A STUDY OF CERIUM EXTRACTION FROM BANGKA TIN SLAG USING HYDROCHLORIC ACID

Bangka Tin Slag (BTS) was a tin-smelting waste containing high silica and other elements that have high economic value, including cerium, which is a rare earth element. Silica and Ce_2O_3 contents in BTS were 32.86 % and 1.35 % respectively. Other elements that have high concentrations in BTS include 15.46 % of CaO, 10.88 % of Al_2O_3, and 9.20% of Fe_2O_3. The objective of this study was to determine the optimum conditions for cerium extraction using HCl, which includes HCl concentration, temperature, particle size, stirring speed, and dissolution time. In addition, the effect of these parameters on Ce extraction was also studied. The one-factor-at-time method was used to determine the optimum conditions. Pretreatment of BTS with the alkaline fusion method and water leaching was done to reduce both the silica content and increasing its porosity. Alkaline fusion carried out at 700 °C using NaOH converts the silica into water-soluble sodium silicate. Characterization of the slag structure before and after the pretreatment process was completely carried out by using X-ray diffraction (XRD), X-ray fluorescence (XRF), Scanning electron microscope (SEM), and optical microscope. Furthermore, measurement of Ce content in the filtrate of the dissolution process was performed with inductively coupled plasma – optical emission spectrometry (ICP-OES). The results showed that the optimum of 75.16 % Ce was extracted by using some parameter conditions, namely by 2.5 M of HCl concentration, at the temperature of 40 °C, with the particle size of −325 mesh, stirring speed of 150 rpm, and dissolution time of 180 minutes. Each parameter gives a significant effect on Ce extraction, wherein the initial stage, the increase in the value of each parameter gives an increase in Ce extraction and begins to decrease when equilibrium occurs

Keywords: Bangka tin slag, cerium, HCl, alkaline fusion, water leaching, optimum conditions

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1. Introduction

Rare Earth Elements (REE) are 15 elements in the lanthanide group as well as Sc and Y which have similar characteristics with lanthanide elements [1]. The similarity of REE chemical properties makes their separation and purification require selective methods such as liquid-liquid extraction [2]. REE are included in the critical material category because of their high economic importance and supply risk [3–6]. The use of REE often could not be substituted with other materials related to living and technology standards [7]. Cerium (Ce) is a light rare earth element (LREE) that is widely used as a glass polishing material, catalyst, oxygen sensor, and fuel cell. The presence of Ce in the earth's crust is higher than copper (66.5 ppm) and lead (60 ppm) [8]. Ce in nature has two different ionic states, the trivalent and tetravalent Ce which is beneficial for increasing the photocatalystic performance [9]. Tetravalent Ce tends to be more stable than trivalent [8].

The main resources of rare earth elements that have been processed are monazite, xenotime, and bastnaesite [10]. Among the three types of minerals, the lowest amount of cerium was in xenotime while cerium content in monazite was similar to bastnaesite, which was 46–49 % of Ce [11].

Tin mining activities in Bangka produced tin ingot and other forms of tin metal products through the smelting and

refinery process. Generally, cassiterite (SnO_2) concentrates processed in smelters have a minimum tin (Sn) content of 70 %. Thus, 30 % of the impurities in the concentrate, including REE, were also smelted and became slag. The first smelting slag still contained 20–30 % of Sn and was smelted again to obtain slag with only 2–3 % Sn content [12]. This second smelting slag, later called Bangka Tin Slag (BTS), was used in this study. BTS contains LREE including Ce [13].

Generally, tin slag is processed for tantalum and niobium. The economic value of tin slag processing could be increased by extracting other metals including cerium. Therefore, this research needs to be done to obtain the optimum conditions for cerium extraction, especially in the leaching process, so that the extraction process could be more efficient.

