Research Paper

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Solvent Extraction of Yttrium from Rare Earths Concentrate Using Cyanex 272 and PC88A

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ABSTRACT : **The recovery of yttrium using cyanex 272 & PC88A from the REEs concentrate which produced from xenotime–bearing ferruginous sandstone, southwestern Sinai was studied. Cyanex 272 solvent is known as a purified bis(2.4.4-trimethylpentyl) phosphinic acid and PC88A solvent is also known as a purified 2-Ethylhexyl 2-ethylhexy phosphonic acid (HEHEHP). From a solution of rare earths chloride that prepared, the optimum conditions for yttrium extraction included mixture of 1.4 M cyanex 272 & 1.8 M PC88A / 0.25 M isodecanol in kerosene, 1/1 O/A ratio, the prepared liquor pH 3 and contact time 12 min. The decided optimum conditions of yttrium stripping are 3 M HNO3 in 1/1 O/A ratio for contact time 25 min. McCabe-Thiele diagram for yttrium extraction and stripping are created. Very pure product of yttrium was prepared and analyzed using ICP-MS.**

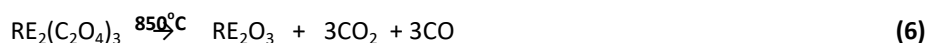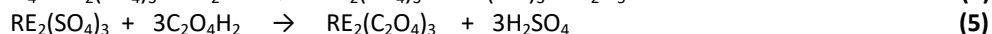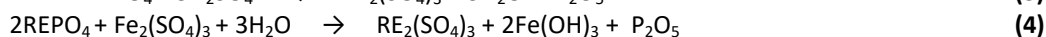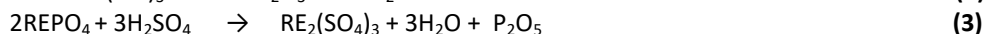***KEYWORDS: Yttrium; Solvent extraction; Cyanex 272; PC88A; Rare earths.***

I. INTRODUCTION

Yttrium is classified as a rare earth element due to its chemical similarity to lanthanides, a silver metallic transition metal [1]. Yttrium does not occur alone in nature and is nearly continuously combined with the lanthanide elements in rare earth minerals [2]. Global reserve of yttrium is evaluated at more than 500 thousand tons, most of which are found in Australia, Brazil, Canada, China and India [3]. Yttrium is utilized within in the make of thermal plasma sprays to secure high-temperature flight surfaces, electrolytes, electrodes, superconductors, electronic filters, and lasers [4-8]. Given its superior thermal stability and highly attainable hydrogen density, yttrium hydride is an great high-temperature moderator material in progressed nuclear reactors that require small core volumes [9]. Moreover, yttrium is utilized in radars and as an additive within alloys in high tech devices [10,11].

Xenotime, a phosphate mineral, is one of the important mineral deposits of rare earths (>40% Y) and most of its elements are included within the operation of atomic reactor. Xenotime mineral can be processed by either caustic fusion [12] or by the sulfuric acid process [13] or by ferric sulfate leaching [14] followed by addition of oxalic acid on rare earths sulfate to get on rare earths oxalate. The rare earths hydroxide and rare earths oxalates are ignited at 850°C to get on rare earths concentrate and outlined by the Eqs. 1 - 6. From the latter, yttrium is extracted by three diverse strategies; direct precipitation [15,16], ion exchange resin [17-19] and solvent extraction [20,21].

$$REPO_4 + 3NaOH \quad \rightarrow \quad RE(OH)_3 + Na_3PO_4 \tag{1}$$

$$2RE(OH)_3 \quad \xrightarrow{850^oC} \quad RE_2O_3 \ + \ 3H_2O \tag{2}$$

$$2REPO_4 + 3H_2SO_4 \quad \rightarrow \quad RE_2(SO_4)_3 + 3H_2O + P_2O_5 \tag{3}$$

$$2REPO_4 + Fe_2(SO_4)_3 + 3H_2O \quad \rightarrow \quad RE_2(SO_4)_3 + 2Fe(OH)_3 + P_2O_5 \tag{4}$$

$$RE_2(SO_4)_3 \ + \ 3C_2O_4H_2 \quad \rightarrow \quad RE_2(C_2O_4)_3 \ + \ 3H_2SO_4 \tag{5}$$

$$RE_2(C_2O_4)_3 \quad \xrightarrow{850^oC} \quad RE_2O_3 \ + \ 3CO_2 + 3CO \tag{6}$$
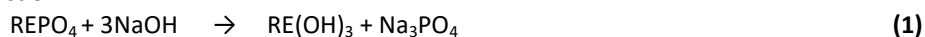
Solvent extraction has demonstrated to be the leading strategy for separating yttrium from the rare earth (REE) chemical complex and is the only strategy currently utilized on an industrial scale that can recover yttrium oxide with a purity of up to 99.99%. Liquid-liquid extraction is additionally known as solvent extraction as well as solid-liquid extraction. Both sorts of solvent extraction are based on the same rule, the separation of

