Extraction of Yttrium from Nd Hydroxide concentrate by using D2EHPA

Suyanti and R A Amiliana
Center for Accelerator Science and Technology (CAST)
National Nuclear Energy Agency, Yogyakarta, 55281, Indonesia
E-mail: suyanti@batan.go.id

Abstract. Extraction of yttrium from Nd hydroxide concentrate by using Di-(2-ethylhexyl) phosphoric acid (D2EHPA) has been performed. Nd hidroxide concentrate (Nd(OH)₃) is a product of rare earth hydroxide (REOH) pilot plant in the Center for Accelerator Science and Technology, BATAN Yogyakarta, which still contains other rare earth metal elements, such as yttrium (Y). In the extraction process, the aquatic phase is Nd(OH)₃ concentrate containing yttrium in HNO₃ and as extractant or organic phase is D2EHPA in kerosene. The parameters studied were the concentration of HNO₃, the concentration of D2EHPA in kerosene, and stirring time. The result of the optimization process from the research get the following conclusions: the optimum concentration of HNO₃ is 1 M, the concentration of D2EHPA in kerosene is 30%, and the optimum stirring time is 25 minutes. In this condition the Kd of Y = 0.979; extraction efficiency of Y = 93.28%; SF Y-Pr, Y-Nd, and Y-Sm are infinite with SF Y-Gd = 6.080 and SF Y-Dy = 1.395. The results of this study are expected to be used as feed on ion exchange columns to separation and purification of other LTJ elements.

1. Introduction
Yttrium (Y) is a transition metal IIIB which belongs to the rare earth group (REE) because of its similar chemical and physical characteristic to other rare earth elements [1]. Even though yttrium has lighter atomic weight compared to some other rare earth elements, yttrium is classified in heavy rare earth element group (HREE) because of the similarity of chemical properties [2]. Yttrium is almost always found with other rare earth metal minerals and never exists in nature as a free element [3].

Yttrium is widely used in ceramics, laser crystal, and superconductor industries, and also in manufacturing devices of visual displays [4][5],[6]. Yttrium has been used in certain commercial metal alloys to improve it’s mechanical properties, like increasing hardness, and it is usually applied in the material that works at high temperatures [7]. In 2009, Susita et.al. has been successful in implanting yttrium ions on TiAl alloys to inhibit the oxidation rate at 800 °C [8].

The large use of yttrium for industrial development has led to increase in the market demand of purified yttrium compounds [9]. In 2014, the estimated supply of yttrium metal was 11,750 tons, while the need of yttrium metal in 2019 was 12,100 tons. Based on fact, there will be 350 tons of yttrium metal deficit which can be an opportunity to develop the yttrium metal processing plant [10]. According to the United States Department of Energy, yttrium is included in five most critical rare earth metal elements that needed for industrial development, along with Neodymium (Ne), Dysprosium (Dy), Europium (Eu), and Terbium (Tb) [1]. Therefore, the separation of yttrium element from other rare earth metal elements is urgent to be done, considering the demand for this metal for industrial development.
Nd hydroxide (Nd(OH)_3) concentrate as a product of the RE-Hydroxide pilot plant in BATAN Yogyakarta still contains other rare earth metal elements, including yttrium. The yttrium content in Nd(OH)_3 concentrate is around 10% [4], but it can affect the magnetic properties of Nd and also influence the selling price of Nd(OH)_3 concentrate. On the other hand, some other rare earth elements contained in the Nd(OH)_3 concentrate, such as Pr, Sm, Gd and Dy, can improve the magnetic properties of the Nd metal. Therefore the separation of yttrium from Nd(OH)_3 concentrate is very urgent because of its high demand for yttrium metals in the industrial world and the element separation can also increase the selling price of the Nd(OH)_3 concentrate product.

Rare earth elements are difficult to be separated from each other because of their similar physical and chemical properties. The separation process based on ion exchange and solvent extraction techniques has been developed to obtain high purity rare earth elements. Even so, the solvent extraction process is more widely used as a commercial technology because of its easier and can be applied on a large scale [11]. Solvent extraction is a separation of a particular substance from a mixture by dissolving the substance in a solvent without dissolving any other substance in the mixture. Research on the extraction of yttrium from a mixture of rare earth metals is still being undertaken and developed because the yttrium extraction process also shows different results for each different system [12].