2. Literature review and problem statement

The authors of the paper [14] present the results of research on characterization of tin slag using microprobe analysis. They show that tin slag has an amorphous structure, and determined the matrix of the elements, called pseudo-structure, contained in the slag. The pseudo-structure gave advantages to predict the impact of chemical treatment on tin slag. According to the paper [15], the decomposition of slag with NaOH was effective to damage the silica structure in the slag and increase its porosity and provide good acid leaching results in the extraction of Ta and Nb. In tantalum and niobium extraction from tin slag, studies that have been carried out reveal several alternative processes, including leaching with H_2SO_4 and HF, decomposition of slag with NaOH at high temperatures, or a combination of acid-base leaching. The reaction of silica with sodium hydroxide resulted in sodium silicate (Na_2SiO_3) which is soluble in water [12]. REE, including Ce contained in slag, was assumed to be in the form of hydroxide after going through an alkaline fusion process. Ce or other REE hydroxides can be dissolved in strong acids such as HCl, H_2SO_4, and HNO_3. The paper [16] reports the results of studying the REE dissolution from phosphogypsum, it was known that HCl and HNO_3 are more efficient than H_2SO_4, and HCl is more economical than HNO_3. The study of lanthanum extraction from REE slag using sulfuric acid conducted by the authors [17] shows that under the conditions of 1.5 g/L slurry density, 0.3 M H_2SO_4, 750 rpm of stirring speed, leaching time of 5 h, and 30–80 °C of temperature, the reaction rate was controlled by chemical reactions in the first stage and diffusion in the ash layer in the second stage.

The authors of the papers [18–23] had performed the research in leaching of REE from different raw materials, including bastnaesite, Korean monazite, clay minerals, and bauxite residue. Unfortunately, studies on the dissolution of REE, especially Ce, from BTS using HCl after alkaline fusion and water leaching have not been conducted. Their reports indicated the parameters that affected the leaching recovery. In dissolution using HCl, a high concentration of HCl can increase the effectiveness of dissolution because the reaction speed is affected by the reagent concentration [18]. The dissolution process is generally also affected by temperature, as the reaction speed increases with increasing temperature [19]. The particle size also affects the result of the dissolution process, because a large surface area gives an increasingly better dissolution reaction [20]. The surface area of the particle can be increased by comminution or size reduction. Furthermore, turbulent stirring in the dissolution process makes slurry homogenization better, and also helps the process of ion diffusion from solids to bulk solutions [21]. Chemical reactions, including the dissolution process, concentration variation, have a certain rate, which is a function of time, so resident time plays a role in increasing percent extraction [22, 23]. This approach was used in the study of Ce dissolution from BTS.

3. The aim and objectives of the study

The aim of the study is to determine the optimum conditions of the parameters that affected the Ce dissolution from BTS using HCl after alkali fusion and water leaching.

To achieve this aim, the following objectives are accomplished:

– to obtain the optimum HCl concentration, temperature, particle size, stirring speed, and dissolution time;
– to analyze the effect of parameters on Ce extraction.

4. Materials and methods to study optimum conditions in dissolution of Ce from BTS using HCl

4. 1. Materials
Table 1, 2 show the materials used in these experiments which is BTS with a fraction of the particle size and the composition. The particle size in the range between the +20 to –65 mesh scale with various weight percentages was displayed in detail in Table 1. While the compound of composition with other elements of BTS was shown more completely in Table 2.

Table 1

Particle size fraction of BTS

Particle size (mesh)	+20	-20+48	-48+65	-65
Weight percentage (%)	3.81	54.85	34.78	6.56

Table 2

Results of BTS analysis by XRF

SiO_2 (%)	Fe_2O_3 (%)	CaO (%)	Al_2O_3 (%)	Ce_2O_3 (%)	Other REEs (%)	Other elements (%)
32.86	9.20	15.46	10.88	1.35	2.11	28.14

The XRF analysis showed that SiO_2 (32.86 %) was the major component in tin slag. REE contained in tin slag was 3.46 %, with 1.35 % of Ce_2O_3 content. CaO, Al_2O_3, and Fe_2O_3 are the other components that have a high concentration with their concentration of 15.46 %, 10.88 %, and 9.20 %, respectively.

4. 2. Pre-treatment
BTS in the amount of 250 g was mixed with 500 g NaOH in an alumina crucible. The mixture was heated in a muffle furnace at a temperature of 700 °C. Frit was leached with 1 L of water for 1 hour, then filtered. The water leaching residue was pulverized with a ball mill and sieved with a sieve size of 100, 150, 200, and 325 mesh. The particle size fraction obtained is presented in Table 3.