REEs based on their relative solubility in two different immiscible liquids or the solid compound. Illustrations of the utilized solvents are Cyanex 923, bis(2,4,4-trimethylpentyl) dithiophosphinic acid, trialkyl phosphine oxide, CA12, Cyanex 527, tributylphosphate, TOA, TOPO and D2EHPA (22-27).

The show work is directed to considering the separation of Y from rare earths concentrate that has been gotten by chemical processing of xenotime - bearing ferruginous sandstone, southwestern Sinai using conc. H_2SO_4. The REEs concentrate is dissolved with HCl to prepare $RECl_3$ solution. From the previous solution, yttrium is recovered by means solvent extraction technique using cyanex 272 & PC88A in modified kerosene and the optimum conditions have been examined.

II. EXPERMENTAL

2.1. Material Characterization

A working rare earths concentrate sample was produced from the xenotime-bearing ferruginous sandstone, southwestern Sinai. The latter is chemically treated with conc. sulfuric acid at 1/2.5 ore/acid ratio and the dissolution temperature of 250^0C for 3 h for nearly complete dissolution of the rare earth elements. This is followed by precipitation of REEs as oxalates and ignited at 850^0C for 1h to get on RE_2O_3 which known as rare earth concentrate [13].

2.2. Experimental Procedures

2.2.1. Preparation of the chloride liquor of REEs concentrate

10 g of sample portion of the rare earths concentrate (RE_2O_3) which produced from xenotime-bearing ferruginous sandstone, southwestern Sinai as mentioned above is completely dissolved in 15ml diluted hydrochloric acid with continuous heating. The obtained $RECl_3$ liquor is attain up to volume 1000 ml by addition of dist. H_2O and analyzed as shown in table **(1)** using ICP-MS in the Chemical Warefare of Egypt. The latter indicated that that concentrations of rare earths amounted to 3.79, 0.725, 0.520, 0.49, 0.43, 0.34, 0.24, 0.14 and 0.015 g/l for Y, Dy, Gd, Sm, Tb, Yb, Er, Ho and Lu respectively.

Table (1): Chemical composition of the prepared $RECl_3$ solution.

Metal ion	Conc., g/l	Metal ion	Conc., g/l
Y	3.790	**Yb**	0.340
Dy	0.725	**Er**	0.240
Gd	0.520	**Ho**	0.140
Sm	0.490	**Lu**	0.015
Tb	0.430	**pH**	1.6

2.2.2. Yttrium Recovery Procedure

For extraction of yttrium from the prepared rare earths chloride liquor, solvent extraction technique is applied using the synergistic system of Cyanex 272 & PC88A / isodecanol in kerosene as diluent to prepare different concentrations thereof. Isodctanol ($C_{10}H_{22}O$) was used as modifier to avoid the third phase formation and progress the extractants solubility during the extraction of Y. Cyanex 272 solvent is known as a purified bis(2.4.4-trimethylpentyl) phosphinic acid and PC88A solvent is also known as a purified 2-Ethylhexyl 2-ethylhexy phosphonic acid (HEHEHP).

In each extraction test, the organic phase (cyanex 272 & PC88A) and the aqueous phase (prepared yttrium chloride) were shaken together in a separating funnel for a certain time. The two phases were at that point permitted to settle, separated and an aliquot of the aqueous phase was analyzed for its remaining metal content in order to calculate the extraction efficiency. The latter is communicated as the ratio of extracted yttrium and the total amount of yttrium in a certain sample. The relevant solvent extraction factors have hence been studied; specifically the concentration of solvent cyanex 272 & PC88A alone or together, the phase modifier concentration, the contact time and the pH of the chloride liquor as well as the aqueous / organic phase ratio (A/O). On the other hand, a loaded solvent sample was prepared for examining the stripping behavior of the extracted yttrium value using different reagents. Therefore, the relevant factors have also been studied; to be specific the contact time and the concentration of reagent.

2.3. Control Analysis

While conducting series of extraction and stripping experiments, yttrium was analyzed in several different working solutions using the ICP-MS apparatus in the Egyptian Chemical Warfare. Also, the obtained yttrium precipitate at the end was analyzed with the same apparatus.

