



ISSN 1110-0451



(E S N S A)

Leaching the Co-Precipitated Thorium from Light Rare Earth Double Sulfate and Preparation of High-Grade Thorium Concentrate

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ARTICLE INFO

Article history:

Received: 1st Nov. 2022

Accepted: 18th Jan. 2023

Available online: 25th Feb. 2023

Keywords:

Light rare earth Double sulfate;

Thorium precipitation;

Leaching;

High-grade Concentrate.

ABSTRACT

During selective precipitation of light rare earth elements from concentrated sulfuric acid monazite sulfate leach solutions, substantial amount of thorium was co-precipitated. The current study deals with leaching most of this co-precipitated thorium under various conditions of pH, solution/solid precipitate ratio, agitation time, and leaching temperature. Additionally, the preparation of high-grade thorium concentrate suitable for further purification steps was involved from the produced leach liquors. This is conducted through some thorium selective precipitation methods such as precipitation at controlled pH value and hydrolysis. The optimized conditions reveal that leach water of pH =1.0, the ratio to light rare earth double sulfate solid precipitate is 20.0 and agitation for 6.0 h at 80°C dissolve more than 79% of Th with only minimum of light rare earth elements as 2.8%. However, the initial prepared thorium concentrate contains about 45% Th, the final concentrate contains more than 70% thorium which seems to be suitable to conduct further purification steps.

1- INTRODUCTION

Thorium is a naturally occurring radioactive element with a single isotopic form (²³²Th) [1]. On the other hand, thorium is more abundant than uranium by about three to five times. However, thorium is very insoluble in water, which explains why it is not present in seawater as uranium. Thorium occurs naturally in relatively small number of minerals which are enriched with it such as thorite {(U,Th)SiO₄}, thorianite {ThO₂}, monazite {(Ce,La,Nd,Th,U)PO₄}, bastnaesite {(La,Ce,Th) CO₃F}, and thorogummite {(Th,U)(SiO₄)_{1-x}(OH)_{4x}} [2].

The main world resources of thorium are generally coupled with rare earth minerals as monazite and bastnaesite, where the mineral monazite contains up to 12 wt.% ThO₂, however, its average in this mineral is 6 - 7%, while bastnaesite mineral contains up to 2.8 wt.% ThO₂. So the mineral monazite was considered the most common resource of thorium [2]. However, thorium production in many countries is not economic without commercial rare earth recovery, extraction of thorium is a by-product from the rare earth elements

Earth mineral monazite seems to be the most feasible at this time [3]. The richest concentrations of monazite are found in black sand beach deposits which are concentrated (with other heavy minerals) by wave and current action. Monazite is widely distributed mainly in some countries including India, Brazil, Australia, Venezuela, USA, Egypt, and other several countries, where it is found in large quantities and contains

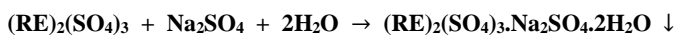
about 6.4 million metric tons of thorium. However, Egypt has 0.38 million metric tons of these world resources and represents the fifth country over the world for this resource [2, 4 – 6].

Thorium's radioactivity was widely acknowledged during the first decades of the 20th century, so in the second half of the century, thorium was replaced in many uses, however, thorium is still being used in many non-nuclear (domestic) and nuclear fields. In this regard, thorium oxide or thoria (ThO₂) has one of the highest melting points of all oxides "more than 3300°C "; accordingly, it was applied in arc-light lamps, welding electrodes and to make heat-resistant ceramics and

crucibles [7]. Thorium oxide is also used as a catalyst for industrial chemical reactions such as in the production of sulfuric acid (H₂SO₄) and nitric acid (HNO₃) (thorough oxidation of ammonia (NH₃)) as well as in the cracking of petroleum products [8]. Thorium tetrafluoride is used as an anti-reflection material in multilayered optical coatings [9]. On the other hand, thorium is used to coat tungsten filaments in light bulbs that are used in electronic devices [10] such as in television sets. Thorium-magnesium alloys are light in weight, of excellent strength and of creep resistance at high temperatures when compared to magnesium, so it was used in the aerospace industry for aircraft engines [11]. Recently, thorium is used as inorganic extractant in the form of acrylamide [12].

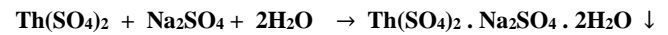
From nuclear fields point of view, ²³²Th is considered a fertile material because when absorbing a neutron, it will transmute to ²³³U which is an excellent fissile fuel material. Because of this, thorium is a potentially attractive alternative to uranium in mixed oxide nuclear fuels to minimize the generation of transuranic and maximize the destruction of plutonium [13]. Moreover, thorium results in a safer and better-performing reactor core because ThO₂ has a higher melting point, higher thermal conductivity, and a lower coefficient of thermal expansion and is more chemically stable than the now-common fuel UO₂ [14, 15].