Table 3

Particle size fraction of dissolution feed material

Particle size (mesh)	+100	−100+150	−150+200	−200+325	−325
Weight percentage (%)	10.75	4.74	5.40	3.36	75.75
Ce content (%)	0.95	1.17	0.98	0.98	1.35

Milling process resulted in more than 70 % of the water leaching residue in size of −325 mesh. The Ce content in five variations of particle sizes did not have a significant difference.

4. 3. Dissolution

The experiment was carried out in a 400 mL beaker glass equipped with a digital mixer IKA RW 20 with PTFE pitch-blade turbine 45° impeller type. The beaker glass was placed on a Thermo Scientific Cimarec hot plate. 200 ml of 32 % HCl was put into the beaker glass, then 20 g of water leaching residue was added while stirring at 150 rpm. Stirring was carried out for several minutes according to the observed time parameter. After the dissolution time was reached, the slurry was filtered and the filtrate obtained was diluted. Dilution was aimed to prevent gel formation. Process parameters observed included HCl concentration, temperature, particle size, stirring speed, and dissolution time. Variations of each process parameter are presented in Table 4. Analysis of filtrate samples was carried out using the PerkinElmer ICP-OES Optima 8300 type.

Table 4

Variation of the dissolution process parameters

Parameter	HCl Concentration (M)	Temperature (°C)	Particle Size (mesh)	Stirring Speed (rpm)	Dissolution Time (minutes)
HCl Concentration	0.5; 1; 1.5; 2; 2.5; 2.75	Ambient	−325	150	5
Temperature	2.5	ambient; 40;50; 60; 70	−325	150	5
Particle Size	2.5	40	+100;−100+150; −150+200; −200+325;−325	150	5
Stirring Speed	2.5	40	−325	100; 150; 200; 250; 300; 350	5
Dissolution Time	2.5	40	−325	150	3; 5; 15; 30; 60; 120; 180; 240

The percentage of Ce extraction was calculated using the following equation:

$$Ce\ Extraction = \frac{Weight\ of\ Ce\ in\ the\ filtrate}{Weight\ of\ Ce\ in\ the\ feed} \times 100\ \%. \quad (1)$$

Table 4 shows the design of experiments in determining the optimum operating conditions, following the one-factor-at-time method in which the operating parameters studied were varied while the other parameters were fixed [24].

5. Results

5. 1. Pre-treatment of BTS

The XRD and SEM characterization of the BTS sample, before and after pre-treatment is shown in Fig. 1. While photo microscope optic of the BTS sample, before and after pre-treatment are shown in Fig. 2.
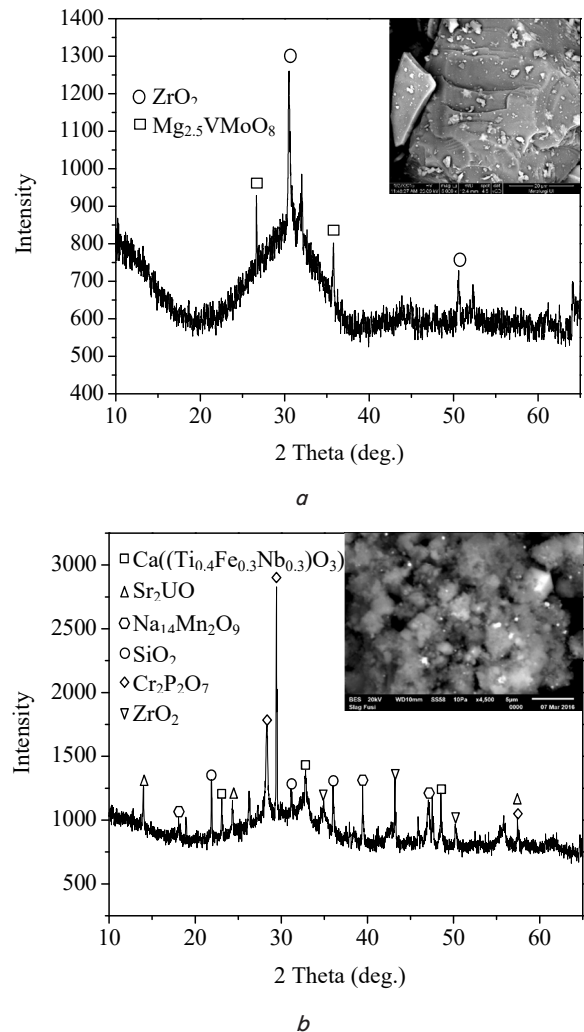

