction efficiency of Ta and Nb increased from 24.1 to 99.7 % and from 19.2 to 99.3 % when isodecanol concentration increased from 25 to 100 %, respectively.

Table 7. Effect of isodecanol concentration upon extraction efficiency of Ta and Nb.

Solvent Concentration, %	Extraction efficiency, %	
	Ta	**Nb**
25	24.1	19.2
50	46.3	56.7
75	76.6	80.1
100	99.7	99.3

Effect of pH of the treated sulfate liquor

To study the effect of pH of the treated sulfate solution upon Ta and Nb extraction efficiencies, a set of experiments was performed at different pH values of the treated sulfate liquor ranging from pH 2.1 to 0.9 at the fixed conditions of A/O 1:1, 100 % isodecanol & 12 min as contact time. From the shown results (**Table 8**), it is clear that extraction efficiency of Ta and Nb increased from 53.6 to 99.7 % and from 00.0 to 99.3 % when pH of the treated sulfate liquor decreased from 2.1 to 0.9, respectively.

Table 8. Effect of pH upon the extraction efficiency of Ta and Nb.

pH	Extraction efficiency, %	
	Ta	**Nb**
2.1	53.6	nil
1.8	98.4	19.2
1.5	99.1	45.2
1.2	99.4	74.3
0.9	99.7	99.3

Effect of contact time

To study the effect of contact time upon the extraction efficiency of Ta and Nb using isodecanol, a set of experiments has been performed at different contact times ranging from 4 to 12 min at the fixed conditions of A/O ratio 1:1, 100 % isodecanol concentration & pH value 0.9. The results are shown in **Table (9)**. From the latter, it is obvious that increasing the contact time between the leach liquor and the organic phase from 4 to 12 min, increase Ta and Nb extraction efficiencies from 27.5 to 99.7 % and from 13.6 to 99.3 %, respectively.

Table 9. Effect of contact time upon the extraction efficiency of Ta and Nb.

Time, min	Extraction efficiency, %	
	Ta	**Nb**
4	27.5	13.6
6	57.8	55.5
8	80.1	77.2
10	95.2	89.1
12	99.7	99.3

Construction of McCabe-Thiele diagram for Ta & Nb extraction

From the treated sulfate liquor, the effect of the aqueous / organic (v/v) phase ratio upon the extraction of Ta and Nb were investigated in the range of 3:1 down to 1:3 while fixing the other extraction factors at their determined optimum values (100 % isodecanol, 12 min as contact time at room temperature and the pH value of the aqueous phase of 0.9)**.** On the other hand, McCabe-Thiele diagram is constructed from the obtained equilibrium data of Ta and Nb extraction as shown in **Fig. 1 & 2**. From this diagram, the required theoretical number of extraction stages would be determined. According to the slope of the chosen operating line which was found to attain 3 (A/O), 3 theoretical stages would be required for almost complete extraction of Ta and Nb from the treated sulfate leach liquor (free of REEs).

