

## Preliminary Study of Yttrium Extraction from Tin Slag Using Hydrochloric Acid

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### ABSTRACT

Tin slag, a byproduct of tin smelting, comprises several important metals, including yttrium, tantalum, manganese, iron, aluminum, titanium, and others. As an element that is used a lot in advanced materials, the separation of Yttrium becomes one of the most important steps in rare earth elements extraction. By using the alkali fusion process and leaching method, yttrium in the tin slag has been successfully extracted. The tin slag was subjected to an alkali fusion treatment with sodium hydroxide at a temperature of 700 °C, and the frit was ground into several particle sizes of +100 to -325 mesh using a laboratory ball mill and mortar. The frit was then leached using hydrochloric acid, with four parameters used: temperature, acid concentration, particle size, and stirring speed. The extracted yttrium reached a percentage of up to 86.94% at a temperature of 80 °C, acid concentration of 2 M, particle size of -325 mesh, and stirring speed of 150 rpm.

**Keywords:** yttrium, tin slag, alkali fusion, leaching, hydrochloric acid.

### INTRODUCTION

Rare Earth Elements (REE), also called Rare Earth Metals (REM), are a group of elements consisting of 17 chemical elements. These elements consist of 15 lanthanides together with scandium and yttrium [1]. The demand for rare earth elements (REE) is going to increase in many sectors, such as technology, healthcare, and security, over time [2]. The predominant mineral sources of rare earth elements encompass bastnaesite, monazite, and xenotime [3]. There are also secondary sources, such as industrial waste. An example is tin slag, the waste of the tin ore smelting process [4]. REE are commonly categorized into two distinct classes, namely

heavy rare earth elements (HREE) and light rare earth elements (LREE) [5]. One of the HREE that has significant modern value and is in high demand because of its usage is Yttrium (Y).

Yttrium, an element discovered in 1974, has unique physicochemical properties that provide essential resources in various fields, such as high-tech components, environmentally friendly technologies, and the materials industry [6]. Numerous rare earth elements, including Yttrium, have been detected, as evidenced by minerals such as monazite (Ce, La, Y, and Th) PO<sub>4</sub> and xenotime (YPO<sub>4</sub>). The discovery of rare earth elements in China occurred in 1960, with

element Y recognized as the predominant constituent, containing 60% of the content [7]. Cassiterite is used as a material in mining in Bangka and is the primary source of tin production.

Tin slag is formed as a by-product during the smelting of cassiterite for tin extraction. Specifically, tin slag from Bangka is known to contain rare earth elements, including yttrium (Y) [8], [9], [10], [11]. Rare earth elements play a crucial role as fundamental constituents in diverse contemporary technological applications, underscoring the imperative need to comprehend their origins. The availability of tin slag and other sources is sufficient [11], but the main challenge occurs in the efficient extraction process, particularly in the extraction of yttrium. Therefore, it is important to carry out this research with the aim of determining the best conditions for the yttrium leaching process, thereby increasing the effectiveness of the extraction results.

## **THEORY BASES AND PRESENT RESEARCH**

The remains from tin processing are then known as tin slag, which contains radioactive substances with an amorphous solid structure. Then, the tin slag has an operationally sensitive structure, as demonstrated by leaching the tin slag. Tin slag has matrix elements called "pseudo structure" for chemical processing. The presence of this pseudo-structure reflects the operational sensitivity of tin slag, as evidenced by its behavior during the leaching process. [12], [13]. Based on chemical analysis data, low-grade tin slag contains impurities such as silica, alumina, and iron oxide. The chemical process using NaOH has been proven to remove these unwanted elements. In the extraction of Niobium (Nb) and Tantalum (Ta) from tin slag, alkali treatment and acid