Fig. 1. XRD and SEM analysis of the BTS sample: a — before pre-treatment, b — after pre-treatment
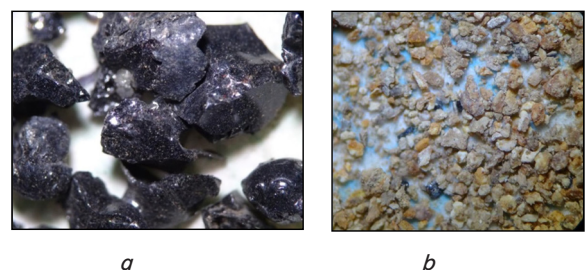

Fig. 2. Photo microscope optic of the BTS sample: a — before pre-treatment, b — after pre-treatment

As shown in Fig. 1, the XRD characterization results show that the BTS structure changes from amorphous to

crystalline. Fig. 1 also shows SEM analysis, which indicates the change in BTS surface, which before the pre-treatment of the granular surface looked compact while after alkaline fusion and water leaching the particle porosity significantly increased. Characterization with an optical microscope at 45x magnification showed changes in the appearance of BTS granules, dark brown silica enveloped BTS granules (Fig. 2, *a*) but after pre-treatment, there was a change in the color and surface structure of the granular (Fig. 2, *b*).

5. 2. Effects of HCl concentration and temperature

The results of BTS dissolution experiments with variations in HCl concentrations in the range of 0.5–2.75 M and temperature variations at ambient temperatures up to 80 °C are shown in Fig. 3.
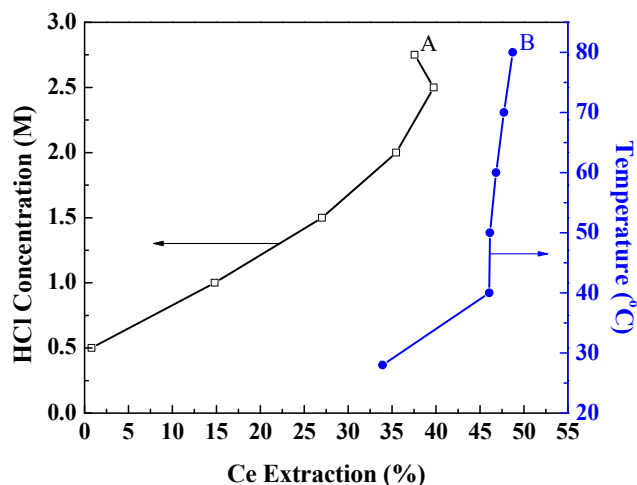

Fig. 3. Ce extraction at various: A — HCl concentrations; B — temperatures

Curve A in Fig. 3 shows the effect of changes in the concentration of Ce extraction, at a concentration of 0.5 M HCl, Ce extraction was still low at 0.81 %. Maximum Ce extraction was obtained at a concentration of 2.5 M which was 39.75 %. Curve B in Fig. 3 shows the effect of temperature changes on Ce extraction. At room temperature, Ce extraction was 33.91 %, which seems to decrease compared to the value in Curve A, suggesting due to deviation formed, even though the operating conditions are the same. The Ce extraction significantly increased from ambient temperature to 40 °C, at which there was an increase of 33.91 % Ce extraction to 46.06 %.

5. 3. Effects of particle size

The results of the experiment of Ce dissolution with HCl in various particle sizes are presented in Fig. 4.

Fig. 4 shows Ce extraction at various particle sizes. At a particle size of +100 mesh, Ce extraction was 39.81 % and tended to be constant up to a particle size of −150+200 mesh. For finer particle size, Ce extraction increased to reach 47.77 % at a particle size of −325 #. The extraction shows a little increase of about 1.71 % than the result value in curve B in Fig. 4, because of the deviation value though the operating conditions are the same as well.
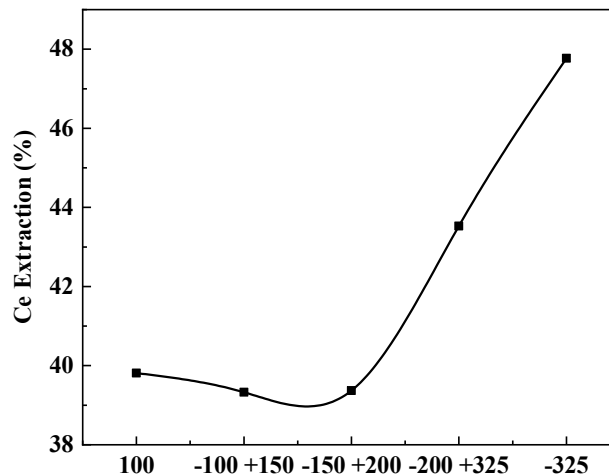