Tantalum and niobium stripping

Tantalum and niobium stripping were carried out by shaking equal volumes of the loaded solvent with a suitable stripping solution for a certain time at room temperature. Factors influencing the stripping efficiency were studied. These include stripping agent type, concentration, stripping time and aqueous to organic phase ratio (A/O). The concentration of Ta and Nb in the loaded solvent was 2709 and 5310 ppm, respectively.
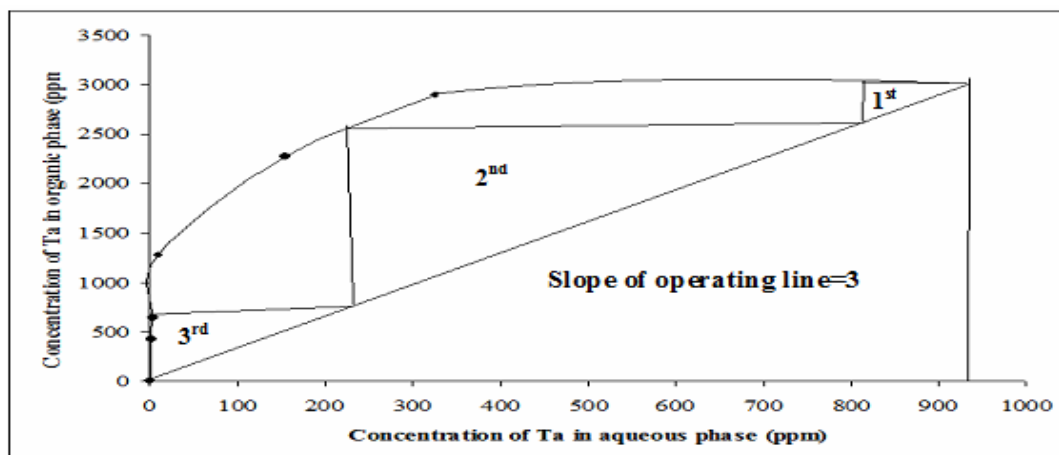
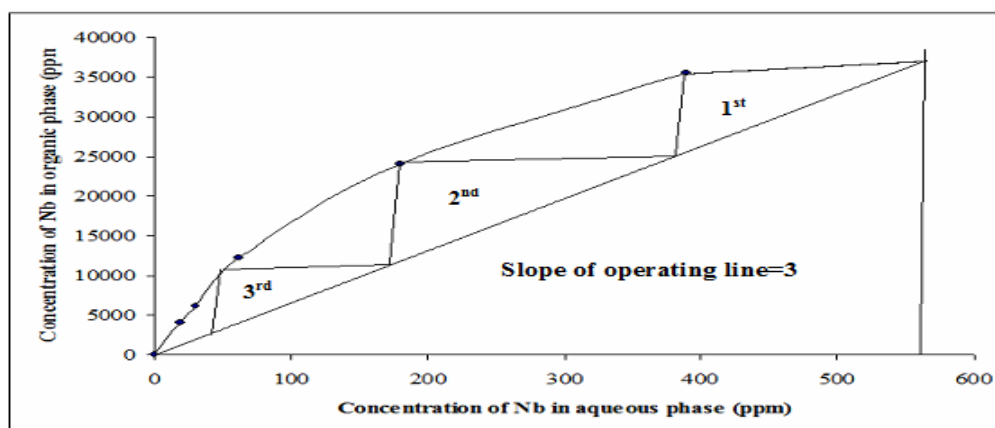Fig. 1. McCabe-Thiele diagram for Ta extraction.

Fig. 2. McCabe-Thiele diagram for Nb extraction.

To strip Ta and Nb from the loaded solvent, different stripping agents were studied. These included distilled water, mineral acids namely; H_2SO_4, HF and HNO_3 (0.3M). The stripping experiments were conducted at 3 min contact time and 1/1 A/O ratio as shown in **Table (10)**. The obtained results revealed that distilled water is the most efficient for stripping Ta from the loaded solvent while Nb remains in the organic phase. On the other hand, HNO_3 is the most efficient for Nb stripping from the loaded solvent.

Table 10. Effect of stripping agent type upon Ta and Nb stripping efficiencies.

Stripping agent	Stripping efficiency, %	
	Ta	**Nb**
Dist. H_2O	39.73	nil
0.3M H_2SO_4	10.95	16.70
0.3M HF	5.32	29.90
0.3M HNO_3	nil	49.70

Tantalum stripping
i) Effect of stripping time
The effect of stripping time upon Ta stripping efficiency was studied in the range from 3 to 9 min at the fixed conditions of A/O ratio of 1:1 using dist. water as stripping medium. The corresponding Ta stripping efficiencies were measured using ICP-MS and tabulated in **Table (11)**. From the obtained results, it was found that 9 min stripping time is sufficient to bring almost complete Ta content (99.34 %) in the strip solution while Nb remains in the organic phase.

Table 11. Effect of time upon Ta stripping efficiency.

Time, min	Stripping efficiency, %	
	Ta	**Nb**
3	39.73	nil
5	68.90	nil
7	81.32	nil
9	99.34	nil