The use of D2EHPA as yttrium extractant is quite extensive on a commercial scale because it has high selectivity and efficiency to separate yttrium from other compounds [13]. The D2EHPA of Di-(2-Ethylhexyl)phosphoric acid is an organophosphorus compound that has one H atom that can be replaced by metal ions, so these compounds are commonly called a ion-exchange compound. The D2EHPA has also a wide and different distribution coefficient for each rare earth metal element, so it can be separated from other elements in the solution [11]. Organic solvents such as D2EHPA generally have high density and viscosity, which causes the difficulty in transferring from the aquatic phase to the organic phase, so the organic phase viscosity has to be reduced by adding a diluent. One of the commonly used organic diluents is kerosene [4].

In this study, the yttrium will be extracted from Nd hydroxide concentrate (Nd(OH)_3) as the product of a rare-earth hydroxide (REOH) pilot plant. As an aquatic phase is Nd(OH)_3 concentrate containing yttrium in HNO_3 and as extractant or organic phase is Di-(2-Ethylhexyl)phosphoric acid (D2EHPA) in kerosene. This paper reports the result of the study on extraction variation of the concentration of HNO_3 in feed, the D2EHPA concentration in kerosene, and the stirring time.

2. Experimental method

2.1. Materials and reagents

Neodymium hydroxide concentrate (Nd(OH)_3) is a product of rare-earth hydroxide (REOH) pilot plant in CAST BATAN, Yogyakarta. The dissolution of feed was using nitric acid (HNO_3) in the aquatic phase. The D2EHPA was used as extractant and it dissolved in kerosene in the organic phase. The analysis of the products of the process was carried out using X-ray fluorescence spectroscopy (XRF) Ortec 7010 with the uncertainty of the analysis results is 5%.

2.2. The dissolving of Nd(OH)_3 concentrate

Nd hydroxide concentrate was dissolved in the aquatic phase using nitric acid as a feed of the extraction process. The concentration of nitric acid was varied in 1 M; 1.5 M; 2 M; 2.5 M; and 3 M. The results of dissolution then analyzed by using XRF. The best HNO_3 concentration which shown the best extraction parameters will be used to the extraction stage.

2.3. Extraction by using D2EHPA

The volume ratio of feed and extractant was 1:1. The D2EHPA as extractant was dissolved in kerosene. The concentration of D2EHPA in kerosene was studied at a range of 10% - 30%. The stirring speed was 250 rpm with the variation of stirring time between 10 – 30 minutes. After the extraction process, the aquatic and the organic phases were separated using a volume pipette, then the aquatic phase was
analyzed by using an XRF (Ortec 7010) to determine the content of elements that were not extracted by extractant.

3. Result and Discussion

The extraction parameters used are distribution coefficient, separation factor of yttrium from other rare earth elements, and efficiency of yttrium extraction. The distribution coefficient refers to Nernst distribution basic law, i.e. when a solute is taken up with two immiscible liquids, in both of which the solute is soluble, the solute distribute itself between the two liquids in such a way that the concentration ratio in the two liquid phases will constant at a given temperature [14]. The distribution coefficient is expressed as:

\[ K_d = \frac{C_o}{C_a} \]  

where \( K_d \) is the distribution coefficient, \( C_o \) is the solute concentration in the organic phase, and \( C_a \) is the solute concentration in the aquatic phase. From that equations, if \( K_d \) increase, the solute quantitatively will tend to be distributed more into the organic phase, and so does the opposite [15].

The separation factor (SF) is also a parameter to be used to evaluate the extraction process. The separation factor shows the comparison between a solute distribution with other solute distribution during extraction [16]. The equation for calculating the separation factor is:

\[ SF = \frac{K_{d1}}{K_{d2}} \]  

where \( K_{d1} \) is the distribution coefficient of the first element and \( K_{d2} \) is the distribution coefficient of the second element. The efficiency in the extraction process can be expressed by the percentage of solute extracted which can be calculated by the following equation:

\[ E = \frac{C_o}{F} \times 100\% \]  

where \( E \) is extraction efficiency (%), \( C_o \) is the solute concentration in the organic phase, and \( F \) is feed concentration to extraction.

3.1. Effect of HNO₃ Concentration in Feed

Feed in the form of Nd(OH)₃ concentrate powder was dissolved in nitric acid with varying concentration, i.e. 1 M, 1.5 M, 2 M, 2.5 M, and 3 M. The effect of the changes of the concentration of HNO₃ on yttrium concentration in raffinate can be seen in Figure 1 with the initial content of Y in the feed was 8966.658 ppm.