leaching are applied sequentially to dissolve unwanted impurities, enriching the residue for further processing[14]. In paper [15], a strong acid solution of hydrofluoric acid (HF) can dissolve silica. Also, in another paper [16], using fluorosilica acid to eliminate silica compounds from tin slag is proven effective with a ratio of  $\text{H}_2\text{SiF}_6:\text{H}_2\text{O} = 2:1$ . Yttrium (Y), included in REE in tin slag after going through an alkaline treatment process using NaOH, is dissolved using an acid solution such as  $\text{H}_2\text{SO}_4$  and HCl [14], [17]. The paper reports that the extraction of uranium and thorium from tin slag is facilitated using strong acids (HF, HCl) and a strong base (NaOH). This process involves the dissolution of BTS-roasting-quenching-sieving (BTS-RQS) residues in a solution containing 8% HF, 6 M HCl, and 10 M NaOH, which enhances the concentration of these elements [18]. Another paper states that strong acids can dissolve rare earth from phosphogypsum and leach efficiently when using nitric acid and hydrochloric acid [19].

The extraction of rare earth metals is also carried out from various other materials. As in the paper [20], the extraction of rare earth metals from bastnaesite mineral uses a hydrochloric acid solution. Then, in another paper [21], xenotime concentrate mineral was extracted by separating LREE from other rare earth elements into three groups using selective washing. In other research [22], the recovery of rare earth from zircon tailings using  $\text{H}_2\text{SO}_4$  showed that the percentage of rare earth recovery increased as the concentration of  $\text{H}_2\text{SO}_4$  increased. The results gained can be linked to the increased solubility of HREEs  $(\text{OH})_3$  in acidic environments, particularly in the presence of strong acids. Furthermore, in the paper [23] it is concluded that the extraction of yttrium can be done from waste fluorescent lamps.

In previous research [24], [25], leaching of uranium, thorium, and REE precipitate using HCl, H<sub>2</sub>SO<sub>4</sub>, and HNO<sub>3</sub> has been conducted. From the research, the precipitate was able to be dissolved by using all 3 types of acid, but the acid consumption of H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> was 2-3 times more than HCl, making leaching using HCl the most efficient [24], [25]. In the leaching process using a hydrochloric acid solution, the leaching rate of rare earth metals is influenced by the concentration of the hydrochloric acid solution used. In the paper [26], the author states that the leaching rate of rare earth metals increases as the concentration of hydrochloric acid increases. Temperature significantly impacts the dissolution process, as the reaction speed increases with temperature [17]. Acid roasting uses high temperatures with sulfuric acid to remove impurity minerals contained in the material [27]. Another study reported that HCl was utilized to extract yttrium from "zircon tailings," achieving a recovery rate of 87% under optimal conditions at a temperature of 60°C [28]. In the paper [29], the author carried out the leaching of rare earth metals using magnesium sulfate solution, saying the smaller the particle size, the greater the mineral's surface area, and the rare earth's leaching rate was increased by exchanging rare earth ions with the same leaching time. Furthermore, another paper stated that particle size can influence the process of leaching rare earth metals using a hydrochloric acid solution by increasing the specific surface area, so that there is more contact with the solvent and speeding up the reaction rate [30]. Comminution or size reduction can increase the surface area of particles. Furthermore, the diffusion process from solid to bulk solution is improved by turbulent stirring in the dissolution process, improving slurry homogenization [31].

## METHODOLOGY

The research was carried out at the Research Center for Nuclear Fuel Cycle and Radioactive Waste Technology. The method for extracting yttrium from tin slag begins with refining the sample particle size, followed by alkali fusion treatment using NaOH solution, and subsequently proceeding with the leaching process using HCl. The result sample from each step was subjected to XRF analysis to figure out the chemical makeup of the sample, including the amount of yttrium in the tin slag and the chemical makeup of the leaching residue in the water used as leaching feed in hydrochloric acid solution.

## Materials

The material used in this experiment is tin slag from Bangka, which will be divided into several sizes. The solutions utilized in this experiment consisted of NaOH, HCl, and water. The samples were examined using X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectroscopy 8300 (ICP-OES).