ii) Construction of McCabe-Thiele diagram for Ta stripping
From the equilibrium stripping data and the corresponding McCabe-Thiele diagram shown in **Fig. 3**, the theoretical number of stripping stages would be determined after fitting of the operating line. These data have been obtained by contacting several loaded solvent aliquots with distilled water for 9 min using different A/O ratios. Accordingly, three theoretically stripping stages would be required for almost complete Ta stripping.
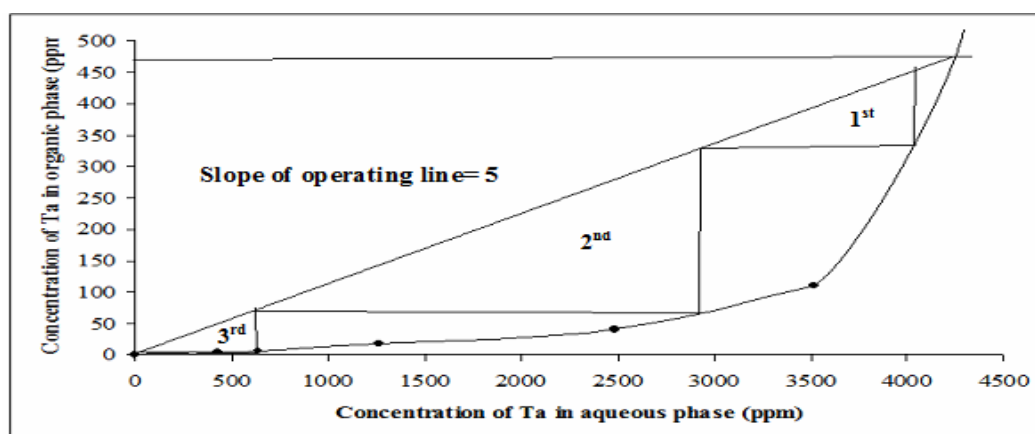

Fig. 3. McCabe-Thiele diagram for Ta stripping.

Niobium stripping
Referring to **Table (10)**, HNO_3 is the most efficient for Nb stripping from the loaded solvent free of Ta.

i) Effect of acid concentration

The different concentrations of HNO_3 were varied from 0.3 to 1.2 M at the fixed conditions of A/O ratio of 1/1 and 12 min as contact time. The corresponding stripping efficiencies are measured and tabulated in **Table (12)**. From the obtained data, it is concluded that the best nitric acid conc. for stripping of Nb is 1.2 M to realize 99.55 % stripping efficiency.

Table 12. Effect of HNO_3 concentration upon Nb stripping efficiency.

HNO_3 conc., M.	Nb Stripping efficiency, %
0.3	55.24
0.6	70.90
0.9	89.50
1.2	99.55

ii) Effect of stripping time

The effect of stripping time upon Nb stripping efficiency was studied in the range from 3 to 12 min. at 1.2 M HNO_3 and O/A ratio 1/1. From the obtained results **(Table 13)**, it is found that 12 min as stripping time is sufficient to bring almost all Nb content into the stripping solution.

Table 13. Effect of time upon Nb stripping efficiency.

Time, min	Nb Stripping efficiency, %
3	63.50
6	74.66
9	87.30
12	99.55

iii) Construction of McCabe-Thiele diagram for Nb stripping

From the equilibrium stripping data and the corresponding McCabe-Thiele diagram as shown in **Fig. 4**, the theoretical number of stripping stages would be determined. These data have been obtained by contacting several loaded solvent aliquots with a mixture of 1.2 M HNO_3 for 12 min using different A/O ratios. From the McCabe- Thiele diagram, and according to the given operating line, theoretically four stripping stages are required for almost complete stripping of Nb from the loaded isodecanol.
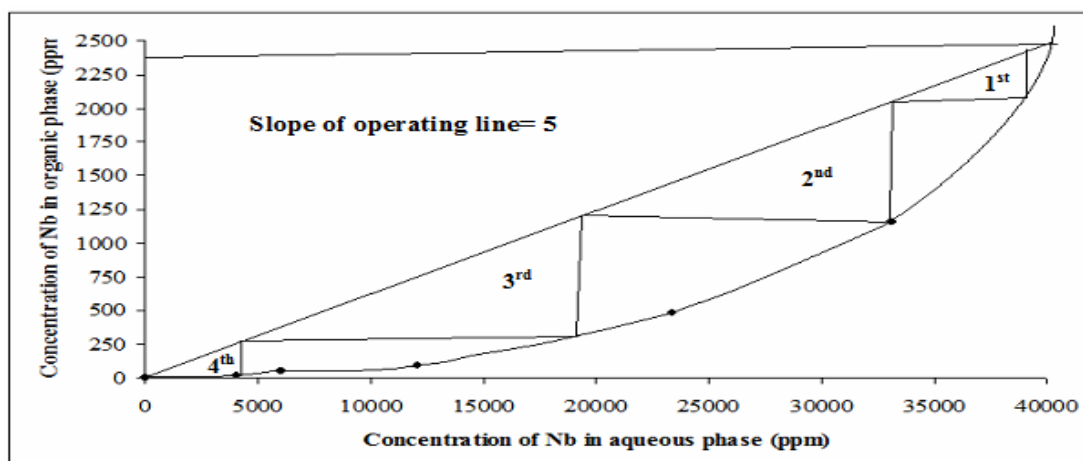
Fig. 4. McCabe-Thiele diagram for Nb stripping.