![Figure 1. Effect of HNO₃ concentration to Y element content in raffinate](image-url)

\[ y = 2601.4\ln(x) + 4952.9 \]

\[ R^2 = 0.8406 \]
From Figure 1 it can be seen that the higher HNO$_3$ concentration makes the yttrium content in raffinate or in aquatic phase increase, which shows that the higher HNO$_3$ concentration, the less yttrium metal is extracted to the organic phase. This happens because by increasing the concentration of HNO$_3$ will increase the formation of H$^+$ ions, so the reaction will shift to the left. The reaction that occurs between HNO$_3$ and yttrium metal can be seen in these following equation:

$$
HNO_3 \rightleftharpoons H^+ + NO_3^- \quad (4)
$$

$$
Y(OH)_3 + 3HNO_3 \rightleftharpoons Y(NO_3)_3 + 3H_2O \quad (5)
$$

In addition, the presence of excess HNO$_3$ will dissolve the D2EHPA and cause a decrease in the D2EHPA amount that reacts with metals. Thus, the higher concentration of HNO$_3$ in the feed will reduce the rate of element transfer to the organic phase and cause a decrease of distribution coefficient value ($K_d$), as can be seen in Table 1.

**Table 1. Effect of HNO$_3$ concentration in feed to the distribution coefficient ($K_d$)**

| HNO$_3$ Concentration | Y   | Pr  | Nd  | Sm  | Gd  | Dy |
|-----------------------|-----|-----|-----|-----|-----|-----|
| 1 M                   | 0.519 | 0   | 0   | 0.102 | 0.060 | 0   |
| 1.5 M                 | 0.246 | 0.101 | 0   | 0   | 0.099 | 0   |
| 2.0 M                 | 0.187 | 0.021 | 0   | 0   | 0   | 0   |
| 2.5 M                 | 0.172 | 0.049 | 0   | 0.050 | 0   | 0   |
| 3.0 M                 | 0.149 | 0.044 | 0   | 0.115 | 0   | 0.021 |

From Table 1, it can be seen that the yttrium element has the highest distribution coefficient compared to other elements, especially at 1 M concentration of HNO$_3$. The value of 0 in the distribution coefficient calculation means that the element was not extracted into the organic phase. The use of D2EHPA as an extractant will cause more yttrium extracted to organic phase than Pr, Nd, Sm, Gd, and Dy elements, and therefore more easily separated from Nd hydroxide concentrate. The parameter of extraction can also be seen from the extraction efficiency as shown in Figure 2.

![Figure 2. Effect of HNO$_3$ concentration in feed to extraction efficiency of Y](image)

The results of the efficiency calculation of yttrium extraction show that the largest extraction efficiency of yttrium from Nd(OH)$_3$ concentrate is obtained at 1 M HNO$_3$ concentration. The yttrium
efficiency decrease as the increase of HNO₃ concentration as shown in Figure 2. The highest value of yttrium extraction efficiency was 51.94% at 1 M HNO₃ concentration and the lowest at 3 M HNO₃ concentration with a value of 14.93%. The separation factor of yttrium element with other rare earth metal elements has to be considered in determining the optimum condition, as shown in Table 2.

Table 2. Effect of HNO₃ concentration in feed to Y separation factor

| HNO₃ Concentration | Y Separation Factor with Other REE |
|-------------------|----------------------------------|
| 1 M               | Pr ∞ Nd 5.073 Gd 8.726 Dy ∞      |
| 1.5M              | 2.430 ∞ ∞ Gd 2.480 Dy ∞         |
| 2.0 M             | 8.701 ∞ ∞ ∞ ∞                   |
| 2.5 M             | 4.524 ∞ 4.424 ∞ ∞ ∞            |
| 3.0 M             | 3.384 ∞ 1.296 ∞ 6.947           |

The infinite value on the separation factor in Table 2 shows that the Y element can be separated well from these rare earth metal elements. Based on the calculation of the separation factor, the best concentration of HNO₃ to separate Y element from other rare earth elements was 2 M, because only the Pr element that hasn’t been separated completely at that concentration. However, to determine the optimum extraction conditions, other extraction parameters have to be considered, such as distribution coefficient and extraction efficiency. In this variation, the optimum conditions for yttrium extraction that were selected were 1 M acidity of feed, because in these conditions the extraction efficiency and Y distribution coefficient show the highest value with a separation factor Y which was good enough compared to other rare earth elements.