## Alkali Fusion Treatment

The initial investigation of the tin slag samples commenced by subjecting them to XRF testing to identify their chemical composition. Then, prepare the leaching feed through an alkali fusion process, followed by leaching the obtained product in water. The alkaline fusion process uses a NaOH solution to decompose silica into silicate compounds readily soluble in water. Through a leaching process using NaOH, most of the silica can be removed [32]. This process uses a temperature of around 700 °C with a weight ratio of tin slag: NaOH = 1: 2. Then, the fusion product is leached using water for 60 minutes, filtered, and the residue is washed using water until the pH of the water reaches 9.

After washing, the residue is crushed into several particle sizes, namely 325, 200, 150, and 100 mesh. Next, samples were taken with a size of -325 mesh for analysis using ICP-OES to determine the concentrations of Yttrium metal and other metal impurities. Also, analytical tests are conducted to determine the concentration of Y in different particle sizes.

### Acid Dissolution

After going through the alkali fusion process, the next step is leaching using hydrochloric acid. At this stage, several variations of experiments were carried out, including variations in acid concentration, particle size, temperature, and sample stirring speed.

Table 1. Variations in the experiment

Acid concentration (M)	Particle size (mesh)	Temperature (°C)	Stirring speed (rpm)
0.5	+100	30	100
1	100 - 150	40	150
1.5	150 - 200	50	200
2	200 - 325	60	250
2.5	-325	70	300
2.75		80	350

An approach to experimentation in which one variable or factor is changed while all other factors remain constant. Methods like this aim to determine the optimal conditions for an experiment [33]. The extraction percentage Y is calculated by using the following equation:

$$\%E_y = \frac{[W]_{fi}}{[W]_{fe}} \times 100 \% \quad (1)$$

where  $\%E_y$ ,  $[W]_{fi}$ , and  $[W]_{fe}$  represent yttrium extraction percentage, weight of yttrium in the filtrate, and weight of yttrium in the feed, respectively.

## RESULTS AND DISCUSSION

The elemental composition of tin slag samples analyzed by using XRF is shown in Table 2, and the results of the analysis on the chemical makeup of frit, the residue left behind after leaching in water, are shown in Table 3.

Table 2. Results of XRF analysis before alkali fusion

Si (%)	Y (%)	Fe (%)	Al (%)	Na (%)	Other elements (%)
26.49	1.05	15.02	9.50	0.747	47.10

Table 3. Results of analysis of the chemical composition of frit leaching residue

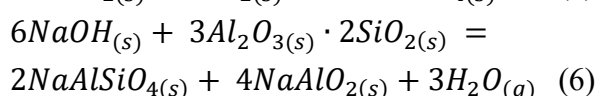
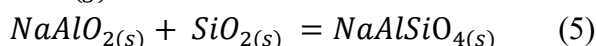
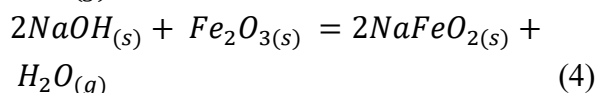
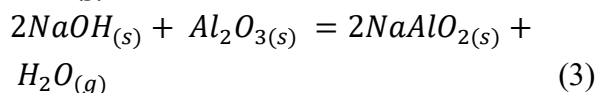
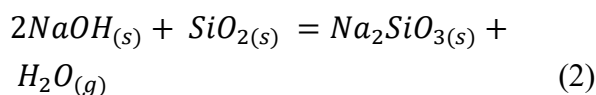
Si (%)	Y (%)	Fe (%)	Al (%)	Na (%)	Other elements (%)
11.98	0.7	6.741	3.30	33.14	43.50

### Effects of Alkali Treatment

The analysis results table before and after the alkali fusion process shows that the percentage of silica has decreased. Previously, it was 26.49% in Table 2. Decomposition of the silica structure in tin slag using NaOH at a temperature of 700 °C produces sodium silicate compounds, which are soluble in water, so that the silica content in the frit results in alkali fusion decreases after water leaching, with a silica percentage of 11.98% in Table 3.