III. RESULTS AND DISCUSSION

3.1. Results of Yttrium Recovery

3.1.1. Optimization of the Relevant Factors of Yttrium Extraction

Several Factors are studied for extraction of yttrium from the prepared REEs chloride solution namely; cyanex 272 and PC88A concentrations alone and together, phase modifier, pH of the prepared chloride solution and contact time.

3.1.1.1. Effect of Cyanex 272 Concentration

A series of extraction tests were conducted to extract yttrium from a solution of prepared rare earth element chlorides using various concentrations of cyanex 272 from 0.5 M to 2.5 M in kerosene. This was carried out by mixing equal volumes of cyanex 272 as solvent with the prepared chloride liquor for contact time 4 min., at pH 1.6 in presence of 0.1 M isodecanol as a modifier and the obtained data tabulated in table **(2)**.

Table (2): Effect of cyanex 272 concentration on Y extraction efficiency from the prepared chloride liquor.

Cyanex 272 Conc., M	Extraction efficiency of Y,%
0.5	17.15
1.0	26.92
1.5	39.06
2.0	47.23
2.5	40.11

From the show results, it is observed that extraction efficiency of yttrium has increased from 17.15 to 47.23 % when the cyanex 272 concentration was increased from 0.5 to 2.0 M respectively. With increasing the concentration of cyanex 272 at 2.5, the efficiency extraction of yttrium reduced to attain 40.11 % because with a higher cyanex concentration, the movement of yttrium ion from the aqueous phase into organic phase becomes very difficult which can be clarified by the Stokes-Einstein equation. Diffusion is inversely proportional to the solvent viscosity according to the Stokes - Einstein equation, and with greater solvent viscosity, diffusion from the aqueous phase into the organic phase becomes very difficult, thereby decreasing the yttrium extraction efficiency [28,29]. The reaction mechanism of yttrium chloride with cyanex 272 (bis(2,4,4-trimethylpentyl)phosphinic acid) is represented in **figure (1).**

Cyanex 272

Where R_1 = 2,2,4- trimethylpentyl= $CH_3(CH_3)_2CCH_2CH(CH_3)CH_2$-
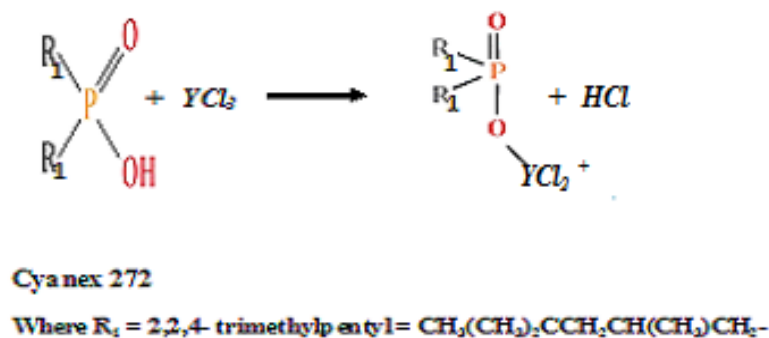
Figure (1): The mechanism reaction of yttrium chloride with cyanex 272

Finally, it is determined that the best cyanex 272 concentration for extracting yttrium from a solution of rare earth element chlorides was 2.0 M. Appropriately, this solvent (cyanex 272) alone was considered not appropriate for extracting yttrium from the prepared chloride liquor of the working rare earths concentrate since it does not accomplish the required result.

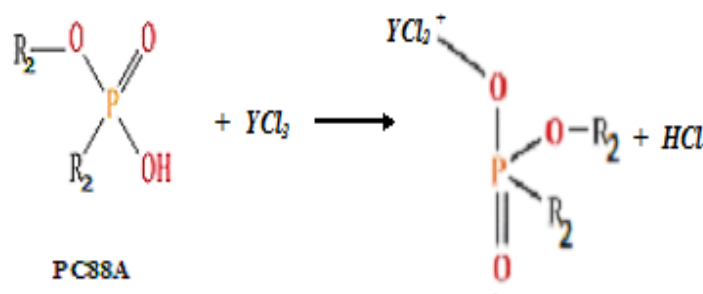### 3.1.1.2. Effect of PC88A Concentration

A sequence of experiments was examined for extraction the yttrium from the prepared solution of rare earth chlorides depending on different concentrations of PC88A from 0.4 M to 2.8 M in kerosene. This was done by mixing equal volumes of the solvent (PC88A) with the prepared liquor (RECl$_3$) for contact time 4 min., at pH 1.6 with expansion of 0.1 M isodecanol as a modifier.