3.2. Effect of D2EHPA Concentration

The variation of D2EHPA concentration has a significant effect on the distribution coefficient and extraction efficiency of the yttrium. The calculation of the yttrium distribution coefficient and other rare earth elements towards Nd can be seen in Table 3.

Table 3. Effect of D2EHPA concentration to distribution coefficient (Kd)

| D2EHPA Concentration | Distribution Coefficient (Kd) Element |
|----------------------|--------------------------------------|
| Y                    | Pr 0.741 Nd 0.059 Sm 0.066 Gd 0.348 Dy 0.389 |
| 10%                  | Pr 0.728 Nd 0.0  Sm 0.0 Gd 0.0 Dy 0.603 |
| 20%                  | Pr 0.883 Nd 0.0 Sm 0.0 Gd 0.029 Dy 0.618 |
| 25%                  | Pr 0.853 Nd 0.0 Sm 0.0 Gd 0.148 Dy 0.731 |
| 30%                  | Pr 0.979 Nd 0.01 Sm 0.019 Gd 0.161 Dy 0.702 |

Based on Table 3, the higher concentration of the D2EHPA for the extraction process gives more yttrium elements that can be separated, marked by the higher value of the yttrium distribution coefficient (Kd). The highest Kd for Y is obtained when the concentration of D2EHPA was 30%, which is equal to 0.979, means that there were quite a number of yttrium element that move to the organic or extracted phase. But in the 30% D2EHPA concentration, other elements, such as Pr, Sm, Gd, Dy, also begin to move to the organic phase. It is predicted that at higher D2EHPA concentration, all element will move to the organic phase and will be more difficult to separate the Y element. The highest value of yttrium extraction efficiency also at 30% D2EHPA concentration, which is equal to 93.28%. The efficiency of yttrium extraction in each variation of D2EHPA concentration can be seen in Figure 3.
The Di-(2-Ethylhexyl)phosphoric acid (D2EHPA) is a strong donor, has one H atom which can be replaced by metal ions, so this compound is commonly called an ion-exchange compound. In addition, the D2EHPA compound has a P = O group which can coordinate with metal ions. The bond structure of D2EHPA can be seen in Figure 4.

The increasing concentration of D2EHPA causes more bonding of metal ions by phosphate groups. It makes more yttrium extracted into the organic phase so that increasing extraction efficiency. However, if the concentration of D2EHPA is too high in the organic phase, it will cause difficulty in the process of transferring solute from the aquatic phase to the organic phase due to the high viscosity of the organic phase solution.

![Structure Formula of D2EHPA](image)

The chemical reaction that occurs between rare earth metals with D2EHPA are as follows:

\[
M^{3+} + 3(H_2X)_2 \rightleftharpoons [M(X)_3(HX)_3] + 3H^+ 
\]  

The separation factor of Y element with other rare earth elements can be seen in Table 4.

### Table 4. Effect of D2EHPA concentration to Y separation factor

| D2EHPA Concentration | Y Separation Factor with Other REE |
|----------------------|-----------------------------------|
|                      | Pr | Nd | Sm  | Gd  | Dy  |
| 10%                  | 12.660 | 11.203 | 2.128 | 1.907 | 0.824 |
| 15%                  | ∞ | ∞ | ∞ | ∞ | 1.207 |
| 20%                  | ∞ | ∞ | ∞ | 30.404 | 1.429 |
| 25%                  | ∞ | ∞ | ∞ | 5.768 | 1.167 |
Based on Table 4, the best separation of Y element with other rare earth elements occurs at 25% of D2EHPA concentration. Even so, the separation of Y from other rare earth elements at 30% concentration of D2EHPA is also quite good. Looking at the parameters of the distribution coefficient and extraction efficiency of Y also shows the highest value at 30% D2EHPA concentration, so that at the next stage D2EHPA concentration of 30% can be established as the optimum extraction condition.