The reaction between NaOH and the primary constituents (Si, Al<sub>2</sub>, and Fe<sub>2</sub>) leads to the formation of water-soluble compounds, resulting in the dissolution of the main solid components. This content then reacts with NaOH, causing the conversion of the crystalline phase i.e. quartz (SiO<sub>2</sub>), corundum (Al<sub>2</sub>O<sub>3</sub>), and mullite (3Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>), and the amorphous phase into soluble forms such as silicate (Na<sub>2</sub>SiO<sub>3</sub>), aluminate (NaAlO<sub>2</sub>), and

aluminosilicate ( $\text{NaAlSiO}_4$ ). These soluble forms can dissolve in water or acid, as shown in the following reaction [34]:



In other research [35], the alkali fusion process using NaOH can decompose elements such as Si, Al, and Ca contained in titanium slag using a temperature range from 500 °C to 700 °C.

### Effects of HCl concentration

Tin slag dissolution experiments with varying concentrations of HCl acid in the range 0.5–2.75 M, while keeping all other parameters constant (temperature 30 °C, particle size -325 mesh, stirring speed 150 rpm). The results are shown in Figure 1.

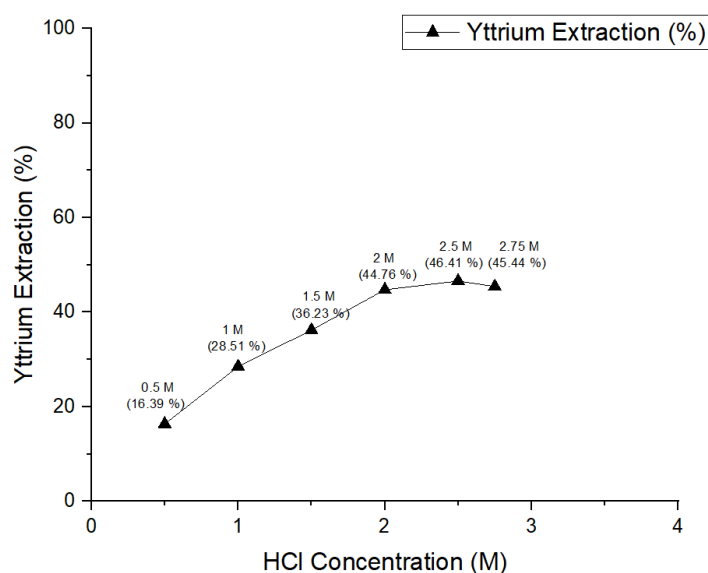
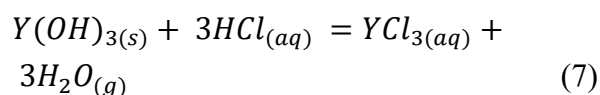


Figure 1. Yttrium extraction at various concentrations

Figure 1 shows the curve effect of changes in HCl concentration on the results of Y extraction. At first, using a concentration of 0.5 M produced the results of Y extraction of 16.39%. As the HCl concentrations of 1 M and 2 M increased, the results of Y extraction increased by 28.51% and 44.76% until it reached the peak point at a concentration of 2.5 M with extraction results of 46.41%, and the results of Yttrium extraction decreased to 45.44% when the acid concentration was added to 2.75 M.

Several studies have shown that strong acids can dissolve the amorphous phase of a mineral, and differences in concentration can affect the percentage of REE mineral extraction [17], [36], [37]. In the alkaline fusion process, tin slag decomposes and produces yttrium in the form of a hydroxide compound. The leaching reaction using a hydrochloric acid reagent is as follows [22]:



It was also reported that dissolving yttrium from "waste ceramic dust", the efficiency of yttrium dissolution increased as the concentration of the HCl acid solution increased and reached its highest point at a HCl concentration of 2 M, with a yttrium dissolution efficiency of 98.6% [38].