Among several precipitation method for REE, precipitation of light rare earth elements (LREE) from concentrated sulfuric acid solutions seems to be interesting method for direct and selective precipitation with anhydrous sodium sulfate (or NaCl) in the form of rare earth sodium double sulfate (REDS) with 98% recovery efficiency [16]. The precipitation reaction proceeds according to the following equation:



The resultant REDS precipitate has many good physical and chemical properties as it was dense and crystalline so it was easily filtered and washed. In addition, it can be easily converted to hydroxides by digestion with caustic soda. The other advantages include the precipitation method which was conducted directly upon the concentrated sulfuric acid solutions, this would permit recycling the solutions containing mixed acids (sulfuric and phosphoric) for subsequent monazite breakdown process which allow the breakdown to be more efficient than sulfuric acid alone, owing to the greater solubility of monazite constituents in these acids [17].

The product of REDS composed mainly of the majority of light rare earth elements (La, Ce, Nd and Pr) which is convenient and easily stored as starting material for the manufacture of rare earth materials, but appreciable amount of thorium can be co-precipitated at 25°C [16 -18] according to the following equation:



The present study deals with minimization of this co-precipitated amount of thorium from the REDS precipitate through leaching step then preparation of high grade thorium concentrates from the produced leach liquor through several precipitation and treatments.

2- EXPERIMENTAL

The used chemicals and reagents in the present work are of analytical grade for the analysis procedures, except for commercial chemicals which are used as in the preparation steps for high-grade thorium concentrate from an the Egyptian low-grade monazite sample. It is worthy also to mention that tap water was used during these preparation steps, while double distilled water was used during the chemical analysis.

UV/VIS spectrophotometer, multi positions, double beam model T 80 version 24-1884-010001, with glass cell of 10 mm was used for Th and Ce determination.

Thorium was determined using 0.1% Thoron (I) reagent according to the method described by Marczenko [19]. Cerium was determined after oxidation using ammonium persulfate reagent according to the method described by Dean[20] .

PH-mv-temp.-meter, HAANA instruments provided with H1 1270 combination electrode and temperature sensor was used for solution samples pH adjustment and temperature measurement.

Scanning Electron Microscope with Energy Dispersive X-Ray (SEM-EDX), model Philips XL 30, was used for elements identification and qualitative analysis under conditions of low vacuum, 30 KV.

2.1 Preparation of High-Grade Thorium Concentrate

High-grade thorium cake was prepared from the crude (or low grade) Egyptian monazite sand of grade about 47% which can be suitable as starting material for conducting the further purification steps through extraction. This crude monazite was produced as a by-product during the concentration of the more abundant minerals such as ilmenite, rutile, zircon, magnetite, garnet, etc. from the black sand placer deposits at Rosetta area on the Mediterranean Coast, Egypt. From

previous works and gained experience [16, 17, 22 – 25], the Egyptian crude monazite sand was treated through the sulfuric acid digestion process followed by leaching, light rare earth double sulfate precipitation.

3- RESULTS AND DISCUSSION

3-1 Preparation of Light Rare Earth (LREE) double sulfate

After digestion of an Egyptian low-grade monazite sample, from Rosetta area, leaching then the LREE was precipitated as double sulfate, this precipitate contains appreciable amount of thorium. Preparation of thorium concentrate after its leaching from the produced LREDS was considered.

Results of digestion, dissolution (or leaching) of the resultant gray paste constituents and the subsequent selective precipitation of the LREE as double sulfate using sodium chloride (NaCl) from the monazite sulfate clear solutions is summarized in Table (1).

Table (1): Results of digestion, dissolution of the gray paste and subsequent selective precipitation of the LREDS

Treatment step	Step Efficiency, %	
	Rare Earth Elements	Th
Digestion	92.6	96.6
Leaching	98.7	99.4
Double sulphate "DS" precipitation	91.5	43.7

On the other hand, concentrations of some main constituents in the clear monazite sulfate solution before the selective precipitation of LREE double sulfate are given in Table (2).

Table (2): Concentration of some Main Constituents in the Monazite Sulfate Solution

Constituent	Concentration, g/l
P	14.73
Ce	14.54
Total REE except Ce	17.87
Th	3.22
U	0.27
Fe	4.41
Ti	0.38

However, as previously mentioned in the literature review that appreciable amount of thorium could be co-precipitated during this precipitation step which is 43.7% from the originally present thorium in the monazite sulfate solution as indicated in Table (1).

Qualitative analysis of the prepared light rare earths double sulfate precipitate using Scanning Electron Microscope with Energy Dispersive X-Ray (SEM-EDX) instrument is illustrated in Fig. (1) and the corresponding Table.

