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Pre-treatments and Leaching of Rare Earth Elements (REE) from Malaysian Bauxite

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Abstract: The importance of rare earth elements (REE) in high-end technology applications has become increasingly notable. Malaysia is rich in mineral resources like bauxite ore, which contains a significant amount of REE. However, REE from bauxite cannot be extracted directly without pre-treatments and the growing concerns of environmental pollution, such as red dust and red mud, are partially due to the increased stockpile of bauxite residue (BR). The study intends to compare the effect of pre-treatments before the REE leaching process of the raw bauxite (RB) from Felda Bukit Goh, Kuantan, Pahang. The methods 1 (M1) and 2 (M2) included roasting, magnetic separation, Bayer process, acid cracking, leaching via $(NH4)_2SO_4$, and precipitation via $C_2H_2O_4$. In M2, lower current intensity in magnetic separation, a lower solid-to-liquid ratio and the absence of H_2O_2 in acid cracking were used. The RB contains light rare earth elements (LREE) between 190.20 to 318.71 mg/L with abundant cerium (Ce) and neodymium (Nd). The RB comprises of Fe_2O_3 44.66%, Al_2O_3 34.15%, TiO_2 8.39% and SiO_2 8.39% whereas gibbsite ($Al(OH)_3$) was the main mineral. In M1, the REE contents decreased by 12.5% (301.31 mg/L) and between 0.4% to 8.4% after roasting and magnetic separation, respectively. Via the Bayer process, REE contents increased by 15.5% (346.81 mg/L) in M1, whereas decreased by 48.4% (119.34 mg/L) in M2. In acid cracking using H_2SO_4 with H_2O_2 in M1, the REE contents majorly remained in BR, while without H_2O_2 the REE contents decreased, indicating REE has been leached out in M2. The H_2O_2 caused the BR to become viscous and harder after drying, leading to the REE interlocking in the residue. The overall leaching performances in M1 and M2 were achieved at 1.34% and 99.0%, respectively, evidently showing that introducing H_2O_2 before the leaching process does not improve the REE extraction. Meanwhile, precipitation via $C_2H_2O_4$, produced rare earth oxalate (REOx) up to 1.59 % in M1 and 31.80% in M2 where, based on element purity, the Ce and Nd in M2 attained were 92.78% and 7.20%, respectively.

Keywords: ammonium sulphate; bauxite; cerium; leaching; magnetic separation, rare earth elements

1. Introduction

Bauxite ore is a sedimentary rock that is enriched with aluminum (Al), which is attributed to the presence of minerals like gibbsite ($Al(OH)_3$), boehmite (γ-AlO(OH)) and diaspore (α-AlO(OH)) [1]. Al is indeed a versatile metal that has broad applications, including transportation, construction, manufacturing, food packaging, electronic appliances and many more. Other than Al, it contains high iron oxides (hematite and goethite), kaolinite, titanate, silicate, phosphate and carbonate minerals [1, 2]. In 2024, Australia is the world's leading bauxite producer, with a total production of 1.1 x 10^{10} tons, which is around 30% globally [3]. Followed by Guinea, China, Brazil and Indonesia at 8.2 x 10^9, 6.0 x 10^9, 3.5 x 10^9, and 2.3 x 10^9 tons, correspondingly. However, Guinea holds the world's most abundant bauxite reserves in 2024, which is about 7.4 x 10^9 tons [4]. Therefore, to produce one metric ton of Al, four metric tons of bauxite ore are required to yield two metric tons of alumina or aluminum oxide (Al_2O_3). The general process of Al production includes the Bayer process, where the bauxite ore is mixed with sodium hydroxide (NaOH) solution to form a soluble sodium aluminate ($NaAl(OH)_4$). Then, the tailing product, that is the red mud or referred as the bauxite residue (BR) in this study, is normally left untreated. The addition of aluminum hydroxide ($Al(OH)_3$) seeds into the solution will enhance the crystal growth of $Al(OH)_3$ [5]. Through calcination, the $Al(OH)_3$ is converted to Al_2O_3, and further electrolysis separation is done for Al metal purification.