Tantalum and niobium precipitation
i) Tantalum precipitation
By neutralizing the corresponding Ta strip solution using ammonia solution, Ta was completely precipitated at pH 5.5. After calcination, a relatively pure Ta_2O_5 was obtained and analyzed using ICP-MS which 96 % purity.
ii) Niobium precipitation
By neutralizing the corresponding Nb strip solution using ammonia solution, Nb was completely precipitated at pH 7.5. The product obtained was calcined and analyzed using ICP-MS to show 94 % purity.

Conclusion
The acid agitation leaching procedure using H_2SO_4 was applied for leaching of Nb, Ta and REEs metal values of the representative ore sample of Um Tomyem area, southwestern Sinai. The optimum conditions of acid agitation leaching step for almost complete dissolution of Nb, Ta and REEs involved 300 g/l H_2SO_4 at S/L ratio 1/2 of 300°C for 3 h. The obtained sulfate liquor is subjected to treatment via precipitation of REEs at pH 0.8 using 10% oxalic acid before applying solvent extraction for Nb and Ta. Subsequently, Ta and Nb recovered together by solvent extraction technique using isodecanol (100%) at different contact times for different pH values and stripped by distilled water and different concentrations of acids. The studied optimum extraction conditions included 100% isodecanol conc., an O/A of 1/1 at a pH 0.9 of the treated sulfate solution and a contact time of 12 min to realize the extraction efficiencies of Ta 99.7 % and Nb 99.3 %. The studied optimum conditions for stripping of Ta are distilled water as stripping agent, A/O ratio 1/1 and 9 min as stripping time while Nb remained in the organic phase. The studied optimum conditions for stripping of Nb are 1.2 M HNO_3 as stripping agent, A/O ratio 1/1 and 12 min as stripping time. Highly pure products of Ta