Fig. 4. Ce extraction at various particle sizes

5. 4. Effects of stirring speed and dissolution time

The results of Ce dissolution experiments with stirring speed variations in the range of 100–350 rpm and dissolution time in the range of 3–240 minutes are shown in Fig. 5.
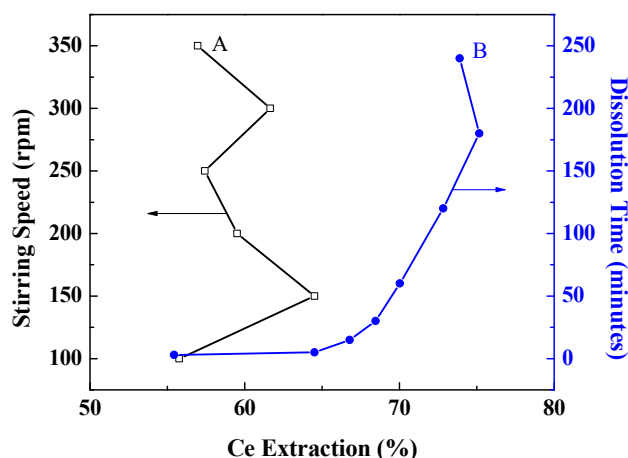

Fig. 5. Ce extraction at various (A) stirring speeds and (B) dissolution times

In Fig. 5, Ce extraction at various (A) stirring speeds and (B) dissolution times are shown. Curve A shows the effect of stirring speed on Ce extraction. At a stirring speed of 100 rpm, the Ce extraction obtained was 55.76 %. Increased stirring speed up to 150 rpm gave an increase in Ce extraction up to 64.51 %. Curve B in Fig. 5 shows the effect of dissolution time on Ce extraction. At the dissolution time of 3 minutes, Ce extraction was 55.43 %. Increasing the dissolution time gave an increase in Ce extraction up to 75.16 % at the dissolution time of 180 minutes.

6. Discussion of experimental results

The decomposition of silica structure in BTS using NaOH at high temperatures produces sodium silicate compounds that are soluble in water, so the silica content in the frits resulting from alkaline fusion decreases after water leaching. The silica released from slag causes the formation or enlargement of pores on the surface. Thus,

the reaction between slag and HCl can occur more effectively.

HCl concentration is the first parameter observed, and the results obtained indicate that increasing HCl concentration causes higher Ce extraction [25]. Unfortunately, an increasing HCl concentration of more than 2.5 M indicates a decrease in Ce extraction. When using HCl 2.75 M, Ce extraction became 37.56 %. This occurred because the equilibrium reaction of the Ce dissolution by HCl was achieved at a 2.5 M HCl concentration, so a further increase in concentration can cause the reaction to reverse or Ce extraction decrease [17]. The reaction of Ce dissolution by HCl is as follows:

$$Ce(OH)_3 + 3HCl = CeCl_3 + 3H_2O. \qquad (2)$$

Ce reacting with HCl increases as temperatures rise [18]. Unfortunately, above 40 °C, the increase in Ce extraction was very small and only 48.72 % at a temperature of 80 °C. This is similar to the study conducted on the dissolution of Ce from phosphogypsum, which shows that an increase in temperature from 50 °C to 80 °C did not provide a significant increase in Ce extraction [16].