Without a doubt, the ore also consists of significant rare earth elements (REE) with the most abundant elements such as scandium (Sc), lanthanum (La), cerium (Ce), neodymium (Nd) and yttrium (Y) [1, 6, 7]. The REE remaining in the BR can be extracted, as well as other valuable compounds, including iron oxide. Several studies have been conducted on the REE extraction from BR. Borra [8] used water washing to remove NaOH from the BR and then leached using various types of acids nitric (HNO_3), hydrochloric (HCl), sulfuric (H_2SO_4), citric ($C_6H_8O_7$), methane sulfonic (CH_3SO_3H), and acetic (CH_3COOH). The findings show that the REE can be extracted between 70 – 80% at 6N HCl. Another study conducted by Borra and the team performed a combination of pyrolysis and leaching methods on BR for REE extraction [9]. After smelting at 1500 ºC with the presence of calcium silicate ($CaSiO_3$) as a flux, the BR was further leached using HNO_3, HCl and H_2SO_4. The results revealed that Sc and other REE can be recovered above 95% and 70%, for all the acids at a concentration of 3.0 N, respectively. Several research studies have also performed REE extraction from bauxite ore before the alumina is extracted. Kursrini *et al.* [10] employed heap leaching with H_2SO_4 as the lixiviant to extract La and Y from a low-grade of BR. Via this method, La and Y can be leached out at 0.03411 ppm and 0.05568 ppm, respectively. Precipitating the REE using trisodium phosphate and phytic acid provides a positive result where La and Y can be enriched up to 0.04411 ppm and 0.28431 ppm, respectively. Other than that, performing a two-step process based on dry digestion via H_2SO_4 or HCl followed by water leaching was demonstrated to yield around 40 wt.% of Sc as reported by Rivera and co-workers [11].

Malaysia is also rich in mineral resources, including bauxite, which is mainly distributed in Pahang, Johor and Sarawak. The production of Malaysian bauxite has declined from 2015 (27.7 x 10^6 metric tons) to 2021 (0.04 x 10^6 metric tons) [12, 13]. The unstable changes in mineral export policy have likely affected the yearly production. In addition, there were concerns about environmental issues, such as red dust from the bauxite ore polluting the road from the mining site to the town area, which occurred in Kuantan, Pahang, Malaysia [4]. Regardless of that, in reality, bauxite in Malaysia has not been fully exploited as miners export the raw bauxite ore, especially to China, without considering the precious elements that can be potentially extracted, such as Al, Fe and REE. For the past 5 years, the interest in REE in Malaysia has attracted a lot of attention from the government and private sectors.

REE deposits in the form of alkaline igneous rocks are the REE primary resource including monazite and xenotime [14]. In Malaysia, monazite and xenotime can be attained from the tailing product or referred as amang produce from tin mining. However, the resources contain high level of radioactive materials (RM) such as thorium (Th) and uranium (U) [15] make it less unfavorable resource. According to Atomic Energy Licensing Board (AELB) of Malaysia the Th or U cannot be exceeded than 0.05 wt. % [16], where the average Th in Malaysian monazite and xenotime detected were 6.5 – 7.5 wt. % and 1.5 wt. %, respectively [17]. At present, ion adsorption clay (IAC) has become the favor as the secondary resource for REE in Malaysia [18, 19]. By far only one pilot plant operated in Kenering, Perak has successfully produced a commercial grade of rare earth carbonate from in situ leaching (ISL) mining method adopted from China technology [20]. Aside from IAC, bauxite ore is also categorized as REE secondary resource where both deposits contain very low RM which is less than 10 ppm. The permission limit of Th and U concentrations must be less than 246.5 ppm and 80.9 ppm, respectively [21]. Although IAC is more popular perhaps bauxite could be one of the potential REE secondary resources in the future. The main reason bauxite ore was chosen as the raw material for REE extraction in study is to fully exploit the BR post to the extraction of alumina and to the best of the author’s knowledge the study regards to REE extraction from BR is limited and has not been detailed explore in Malaysia.