From the gotten data appeared in table **(3)**, it is taken note that yttrium extraction efficiency has increased from 13.2 to 53.83 % with increasing the PC88A concentration from 0.4 to 2.0 M respectively. But at 2.4 & 2.8 M PC88A concentrations, the yttrium extraction efficiencies decreased to 49.08 & 40.11 % respectively because of the extraordinary trouble of the solute diffusing from the aqueous phase to the

organic phase and the viscosity of the solvent as mentioned previously (30,31). The reaction mechanism of yttrium chloride with PC88A (2-Ethylhexyl 2-ethylhexyphosphonic acid) is represented in figure (2). So, it is chosen that the best PC88A concentration for extracting yttrium from the rare earth element chloride liquor was 2.0 M. As a result, PC88A as a solvent alone was considered not reasonable for extracting yttrium from the prepared chloride liquor of the working rare earths concentrate since it does not have good extraction efficiency.

Table (3): Effect of PC88A concentration on Y extraction efficiency from the prepared chloride liquor

PC88A Conc., M	Extraction efficiency of Y,%
0.4	13.20
0.8	24.81
1.2	34.31
1.6	46.18
2.0	53.83
2.4	49.08
2.8	40.11

Where R_2 = 2-Ethylhexyl = $CH_3CH_2CH_2CH_2CH(C_2H_5)CH_2$-

Figure (2): The mechanism reaction of yttrium chloride with PC88A

3.1.1.3. Effect of Mixture of Cyanex 272 & PC88A Concentrations

The effect of mixture of cyanex 272 & PC88A concentrations on Y extraction efficiency was studied by contacting equal volumes of the prepared chloride liquor with a mixture of the two previous solvents of different concentrations extending from 0.4 to 1.8 M of cyanex 272 and 0.6 to 2.1 M of PC88A in kerosene and assaying 0.1M isodecanol for contact time 4 min., at pH 1.6. The obtained data are given in table **(4)**.

Table (4): Effect of mixture of Cyanex 272 and PC88A concentrations on Y extraction efficiency from the prepared chloride liquor

O/A Ratio	Cyanex 272 Conc., M	PC88A Conc., M	Extraction efficiency of Y,%
1/1	0.4	0.6	25.07
	0.7	1.0	42.48
	1.1	1.5	65.43
	1.4	1.8	78.36
	1.7	2.0	66.23
	1.8	2.1	54.09

The results showed that extraction efficiency of Y has increased from 25.07 to 78.36 % when the mixture of cyanex 272 & PC88A concentrations was increased from 0.4 to 1.4 M and from 0.6 to 1.8 M respectively because higher the concentration of mixture of two solvents specified over, the more ion exchange with Y^{+3} will happen. Increasing mixture concentration will increase the number phosphinic and phosphonic acids molecules in the organic phase and the number of POOH groups in the solution, so that the bound Y^{+3} will increase and the extraction efficiency of yttrium will also increase (32). The reaction mechanism of the formed Y complex with mixture of low concentrations of cyanex 272 and PC88A is outlined in figure **(3)**.
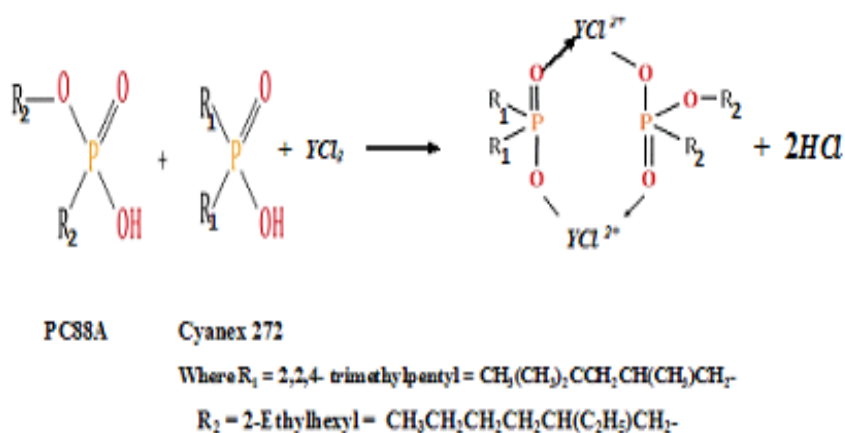
PC88A   Cyanex 272