and Nb have been prepared and analyzed using ICP-MS. Finally, a technical flowsheet is proposed for separation of Ta and Nb from the treated sulfate leach liquor as shown in **Fig. 5**.
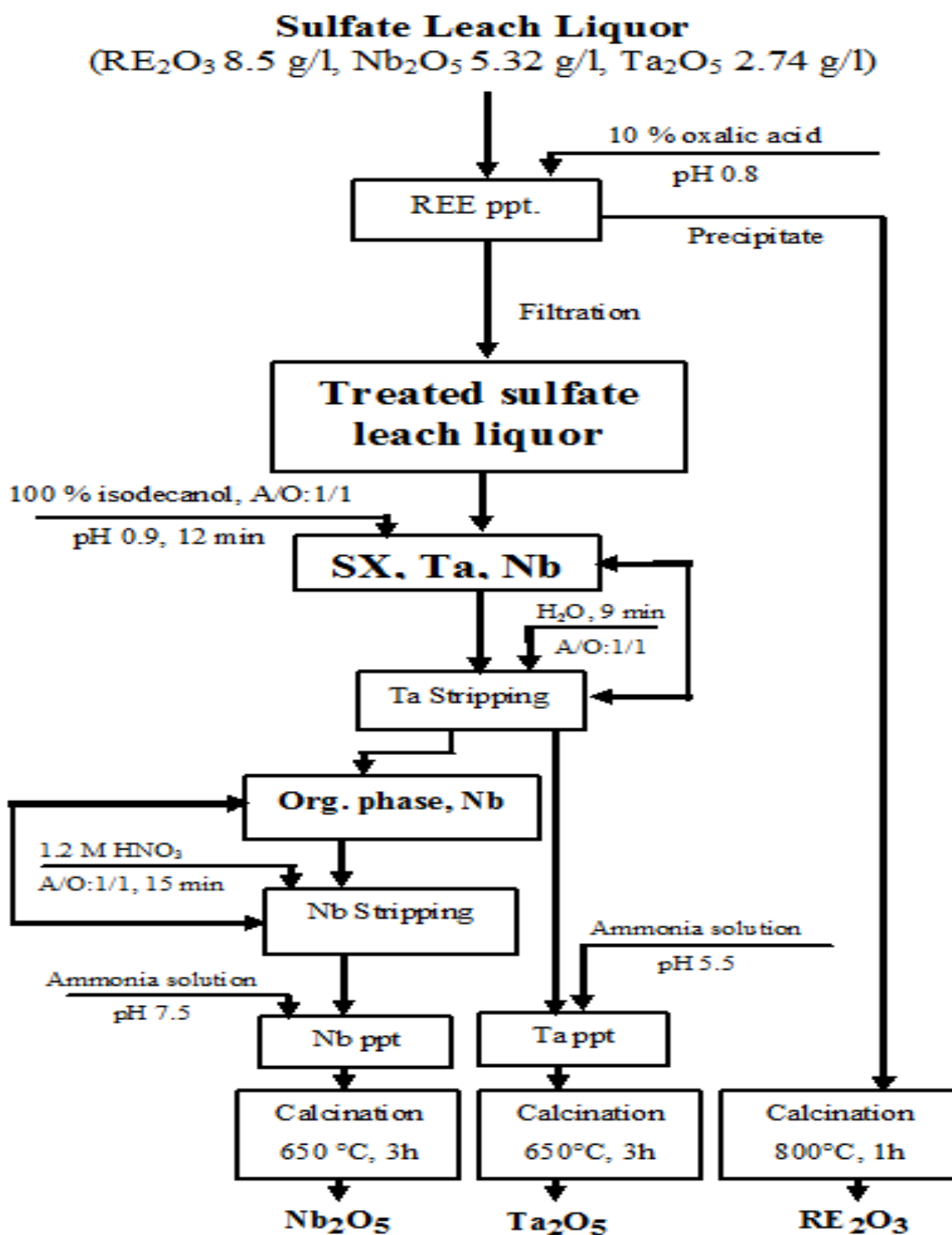
Sulfate Leach Liquor
(RE_2O_3 8.5 g/l, Nb_2O_5 5.32 g/l, Ta_2O_5 2.74 g/l)

10 % oxalic acid
pH 0.8

REE ppt.

Precipitate

Filtration

Treated sulfate leach liquor

100 % isodecanol, A/O:1/1
pH 0.9, 12 min

SX, Ta, Nb

H_2O, 9 min
A/O:1/1

Ta Stripping

Org. phase, Nb

1.2 M HNO_3
A/O:1/1, 15 min

Nb Stripping

Ammonia solution
pH 5.5

Ammonia solution
pH 7.5

Nb ppt

Ta ppt

| Calcination 650 °C, 3h | Calcination 650°C, 3h | Calcination 800°C, 1h |

Nb_2O_5

Ta_2O_5

RE_2O_3

Fig. 5. Proposed technical flowsheet for the treated sulfate solution.

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فصل التنتالوم والنيوبيوم من محلول الكبريتات المعالج مستخدما أيزوديكانول

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هيئة المواد النووية : ص.ب 530 المعادى – القاهرة ـ مصر

الملخص العربى

تناول البحث معالجة عينة تكنولوجية من الصخور الرسوبية متعددة العناصر مجمعة من جنوب غرب سيناء – والتى تحتوى على 2,61 % أكاسيد الأرضيات النادرة (RE_2O_3) وعلى 1,14 % أكسيد التنتالوم (Ta_2O_5) وعلى 2,16 % أكسيد النيوبيوم (Nb_2O_5) ، وذلك بطريقة الأذابة بالتقليب بإستخدام حمض الكبريتيك تحت مختلف الظروف ، وتلى ذلك أسترجاع أكاسيد الأرضيات النادرة من محلول الأذابة فى صورة الأوكسالات ، بينما تم دراسة أستخلاص النيوبيوم والتنتالوم من المحلول المتخلف بمذيب عضوى أيزوديكانول ، وبعد التوصل إلى تحديد الظروف المثلى لكلا من عملتى " الأستخلاص والأسترجاع " للنيوبيوم والتنتالوم. تم تحضير ركاز عالى النقاوة لكلا من منتج النيوبيوم والتنتالوم وتم تحليلها بإستخدام مطياف البلازما (ICP-MS).