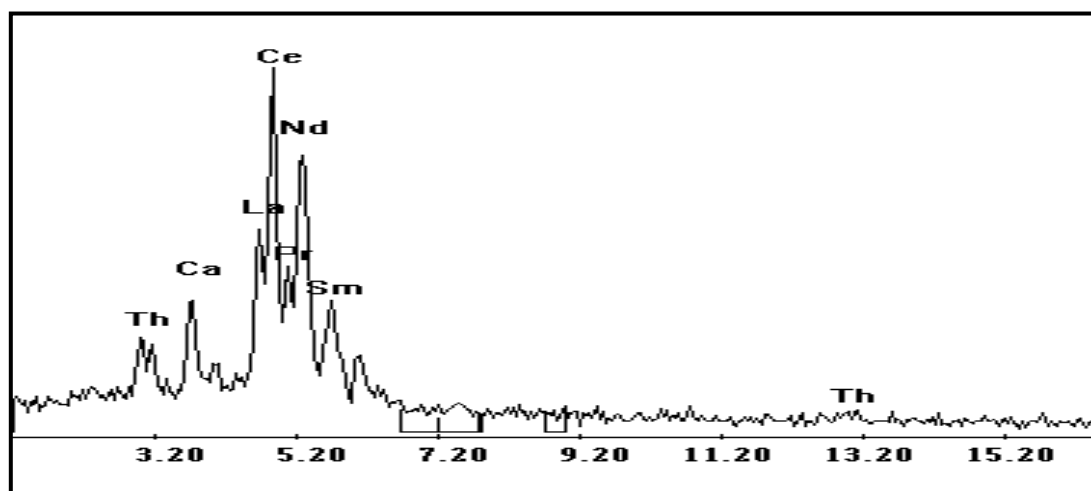


Fig. (1): Qualitative analysis using Scanning Electron Microscope with Energy Dispersive X-Ray (SEM-EDX) for the prepared light rare earths double sulfate precipitate

The analysis reveals that double sulfate precipitate contains 13.5% of thorium which co-precipitated with it. Accordingly, the purpose of this work is:

- To recover this appreciable co-precipitated amount of thorium,;
- Almost decontamination of the light rare earth double sulfate precipitate from the radioactive thorium element through leaching step;
- Preparation of high-grade thorium concentrate, from the produced leach liquors, suitable for conducting further thorium purification step.

The prepared sample of light rare earths double sulfate precipitate containing thorium represents the starting material for the subsequent work which will be discussed in the following sections.

3-2 Leaching of Thorium from the Light Rare Earths Double Sulfate Precipitate

Leaching the appreciable co-precipitated amount of thorium with minimum as possible of the light rare earth elements from the prepared double sulfate precipitate will be discussed herein. However, depending upon the fact that rare earth double sulfate precipitate is stable against solubility even in concentrated acids [26]. After good washing for the precipitate from any contaminates and drying, 25 gram of the precipitate is subjected to leach most of the co-precipitated thorium constituent with acidified water under different conditions as leach water pH, its ratio to the solid precipitate, agitation time and finally leaching temperature. Results of studying these factors will be discussed in the following sections.

3-2-1 Effect of the Leaching Agitation Time

Agitation time represents also a remarkable effect on thorium leaching efficiency from the light rare earths double sulfate precipitate. Thus, it was studied under the conditions of leach water of pH 1.0 and its ratio to the dry cake of light rare earths double sulfate precipitate of 10.0 at ambient temperature. The results shown in Fig. (2) show that the leaching efficiency was increased with proceeding agitation time for both thorium and the light rare earths elements.

In this regard, after one hour the leaching efficiency of thorium was 18.5% with low solubility for the light rare earths elements which has about 4% i.e. Th/Ce solubility ratio was 4.8. Increasing the agitation time, Th/Ce solubility ratio will be decreased gradually to about 3.36 after 3.0 h and reached 2.2 after 12.0 h i.e increasing the solubility of light rare earths elements with increasing the contact time. Accordingly, it was recommended to conduct the leaching step at time from 1.0 to 3.0 h to achieve the maximum solubility for thorium with minimum solubility for the light rare earth elements. It was found that time 3h is the best from an industrial and technological point of view, as it achieves our goal of separating thorium instead of other elements in order to enhance the amount of thorium relative to the rest of the elements.

3-2-2 Effect of the Leach Water/Solid Ratio “V/W”

The effect of the leach water to dry cake of the light rare earth double sulfate precipitate ratio (leach water/solids) was studied to choose the lower ratio that achieve maximum solubility of thorium with the minimum of those light rare earth elements. The study was conducted at the conditions of agitation the slurry of pH 1.0 for 3.0 h at ambient temperature. The results are presented in Fig. (3), from which it is clear that a sharp increase in the leaching efficiency for both Th and LREEs with increasing the ratio from 10.0 to 20.0 has occurred. In this regard, leaching efficiency was 28% and more than 8% at the ratio 10.0 and reach to 60% and about 22% at the ratio 20.0 for thorium and light rare earth elements respectively. However, minimum solubility of light rare earth elements was achieved at the ratio 7.5 with the maximum solubility for thorium (27%), so this ratio seems to be suitable to conduct the leaching step. Here, ratio10 was selected as the best ratio from the industrial and technological point of view, as it achieves our goal in separating thorium instead of other elements in order to enhance the amount of thorium in relation to the rest of the elements. It is also the best from the industrial side in terms of reducing the volume of solutions and thus shrinking the volume of dissolving tanks, and which is considered very important. From an economic point of view, in operating operations in the electricity used to transport the huge amount of these solutions.