Where R_1 = 2,2,4- trimethylpentyl = $CH_3(CH_3)_2CCH_2CH(CH_3)CH_2$-

R_2 = 2-Ethylhexyl = $CH_3CH_2CH_2CH_2CH(C_2H_5)CH_2$-

Figure (3): The mechanism reaction of the formed Y complex with mixture of low concentrations of cyanex 272 and PC88A

With continuing to increase the concentrations of cyanex 272 and PC88A over 1.4 and 1.8 M, the extraction efficiency of yttrium diminished since the phosphinic and phosphonic acids compounds form dimers in the solution. The formation of dimers is actuated by the presence of hydrogen bonds and causes hindrance to the solvent in performing its role, which is its binding to the yttrium ion that hindering the solvent occurs at high concentrations of the aforementioned mixture of solvents, and thus reduces the efficiency of yttrium extraction [33]. The reaction mechanism of the formed dimer structure with mixture of high concentrations of cyanex 272 and PC88A is illustrated in figure **(4).** So, mixture of 1.4 M cyanex 272 & 1.8 M PC88A was thus considered as the optimum mixture concentration for yttrium extraction from the prepared chloride liquor of the working rare earths concentrate and better than cyanex 272 and PC88A alone.
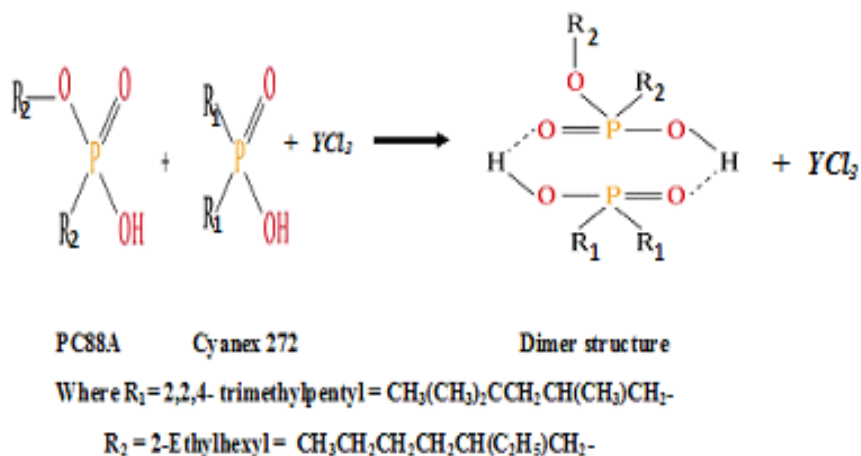

PC88A   Cyanex 272   Dimer structure

Where R_1 = 2,2,4- trimethylpentyl = $CH_3(CH_3)_2CCH_2CH(CH_3)CH_2$-

R_2 = 2-Ethylhexyl = $CH_3CH_2CH_2CH_2CH(C_2H_5)CH_2$-

Figure (4): The mechanism reaction of the formed dimer structure with mixture of high concentrations of cyanex 272 and PC88A

3.1.1.4. Effect of Phase Modifier

Phase modifier aims to increase the solubility of mixture of cyanex 272 & PC88A and avoids the formation of the third phase. So, a few tests were conducted to extract of yttrium from the prepared $RECl_3$ liquor using different concentrations of isodecanol (phase modifier) ranging from 0.05 to 0.3 M. The settled conditions are 1/1 organic/aqueous phases and mixture of 1.4 M cyanex 272 & 1.8 M PC88A at pH 1.6 for contact time 4 min.

From the obtained results in table **(5)**, when the isodecanol concentration increased from 0.05 to 0.25 M, the extraction efficiency of yttrium increased from 60.95 to 83.11 %. With increasing the isodecanol concentration to 0.3 M, the yttrium extraction efficiency does not have a recognizable increase. Subsequently, it was chosen that 0.25 M isodecanol would be the finest concentration to improve the efficiency of the mixture of cyanex 272 & PC88A together, prevent the formation of a third phase, and increase the extraction efficiency of yttrium from the prepared chloride solution.

Table (5): Effect of Isodecanol concentration on Y extraction efficiency from the prepared chloride liquor

Isodecanol Conc., M	Extraction efficiency of Y,%
0.05	60.95
0.10	78.36
0.15	79.42
0.20	81.00
0.25	83.11
0.30	83.64

3.1.1.5. Effect of pH of the Prepared Liquor

To study the impact of pH of the prepared chloride liquor upon extraction efficiency of yttrium and accomplish the optimum condition, seven tests were performed at different pH values ranging from pH 0.5 to 3.5. The surrounding conditions are represented at an A/O of 1:1 using mixture of 1.4 M cyanex 272 & 1.8 M PC88A in kerosene and 0.25 M isodecanol as a modifier for contact time 4 min.