### Effects of particle size and temperature

Various factors, including particle size refinement, solvent extraction, and leaching temperature, can influence the percentage extraction of rare earth elements. The leaching rate of rare earth elements can increase with

increasing temperature, but other factors, such as particle size refinement, can also play a role [39], [40]. Therefore, it can be concluded that there is a positive correlation between the leaching rate and the percentage of extraction.

Tin slag dissolution experiment with particle size variations in the range +100 - 300 mesh by keeping all other parameters constant (temperature 30 °C, acid concentration 2M, stirring speed 150 rpm) and temperature variations in the range 30 - 80 °C with constant parameters (acid concentration 2M, stirring speed 150 rpm, particle size -325 mesh) results are shown in Figures 2 and 3.

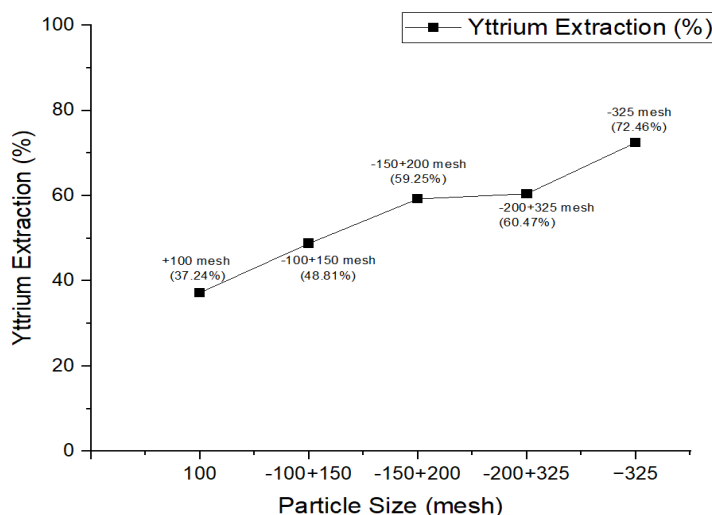


Figure 2. Yttrium extraction at various particle sizes

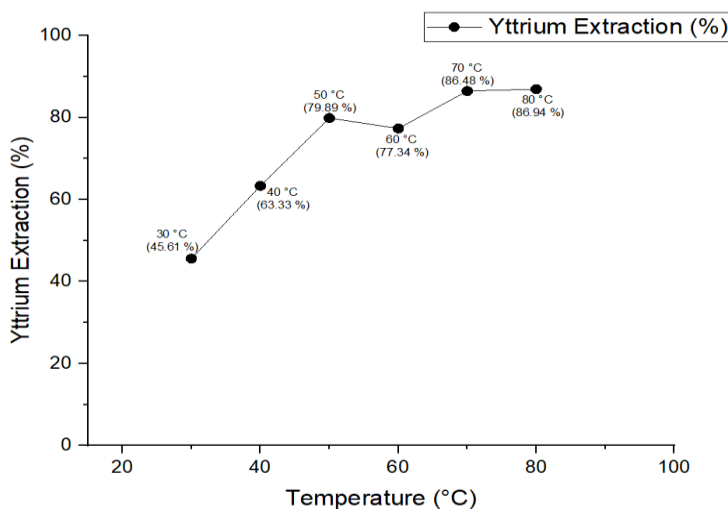


Figure 3. Yttrium extraction at various temperatures

In Figure. 2, with variations in particle size, the results can be seen that initially using tin slag measuring +100 mesh resulted in a yttrium extraction percentage of 37.24%, then using a sample measuring -100+150 mesh resulted in an extraction percentage of 48.81%, then at varying particle sizes -150+ The 200 mesh results showed 59.25%, which was quite large compared to the previous size and was already above 50%. There was a percentage increase of 60.47% at the particle size -200+325 mesh, and the highest increase occurred at the -325 mesh size of 72.46%. This has been proven to reduce particle size enough to provide significant results on the percentage of yttrium extraction. In Figure 3, with temperature variations in the range of 30-80 °C, it can be seen that the results at an initial temperature of 30 °C produced an extraction yield of 45.61%, and then using a temperature of 50 °C produced a yttrium extraction percentage of 86.94%. The extraction percentage reached its highest point of 86.94% at this temperature of 80 °C.