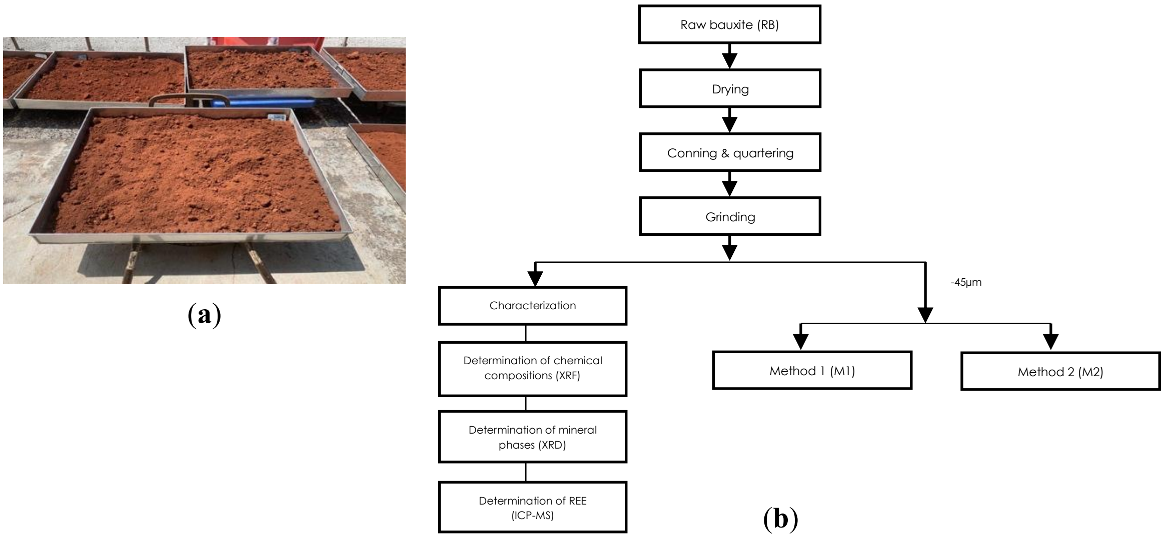
The REE phases in bauxite mostly behave as monazite, xenotime and bastnaesite minerals [22] where cracking process is need to liberate the REE inside the ore. Apart from that, the REE can also be occurred because of presence of ion adsorption form on clay or diaspore surface in bauxite. Commonly, the extraction of REE from bauxite involves pre-treatments prior to REE leaching process as described earlier. Some studies have reported the use of hydrogen peroxide (H_2O_2) in assisting the REE leaching or cracking. The rare earth dissolution can be enhanced as high as 93% from alluvial gold mining waste using 10% of H_2O_2 with sulfuric (H_2SO_4) acid solution [23]. Another study also shows that the REE leching efficiency from spent fluid catalytic cracking can be attained up to 93% using 30% w/w of H_2O_2 with hydrochloric (HCl) acid solution [24]. In IAC, the most popular lixiviant or leaching agent used is the ammonium sulphate ($(NH_4)_2SO_4$) due to its lower consumption, milder conditions and selective ion-exchange mechanism as to compare concentrated acid. In the meantime, not much research addressing the use of $(NH_4)_2SO_4$ for REE leaching from bauxite and only few studies have been reported. Meng and co-workers [25] demonstrated that the REE extraction from BR via a combination of roasting with $(NH_4)_2SO_4$ at 700ºC, followed by a water leaching method. The study found that 90% of Sc can be extracted. It is also claimed by Mouchos and team [26] that REE from Greek bauxite (raw sample and before Bayer process) can be recovered around 19 – 47 wt. % through 0.5 M $(NH_4)_2SO_4$ at pH 4 for 16 hours of mixing. But then, Meng and team only emphasized the extraction of Sc whereas Mouchos and team used $(NH_4)_2SO_4$ for sample characterization purpose.