Table (6): Effect of pH of the chloride leach liquor on the extraction efficiency of Y.

pH	Extraction efficiency of Y, %
0.5	53.03
1.0	62.27
1.6	83.11
2.0	85.23
2.5	87.08
3.0	89.19
3.5	89.45

The obtained data in **table (6)** appears that the better extraction efficiency of yttrium was accomplished by increasing the pH values from 1.6 to 3.0 which attained 83.11 to 89.19 % respectively because the atomic radius of Y is small, the interaction of ion exchange from the solute and the solvent to form complex will proceed better as atomic radius of Y is 180 [34]. As the pH value increases to 3.5, the yttrium extraction efficiency increases non-significantly. At low pH values of 0.5 and 1.0, the noticeable decrease in Y extraction efficiency is due to protonation in the solvent that makes extraction hard to ionize and bind to Y^{3+} ions in the aqueous phase [35]. So, the optimum extraction efficiency of yttrium was achieved at pH 3.

3.1.1.6. Effect of Contact Time

To study the impact of contact time upon the extraction efficiency of Y by mixture of cyanex 272 & PC88A, several experiments have been inspected at different contact time periods extending from 4 to 12 min., whereas settling the other extraction factors at an O/A of 1:1 using mixture of 1.4 M cyanex 272 & 1.8 M PC88A in kerosene and 0.25 M isodecanol as a modifier at pH 3.0. The gotten data are organized in table **(7)**. The last mentioned shown that with increasing the contact time from 4 to 12 min., the Y extraction efficiency will increase from 89.19 to 99.47% respectively as a result of the increase in the mass transfer from the aqueous phase to the organic phase, as increases the Y^{3+} extracted into the organic phase which will result in an increase in the yttrium extraction efficiency [36]. So, the contact time of 12 min., was hence considered as the optimum value for almost complete extraction of Y from the prepared $RECl_3$ leach liquor.

Table (7): Effect of contact time on yttrium extraction efficiency from the prepared chloride liquor

Contact Time, min.	Extraction efficiency of Y, %
4	89.19
6	91.30
8	94.99
10	97.10
12	99.47

3.1.1.7. Construction of McCabe-Thiele Diagram of Yttrium Extraction

From the prepared chloride liquor, the influence of the aqueous phase / organic phase ratio (v/v) on the Y extraction was conducted from 3:1 to 1:3 while the other extraction factors were fixed at their assaying optimum values (mixture of 1.4 M cyanex 272 & 1.8 M PC88A in kerosene, 0.25 M isodecanol, contact time 12

min., at 25 ^{o}C and the aqueous phase pH value 3.0). The resulting equilibrium data were used to create a McCabe-Thiele extraction diagram (figure **5**). From this diagram, it is possible to determine the necessary theoretical number of extraction stages by drawing a specific operating line. Depending on the slope of the selected operating line reaching a value 4 (A/O), 3 theoretical stages would be needed to almost complete extraction of Y from the chloride leach liquor prepared from the rare earths concentrate.
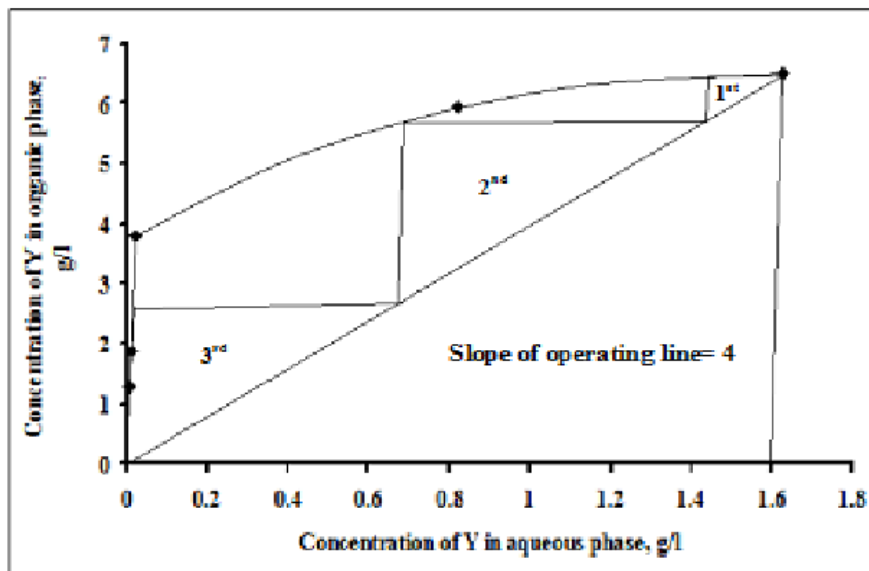

Figure (5): McCabe-Thiele diagram for Y extraction

3.1.2. Optimization of the Relevant Factors of Yttrium Stripping

To strip yttrium, liquor containing cyanex 272 & PC88A/kerosene with a content of 3.77g Y/l prepared and processed with various reagents of acids and alkalis under different tests conditions. These included the concentration of reagent, the contact time and various A/O ratios.