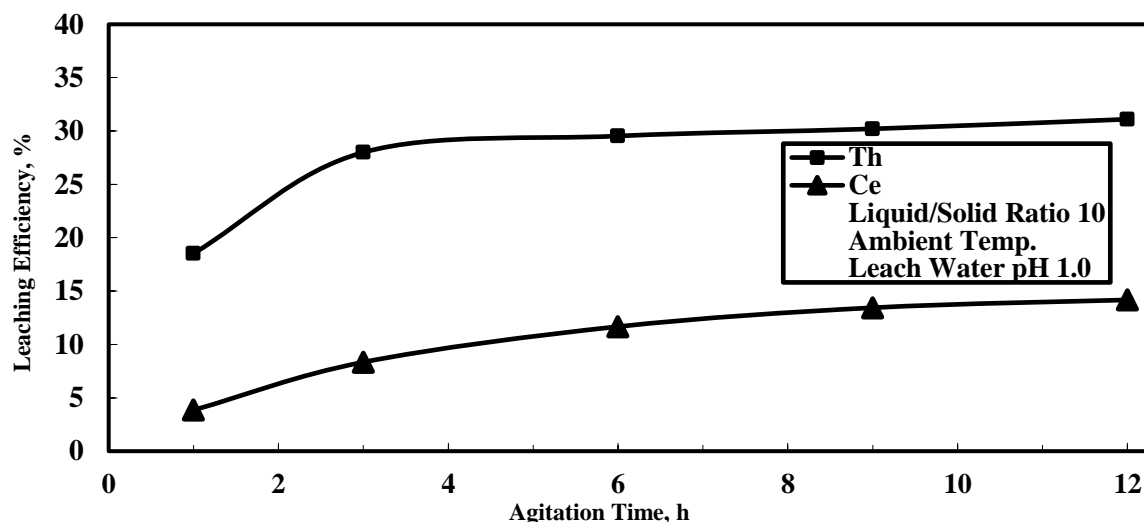


Fig. (2): Effect of Time on Leaching Efficiency for Th and Ce from Double Sulphate Precipitate

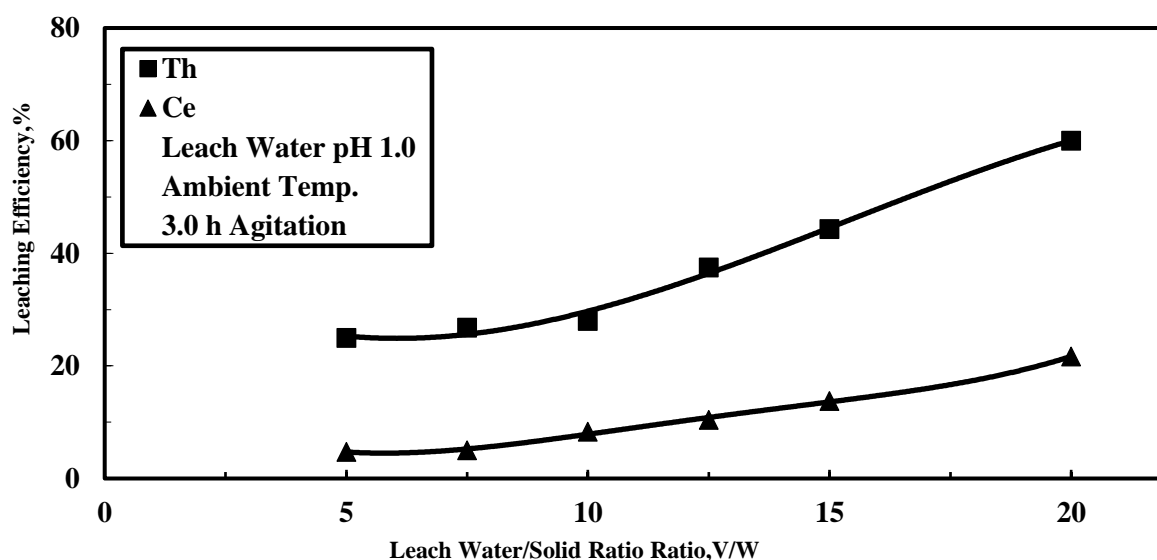


Fig. (3): Effect of Leach Water/Solid Ratio on Leaching Efficiency for Th and Ce from the Double Sulphate Precipitate

3-2-3 Effect of the Leach Water pH

The effect of the leach water pH upon thorium leaching efficiency from the dry cake of the prepared light rare earths double sulfate precipitate was firstly studied under the conditions of leach water/solids ratio of 10 and agitation for 3.0 h at ambient temperature, where pH was adjusted with sulfuric acid. Results of studying this factor are indicated in Fig. (4).