Hence, the present study primarily aims to demonstrate the feasibility study in developing a process route for rare earth oxalate (REOx) production from bauxite as well as to compare the effect of pre-treatment with or without H_2O_2 during acid cracking towards the REE leaching performance via $(NH_4)_2SO_4$ solution. The pre-treatments include conventional roasting, magnetic separation, acid cracking and the Bayer process. Oxalic acid ($C_2H_2O_4$) was selected as the precipitant agent as it is commonly used to precipitate REE into oxalate.

2. Materials and Methods

2.1 Raw material and sampling preparation

The raw bauxite (RB) sample used in this study was collected from Felda Bukit Goh, Kuantan, Pahang, Malaysia via grab sampling. The sample was dried via sun-drying (Figure 1a), mixed homogenously 16 times and then divided based on the coning and quartering sampling technique until 1 kg of the sample was obtained [27]. Subsequently, the sample was ground using a grinding mill at 78 rpm for 10 mins and screened using a 45 µm sieve via wet screening method [28].



(**a**)

(**b**)

Figure 1. (**a**) RB sample and (**b**) process flow chart of sample preparation and characterization.

2.2 Chemical compositions and mineral phases determination

The chemical compositions of RB were determined using X-ray Fluorescence Spectrometer (XRF-1700, Shimadzu, Japan). For XRF sample preparation, 1.1 g of Somar-mix mixed with 10 g of RB, pulverized (PULVERISETTE 9, Fritsch, Germany) at 750 rpm for 5 mins and manually pressed using a hydraulic press (Graseby Specac, UK) at 20 tons to form a pellet. The mineral phases of RB were determined using X-ray Diffraction (XRD-D8, Bruker, Germany). 10 g of RB pulverized at 750 rpm for 5 mins. Subsequently, the beam operated at 40 kV, 40 mA and scanned at rate of 2˚ per min and 2Θ range of 4˚− 70˚ [28]. The mineral phases identification was determined based on the PDF-4 minerals database.

2.3 ICP-MS sample preparation

The REE contents of the RB and BR were outsourced and determined using Inductively Coupled Plasma - Mass Spectrometry (ICPMS) (7500a, Agilent, USA) Central Laboratory, Universiti Malaysia Pahang (UMP), Kuantan, Pahang, Malaysia and Laser Abrasion - ICPMS (NexION 2000, Perkin Elmer, USA), Technical Service Division (BPT), Ipoh, Perak, Malaysia. For solid samples outsourced to UMP, the sample was digested based on the open digestion method and prepared at the Mineral Research Centre, Ipoh, Perak. The digestion method was performed based on in house method [29]. Approximately 0.4 g of solid sample was dissolved into 10 mL of 40% hydrofluoric (HF) acid and placed in a water bath at 95.5°C overnight. Then 10 mL of 37% HCl was added into the dried residue and placed on a water bath again at 95.5°C for 10 mins. 30 mL of distilled water (DW) was added and left to cool at room temperature. The solution was transferred into 400 mL, placed on a hot plate, evaporated until fully dried and left cooled. Afterwards, 3 mL of 95 – 97% H_2SO_4 was added into the beaker and placed on the hot plate for 5 mins. 50 mL of DW was added gently into the beaker, then heated using a hot plate for 30 mins. The solution was transferred into a 250 mL volumetric flask after cooling. For samples outsourced to BPT, the sample was prepared with fusion flux and LA-ICPMS setting according to Hanuar and team [30]. Some of the liquid samples were outsourced to UMP and ICPMS (NexION 300Q, Perkin Elmer, USA) Universiti Sains Malaysia (USM).