3.1.2.1. Effect of reagent type

Different reagents of acids and alkali were examined for stripping of yttrium from loaded mixture cyanex 272 & PC88A/0.25 M isodecanol in kerosene. The fixed conditions involved 5 min., as contact time and A/O 1/1. From the obtained results in table **(8)**, it is clear that 0.5 M HNO_3 is the best reagent for stripping of yttrium.

Table (8): Effect of different reagents on stripping efficiency of Y

Reagent type, M	Stripping efficiency of Y, %
0.5 M H_2SO_4	12.47
0.5 M HCl	15.39
0.5 M HNO_3	22.29
0.5 M Na_2CO_3	5.31

3.1.2.2. Effect of Acid Concentration

To strip Y, a grouping of stripping experiments were performed using HNO_3 solution of various concentrations from 0.5 to 3.5 M with contact time of 5 min., and using 1/1 O/A ratio at 25 ^{o}C. The results obtained are summarized in table **(9)**, which it is evident that 3 M HNO_3 gave a Y stripping efficiency of 81.17 % and remained almost constant since then.

Table (9): Effect of HNO_3 concentration on stripping efficiency of Y

HNO_3 Conc., M	Y Stripping efficiency ,%
0.5	22.29
1.0	33.16
1.5	48.02
2.0	59.16
2.5	71.09
3	81.17
3.5	81.44

3.1.2.3. Effect of Stripping Time

The influence of the stripping time on the stripping efficiency of Y was investigated from 5 to 25 min. at 3 M nitric acid and 1/1 O/A ratio. From the results presented in table **(10)**, it was observed that 25 min., is sufficient stripping time in which the stripping efficiency exceeds 99%.

Table (10): Effect of stripping time upon Y stripping efficiency

Stripping Time, min.	Y Stripping efficiency ,%
5	81.17
10	88.33
15	93.11
20	96.03
25	99.47

3.1.2.4. Construction of McCabe-Thiele Diagram of Yttrium Stripping

From the obtained equilibrium stripping data under different A/O ratios and using 3 M HNO_3 solution for a contact time 25 min., the corresponding McCabe-Thiele diagram was generated, illustrated in **figure (6)**. From the latter, the theoretical number of stripping stages can be derived after adjusting the working line to the isotherm, whose slope reaches 0.25 (A/O ratio). Therefore, theoretically three stripping stages would be required for nearly complete stripping of yttrium.
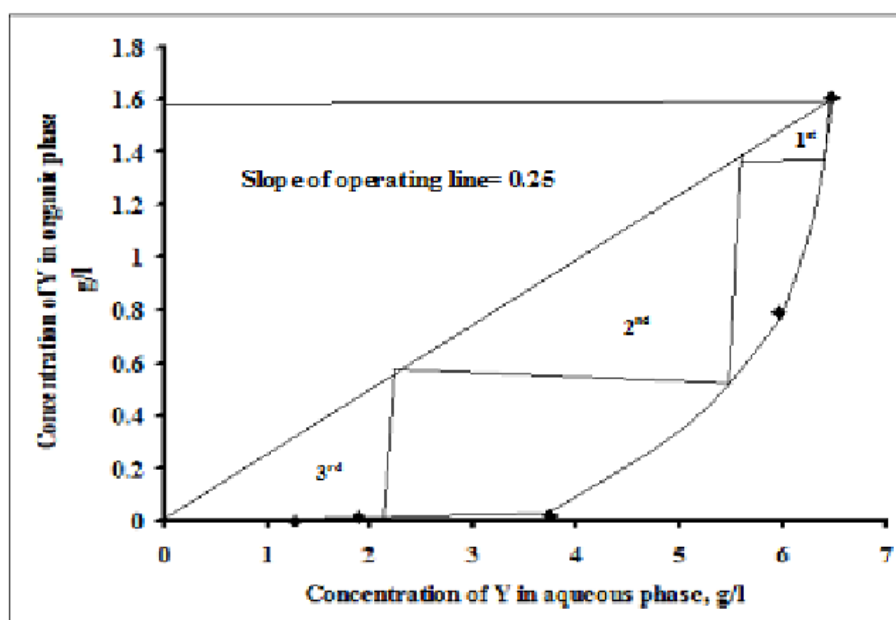
Figure (6): McCabe-Thiele diagram for Y stripping