To find out more about the effects of temperature changes, it is necessary to review the thermodynamics and reaction kinetics. Thermodynamic data from the dissolution reaction of $Ce(OH)_3$ with HCl are presented in a curve in Fig. 6 obtained from the HSC Chemistry 6.0^{TM} software as performed by Sulaksana et al [26].
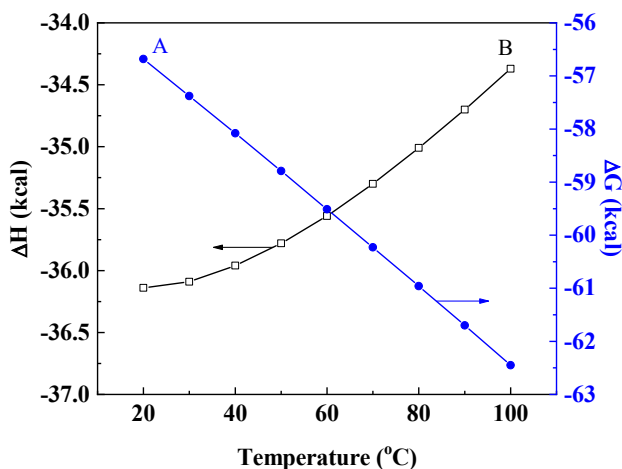

Fig. 6. Changes in enthalpy and Gibbs free energy to temperature in the dissolution of $Ce(OH)_3$ with HCl

In the temperature range of 20–100 °C, Gibbs free energy from the dissolution reaction of $Ce(OH)_3$ by HCl was in the range of −56.68 to −62.45 kcal, so it could be concluded that this reaction could take place spontaneously. With a negative enthalpy value, it was known that this dissolution reaction was exothermic, so an increase in temperature for the exothermic reaction would shift the direction of the reaction towards the reactants [18, 19]. However, in this study, the Ce extraction increased with temperature. This indicates the rate of reaction towards the product was more dominant than the direction of the reactants with an increase in temperature. According to the Arrhenius equation [27]:

$$k = Ae^{\frac{-Ea}{RT}}, \qquad (3)$$

where k was the reaction rate constant, A was the pre-exponential factor, Ea was the activation energy, R was the ideal gas constant, and T was the temperature.

Fine particle size has a large contact surface area and could increase reaction rates. This also applies to the dissolution experiments of Ce from BTS. The size of the particle also affects the performance of the dissolution process because a large surface area gives an increasingly better dissolution reaction. The surface area of the particles could be increased by comminution or size reduction. Ruan et al have also reported that the finer particle size in the process of dissolving the REE concentrate using HCl increases the REE extraction [19].

Stirring helps the process of homogenizing the mixture in obtaining an even distribution of solids in the HCl solution. The more evenly distributed, the better the contact between the solid and the reagent, and the diffusion of the reaction product from the solid to the bulk solution and the diffusion of the reactants to the surface of the solid also increases thereby increasing Ce extraction [21]. Unfortunately, stirring speeds above 150 rpm did not show a stable trend for Ce extraction.

Chemical reactions, including the dissolution process, have a certain rate as a function of time, so time plays a role in achieving high rates of reaction conversion. Lengthening the dissolution time means increasing the chance of contact between the reagent and the solid, so there is more dissolved Ce. Following the equation of the second-order reaction rate [28]:

$$r = \frac{dC_A}{dt} = -kC_A C_B, \qquad (4)$$

where r was the reaction rate, C_A was the concentration of $Ce(OH)_3$, C_B was the concentration of HCl, and t was the time.

Unfortunately, increasing the dissolution time to 240 minutes showed a decrease in Ce extraction. This happened because equilibrium had been reached, so the addition of time caused the reaction to reverse and result in a decrease in Ce extraction. The trend in which an increase in reaction rate occurs at the beginning of time and a decrease in reaction rate over time is similar to the REE leaching study of bauxite conducted by Borra et al. [22].

7. Conclusions

1. Our experimental research has solved the problem of obtaining the optimum conditions to dissolve Ce using HCl. More than 75 % Ce in BTS can be dissolved in the concentration of 2.5 M, temperature of 40 °C, the particle size of −325 mesh, stirring speed of 150 rpm, and dissolution time of 180 minutes.

2. The concentration of HCl, temperature, particle size, stirring speed, and dissolution time gave a significant effect in Ce extraction. An increase in the value of these parameters can result in a higher Ce extraction, but under certain conditions where equilibrium has occurred in the system then an increase in the value further causes a decrease in Ce extraction.

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