The results reveal that the suitable leach water pH was 1.0 which represents peak point at which 28% of thorium was leached with about 8% of cerium (that represents an indication for the leaching of the other light rare earth elements).

The interpretation of this result emphasizes fact that at low pH values, thorium and light rare earths double sulfates are relatively stable against solubility and the formation of double sulfates was enhanced, while at higher pH values (more than 1.0) the solubility is lesser because of there is no presence of enough acid to keep the leaching proceed efficiently. However, at pH 2.0, thorium leaching efficiency was 14% (half of that at pH 1.0) while the corresponding of cerium was 0.4% (1/20 of that at pH 1.0), so leaching at pH 2.0 may be beneficial to leach maximum thorium with as minimum as possible of the LREEs.

3-2-4 Effect of Temperature on Thorium Leaching Efficiency

Temperature represents an important factor that affect the leaching efficiency of thorium from the light rare earths double sulfate precipitate, so this factor was also studied. The study was conducted at the conditions of leach water of pH 1.0 and the ratio to the solids of light rare earths double sulfate precipitate of 10.0 and agitation for 3.0 h. Results of studying this factor were presented in Fig. (5), from which it is clear that thorium leaching efficiency was increased sharply with increasing the temperature from ambient to 90°C and the opposite occurred for the light rare earths elements where it was sharply decreased. This effect was predicted depending on the fact that the solubility of rare

earths sulfate decreases with increasing the temperature (21). The results reveal that the maximum dissolution of 38% for thorium was achieved with minimum of 1.6% for that of light rare earths elements at 80°C.

Accordingly, an optimized experiment was conducted under the conditions of pH 1.0 for the leach water at ratio to the light rare earths double sulfate solid precipitate of 20.0 and agitation for 6.0 h at 80°C, at these conditions more than 79% of Th was dissolved with only minimum of LREEs as 2.8%.

The remained double sulfate precipitate after thorium leaching was specified qualitatively using the Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray (EDX) instrument where result of the analysis was illustrated in Fig. (6) and the corresponding Table.

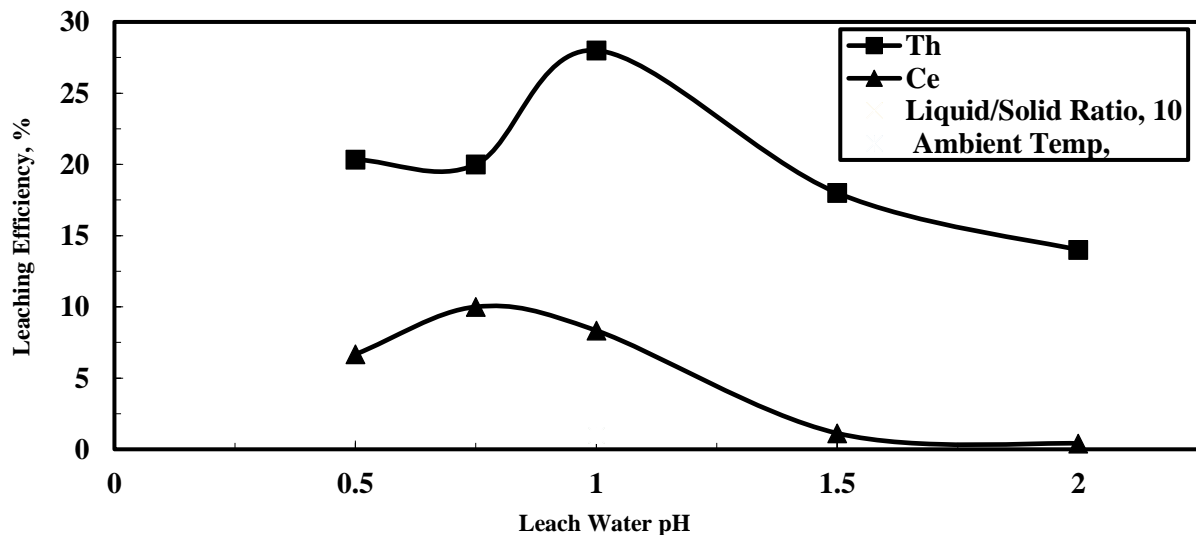


Fig. (4): Effect of pH on Leaching efficiency for Th and Ce from the Double Sulphate Precipitate