3.1.3. Y_2O_3 Precipitation

From the yttrium strip solution, Y is completely precipitated with 20% oxalic acid at pH 0.9 as yttrium oxalate [$Y_2(C_2O_4)_3$] according to the represented reaction in **Eq. (7)**.

$$Y(NO_3)_3 + 3 C_2O_4H_2 \rightarrow Y_2(C_2O_4)_3 + 6 HNO_3 \qquad \textbf{(7)} = 2$$
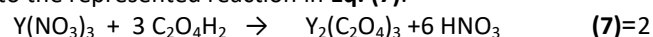
After ignition at 850 ^{o}C, very pure Y_2O_3 was produced as represented in **Eq**. **8** and subjected to a analysis using ICP-MS as shown in table **(11)**.
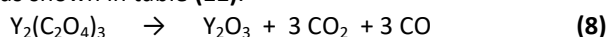
$$Y_2(C_2O_4)_3 \rightarrow Y_2O_3 + 3 CO_2 + 3 CO \qquad \textbf{(8)}$$

Table (11): ICP-MS analysis of the final yttrium precipitate and its impurities

Element	Y	Dy	Al	Na	K
Content,%	76.123	0.0050	0.0125	0.0252	0.0008

V. CONCLUSION

The solvent extraction procedure using mixture of cyanex 272 & PC88A / isodecanol in kerosene was done for extraction of yttrium from the prepared chloride liquor of rare earths concentrate. The decided optimum conditions of yttrium extraction included mixture of concentrations of extractants (1.4 M cyanex 272 & 1.8 M

PC88A) in kerosene, 0.25 M modifier (isodecanol) concentration, 1/1 O/A ratio with the chloride solution pH 3 and 12 min contact time. Stripping of the extracted yttrium was achieved by 3 M nitric acid in 1/1 O/A ratio for 25 min. According to the relevant McCabe Thiele diagrams, three theoretical stages are required for extraction and stripping. Yttrium was precipitated from the stripped solution as yttrium oxalate at pH of 0.9 using 20% oxalic acid solution. The Y oxalates obtained was then ignited at 850 °C for one hour. The obtained oxide was analyzed by ICP-MS. Finally, all the obtained data were meaningfully formulated in a generalized flowsheet (figure **7**).
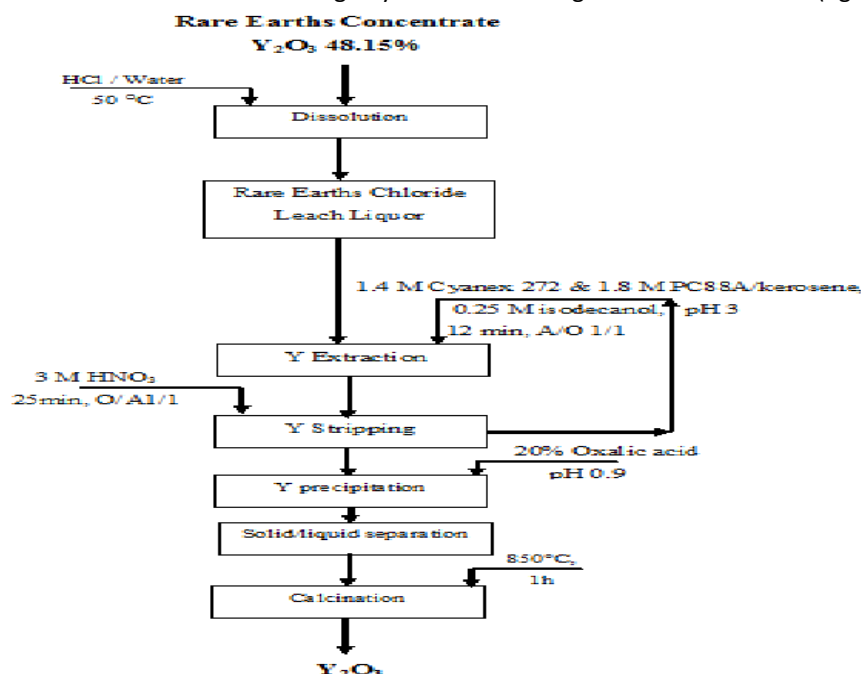
Figure (7): Generalized flowsheet for extraction of yttrium from the rare earths concentrate

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