3.3. Effect of Stirring Time
The variation of stirring time shows that as the stirring time longer, the distribution coefficient and yttrium extraction efficiency will be higher. However, after stirring for more than 25 minutes, there is a decrease in the value of extraction efficiency and distribution coefficient. The distribution coefficient of yttrium and other rare earth elements with variations of stirring time can be seen in Table 5.

| Stirring Time (Minutes) | Distribution Coefficient (K_d) Element |
|-------------------------|----------------------------------------|
|                         | Y | Pr | Nd | Sm | Gd | Dy |
| 10                      | 0.741 | 0.059 | 0.066 | 0.348 | 0.389 | 0.9 |
| 15                      | 0.832 | 0 | 0 | 0 | 0 | 0.874 |
| 20                      | 0.838 | 0 | 0 | 0 | 0.388 | 0.973 |
| 25                      | 0.979 | 0 | 0 | 0 | 0.161 | 0.702 |
| 30                      | 0.834 | 0.264 | 0 | 0.27 | 0.498 | 0.918 |

Based on Table 5, the distribution coefficient of the Y element which has the highest value is 0.979 at 25 minutes of stirring time. Similarly, the highest yttrium extraction efficiency was at 25 minutes with a value of 93.28%. The changes in the yttrium extraction efficiency against the variations of stirring time can be seen in Figure 5.

\[ y = -0.0008x^2 + 0.0389x + 0.4307 \]
\[ R^2 = 0.7722 \]

Figure 5. Effect of stirring time to extraction efficiency of Y

The stirring time is related to the contact time between the yttrium ion and the D2EHPA extractant, so that the increase of stirring time causes the increase of K_d and extraction efficiency. As longer contact time between the aquatic phase and the organic phase during the extraction process, the more amount of elements will be bound by D2EHPA. But when a balanced state is reached, the number of extracted elements is no longer influenced by time. The saturated extractant concentration can affect the
equilibrium of the reaction and affect the extracted yttrium ions. These results are also supported by the separation factor value of Y with other rare earth elements, as shown in Table 6.

| Table 6. Effect of stirring time to Y separation factor |
|-------------------------------------------|
| Stirring Time (Minutes) | Pr   | Nd   | Sm   | Gd   | Dy   |
|-------------------------|------|------|------|------|------|
| 10                      | 12.660 | 11.203 | 2.113 | 1.907 | 0.824 |
| 15                      | ∞     | ∞     | ∞     | ∞     | ∞     |
| 20                      | ∞     | ∞     | ∞     | 2.160 | ∞     |
| 25                      | ∞     | ∞     | ∞     | 6.080 | 1.395 |
| 30                      | 3.159 | ∞     | 3.089 | 1.675 | 0.908 |

The best separation of Y element with other rare earth elements based on a separation factor occurs when the stirring time was 25 minutes. At that duration, separation Y with Pr, Nd, and Sm elements can occur very well with the infinite value of SF, even though the separation with the Gd and Dy elements are not too high. Based on the value of the distribution coefficient, extraction efficiency, and separation factor, the optimum stirring time was set at 25 minutes.

It can be concluded that the optimum condition of yttrium extraction with D2EHPA from Nd(OH)₃ concentrate is at the solvent concentration of 1 M HNO₃, extractant concentration of 30% D2EHPA, and stirring time of 25 minutes, so the Y distribution coefficient (Kₒ) value is 0.979, yttrium extraction efficiency is 93.28%, with the separation factor (SF) of Y-Pr, Y-Nd and Y-Sm are infinite, SF Y-Gd = 6.080 and SF Y-Dy = 1.395. The TBP (Tri Butyl Phosphate) has also been used in extraction to separate rare earth elements in Nd(OH)₃ concentrate and the separation factor (SF) of 2.242 for Nd-Y can be obtained with 19.07% of Nd extraction efficiency [4]. This shows that by extraction using TBP, there are still many Nd elements dissolved in the aquatic phase and the separation of yttrium elements with Nd is not as good as when using D2EHPA.

4. Conclusion
The study of yttrium separation from Nd hydroxide concentrate as the results of the rare earth hydroxide (REOH) pilot plant has been undertaken at the Center for Accelerator Science and Technology, BATAN Yogyakarta by extraction using D2EHPA. The optimum extraction conditions are HNO₃ concentration in the extraction feed of 1 M, the D2EHPA concentration in organic solvent of 30%, and the stirring time of 25 minutes. In this condition, the value of the Y distribution coefficient (Kₒ) is 0.979, the efficiency of yttrium extraction reaches 93.28%, with separation factor (SF) of Y-Pr, Y-Nd and Y-Sm are infinite, SF Y-Gd = 6.080 and SF Y-Dy = 1.395. Yttrium extraction from Nd hydroxide concentrate by using D2EHPA gives a better result than using TBP.

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