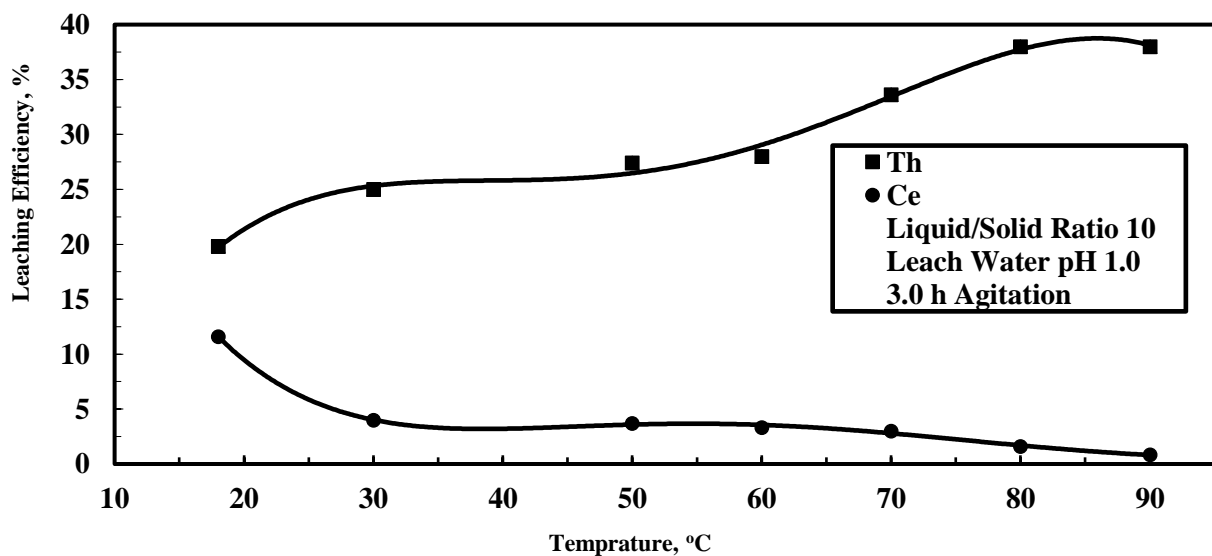


Fig. (5): Effect of temperature on Leaching Efficiency for Th and Ce from the Double Sulphate Precipitate

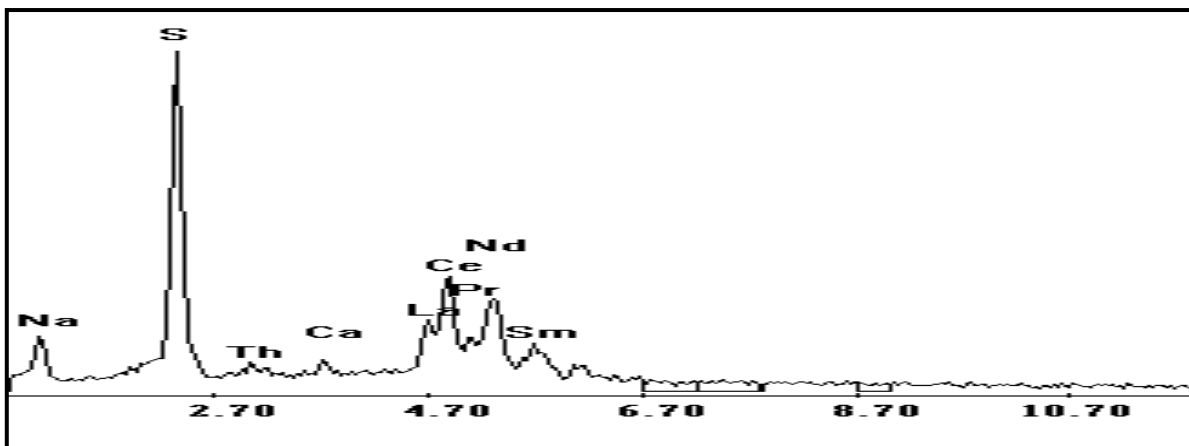


Fig. (6): Qualification of the Remained Double Sulphate Precipitate after Thorium Leaching using SEM- EDX

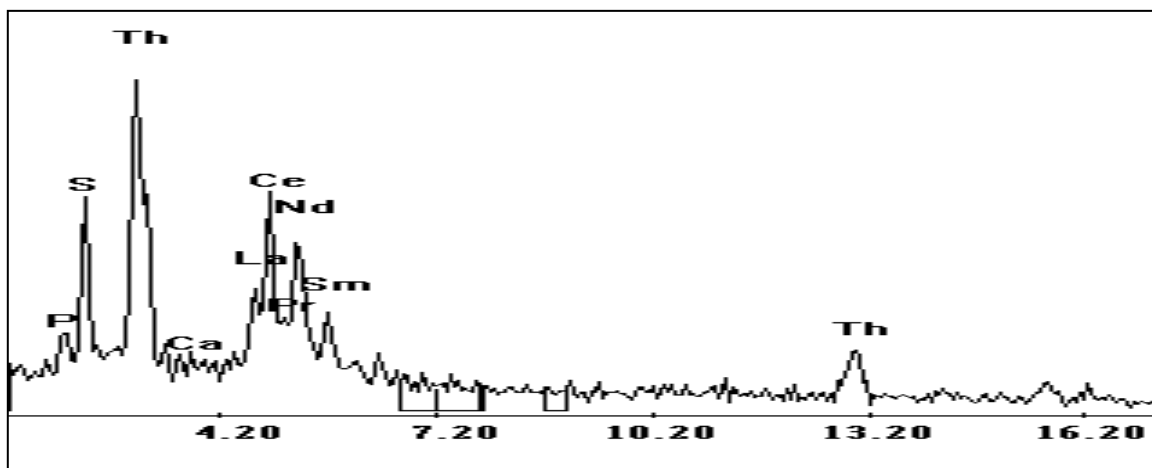


Fig. (7): Qualification of the Initial Thorium Concentrate using SEM-EDX

3-3 Preparation of Thorium Concentrate from the Leach Liquor of the Light Rare Earths Double Sulfate Precipitate and Further Up-grading