Research on the leaching behavior of mixed rare-earth concentrate using

hydrochloric acid found that the leaching rate can be greatly improved after a fine grinding process. The leaching efficiency of rare-earth elements was affected by different particle sizes. The research findings also indicate that the rate of leaching shows substantial variations in response to changes in temperature and the degree of particle size reduction [30]. The leaching of rare earth metals (REMs) with HCl increases with increasing temperature up to 90°C, which was found to be the optimal condition for maximum dissolution (~95%). Further temperature increase was not explored in this study, but excessive heating could potentially affect the solubility and stability of certain rare earth compounds in solution.[41].

### Effects of stirring speed

Experiments on dissolving tin slag with varying stirring speeds in the range of 100–350 rpm while keeping all other parameters constant (HCl concentration of 2 M, temperature of 30 °C, particle size -325 mesh) are shown in Figure 4.

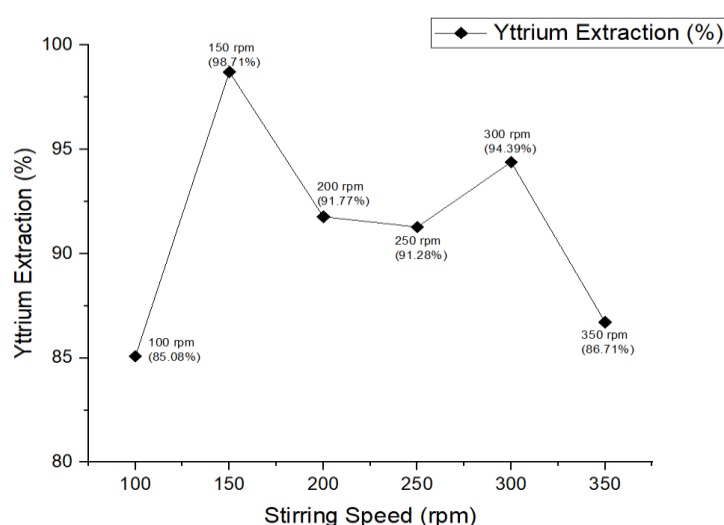


Figure 4. Yttrium extraction at various stirring speeds

In Figure 4, with variations in stirring speed, the results show that at a low speed of 100 rpm, the yttrium extraction percentage was 85.08%. Then, using a speed of 150 rpm, the percentage increased to 98.71%. At speeds of 200 and 250 rpm, the percentage of yttrium extraction decreased to 91.77% and 91.28%. Then, at a stirring speed of 300 rpm, the percentage increased but not significantly enough to 94.39%, and at the highest speed of 350 rpm, unfortunately, the percentage decreased to 86.71%. So, it can be concluded from the variations in stirring speed that the optimal percentage is at a speed of 150 rpm with 98.71%.

In another study, it was also reported that the results of recovering yttrium from "used fluorescent lamps" by varying the stirring speed showed that the optimal recovery point was at a stirring speed of 150 rpm, where increasing the stirring speed, unfortunately, did not provide significant results on yttrium recovery

## CONCLUSION

HCl has proven to be an efficient leaching agent for the extraction of yttrium. The efficiency of yttrium recovery is influenced by several operational parameters, including HCl concentration, particle size, temperature, and stirring speed. However, variations in stirring speed exhibited no statistically significant effect on extraction efficiency. The optimum condition for the extraction of yttrium from the tin slag was at a temperature of 80 °C, an acid concentration of 2 M, a particle size of -325 mesh, and a stirring speed of 150 rpm, resulting in up to 86.94% extraction of yttrium. This research provides a foundation for future advancements in yttrium purification and its potential industrial applications. The integration of the efficacy of HCL as a leaching agent with optimized process

conditions represents a critical step toward the production of high-purity yttrium.

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