3-3-1 Step “I” Precipitation through pH control

The leach liquor produced from the optimized experiment for thorium leaching from light rare earths double sulfate precipitate was subjected for neutralization to pH 5.8 ⁽²¹⁾. The initial thorium concentrate produced was filtered, washed then dried over night at 110°C. The concentrate was specified qualitatively using the Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray (EDX) where the result of the analysis was illustrated in Fig. (7) and the corresponding Table.

The analysis shows that thorium concentrate containing about 45% Th, however, the presence of

high cerium concentration (more than 22%) was due to conversion part of cerium to its tetravalent state which can be co-precipitated with thorium.

3-3-2 Step “II” Precipitation through hydrolysis

This initial thorium concentrate was subjected to further up-grading step through re-dissolution with sulfuric acid in the presence of hydrogen peroxide to reduce any tetravalent cerium to its trivalent state then precipitation of thorium through hydrolysis with water. However, the pH of the solution has a decisive effect upon thorium hydrolysis, so it was firstly studied. The results are given in Table (2), from which it is clear that thorium precipitation efficiency increased sharply with decreasing the solution pH (i.e. with increasing the acidity), also, the required dilution ratio was decreased

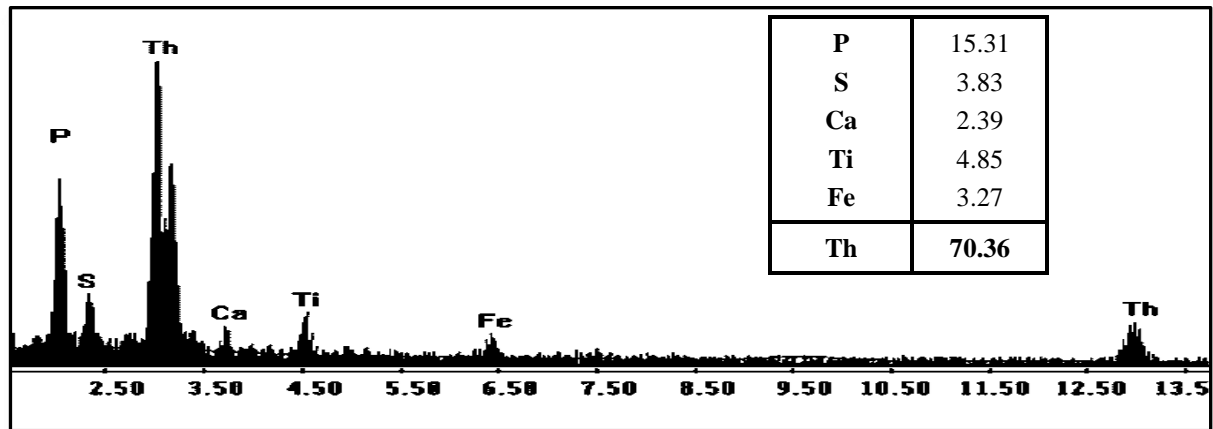


Fig. (8): Qualification of the Final Thorium Concentrate using SEM-EDX

Table (3): Results of thorium hydrolysis Experiments

Solution pH	Dilution Ratio	Thorium Precipitation Efficiency, %
1.0	25	40.35
	50	70.84
	100	80.12
0.9	10	48.83
	25	79.67
	50	85.91
0.8	10	55.75
	20	83.2
	25	89.4
0.75	10	88.9
	20	93.18
	25	94.87

Thus, the solution pH of 0.75 was the effective value as initial pH to achieve maximum hydrolysis and the corresponding dilution ratio is 20 - 25.

The resultant thorium concentrate produced from this hydrolysis precipitation step was filtered, washed then dried over night at 110°C. The concentrate was qualified using the Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray (EDX) instrument where result of the analysis was illustrated in Fig. (8) and the corresponding Table. The result reveals that the concentrate contains more than 70% thorium and it seems to be suitable to conduct the further purification steps.

4- CONCLUSION

The co-precipitated thorium with light rare earth double sulfate concentrate can be leached effectively with only minimum of cerium under the condition of leach water of pH 1.0 and in ratio to the solids of 20 then agitation for 6.0 h at 80°C, at these conditions more than 79% of Th was dissolve with only minimum of LREEs as 2.8%. The leach solutions produced from the optimized experiment for thorium leaching from light rare earths double sulfate precipitate was subjected for preparation high grade thorium concentrate. This was done through two stages, the first one is neutralization to pH 5.8 at which initial thorium concentrate produced contains about 45% Th, while the second stage through re-dissolution with sulphuric acid in the presence of hydrogen peroxide to reduce any tetravalent cerium to its trivalent state then precipitation of thorium through hydrolysis with water under the conditions of the solution pH of 0.75 dilution ratio is 20 - 25. The produced final thorium concentrate from this stage contains more than 70% thorium and it seems to be suitable to conduct the further purification steps. According to these results, the proposal flow sheet (Fig. 9.) was constructed to summarize the suggested process. These results in terms of the scientific methodology are the best because they gave the highest efficiency in all conditions, but from the point of view of industry another opinion is to choose the results according to the specifications of the product needed. It depends whether the needed thorium without any other elements, or a thread between thorium and light rare earth elements, and here principal objective to be achieved is a higher grade thorium concentrate, so the least hits in the other elements is taken to reduce their quantity as much as possible.

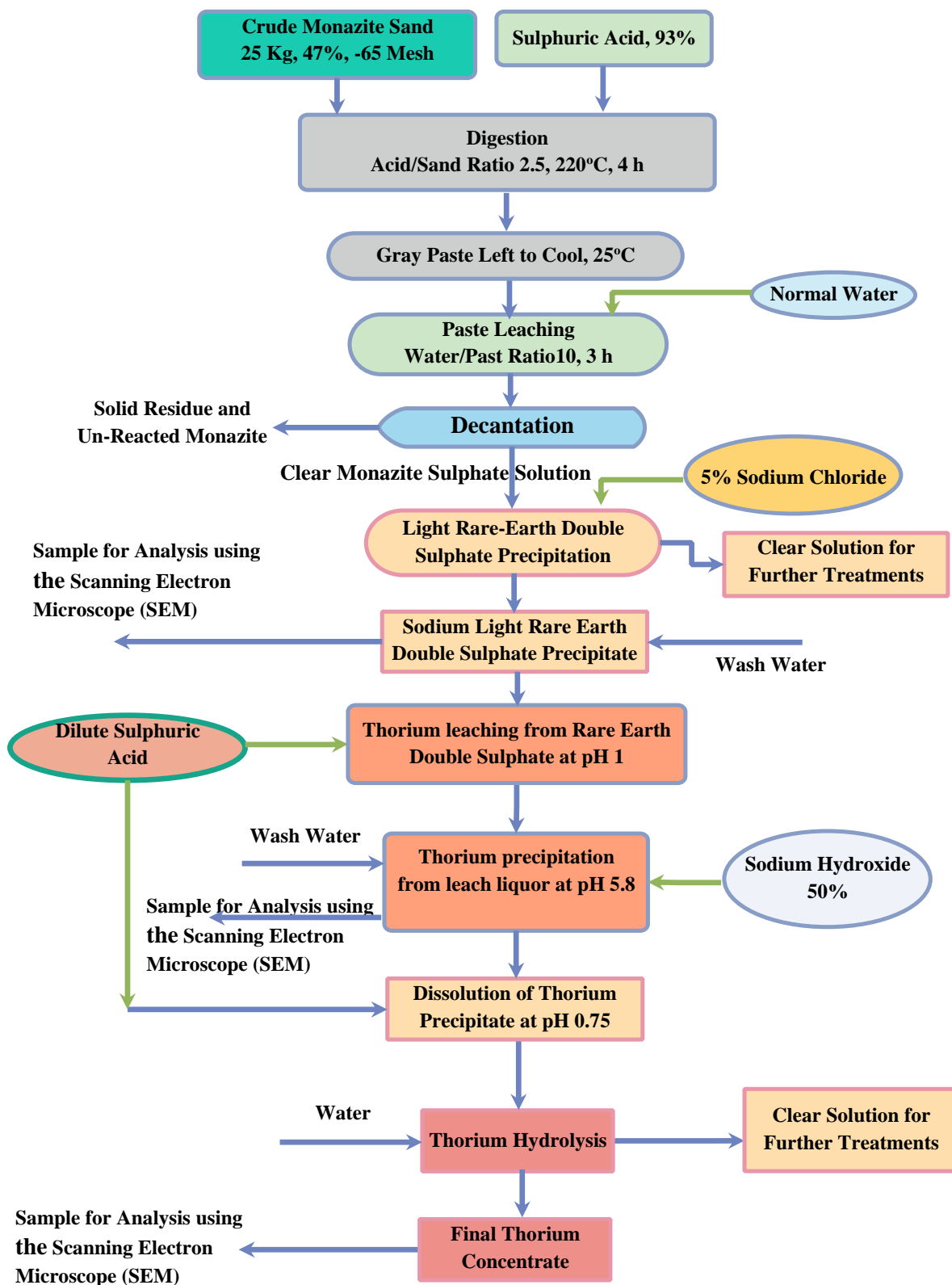


Fig. (9): Methodology of Leaching the Co-precipitated Thorium from the Light Rare Earth Double Sulphate and Preparation of High-Grade Thorium Concentrate from the Leach Liquors

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