2.4 Method 1 (M1): Roasting-Magnetic separation-Bayer-Acid cracking with H_2O_2-Leaching processes

Prior to leaching, pre-treatments such as roasting, magnetic separation, Bayer process and acid cracking were introduced in M1, as shown in Figure 2. The RB was roasted at 500°C [31] for 60 mins using a furnace (CWF1200, CARBOLITE GERO, Germany). Through roasting, it can convert the non-magnetic (e.g., goethite) and paramagnetic minerals to magnetic minerals (e.g., hematite and magnetite) [27]. Then the bauxite-roasted sample (BRS_{M1}) was processed via a wet high-intensity magnetic separator (WHIMS) [32] at 3.0 A with five runs. Two products were attained: bauxite-roasted magnetic (BRM_{M1}) and bauxite-roasted non-magnetic ($BRNM_{M1}$). The aim of using WHIMS is to extract as much iron oxide as possible [33]. The $BRNM_{M1}$ underwent the Bayer process using sodium hydroxide (NaOH) as the leaching agent, and procedures were followed according to Ibrahim *et al.* [34]. The solid-to-liquid (S/L) ratio used was 1:5 with 3.0 M of NaOH, stirred at 400 rpm for 60 mins and heated at 90 ± 1°C. The pH of the solution was adjusted to 12.06 using 1% H_2SO_4. The purpose of the Bayer process was to remove the alumina. After the Bayer process, the sample was filtered using a vacuum pump to separate the pregnant solution 1 ($PS1_{M2}$) and bauxite residue 1 ($BR1_{M1}$). The $BR1_{M1}$ was further processed via acid cracking at an S/L ratio of 1:10 using 1.0 M of H_2SO_4 with the addition of 10 mL of 30% w/w hydrogen peroxide (H_2O_2) [24], stirred for 60 mins at 500 rpm. The filtered products were pregnant solution 2 ($PS2_{M1}$) and bauxite residue 2 ($BR2_{M1}$). Acid cracking aimed to liberate the REE interlock within the minerals of BR. The $BR2_{M1}$ then underwent leaching process 1 using an S/L ratio of 1:3 and 2.0 M $(NH_4)_2SO_4$, the leaching process was adopted and modified from Shia [35]. The mixing time, stirrer speed and pH of the solution were fixed at 60 mins, 500 rpm and 3 to 4, respectively. After the leaching process, two products were attained, namely the pregnant solution 3 ($PS3_{M1}$) and bauxite residue 3 ($BR3_{M1}$). The same steps were used for leaching processes 2 and 3; the products attained were $PS4_{M1}$, $BR4_{M1}$, $PS5_{M1}$ and $BR5_{M1}$. The $(NH_4)_2SO_4$ aimed for the REE ion-exchange and leaching of the other REE phases. For the precipitation process, the $PS3_{M1}$, $PS4_{M1}$ and $PS5_{M1}$ were mixed with 2.2 M oxalic acid ($C_2H_2O_4$) using a liquid-to-liquid (L/L) ratio of 1:1 at 500 rpm for 30 mins. The product was filtered using filter paper (Whatman, ϕ 12 cm with pore size of 2.7 μm) and a vacuum pump (VELP Scientifica, Italy). The REE contents in solid and liquid were sent to BPT and USM, respectively.

2.5 Method 2 (M2): Roasting-Magnetic separation-Bayer-acid cracking-Leaching processes

The processes involved in M2 were almost similar to M1. However, a few adjustments have been made, such as the current intensity of the WHIMS applied at 1.5 A repeated twice, in acid cracking, the S/L ratio used was 1:1.5 without H_2O_2. This was to compare with the acid cracking process with H_2O_2. The reduction of the S/L ratio was to reduce the acid consumption during acid cracking. In addition, the leaching process was employed only once in M2 and then continued to the precipitation process, herein, the $PS2_{M2}$ also underwent the precipitation process as shown in Figure 3. Before $REOx2_{M2}$ formation, the $PS2_{M2}$ was mixed with $(NH_4)_2SO_4$ solution at a ratio of 1:3. The REOx attained in this study was a mix of REE and impurities. The impurities were analyzed using XRF and XRD. The REE contents in the solid and liquid were sent to UMP. The REE recovery (%) and RE metal purity (%) were calculated based on equations (1) and (2), respectively.

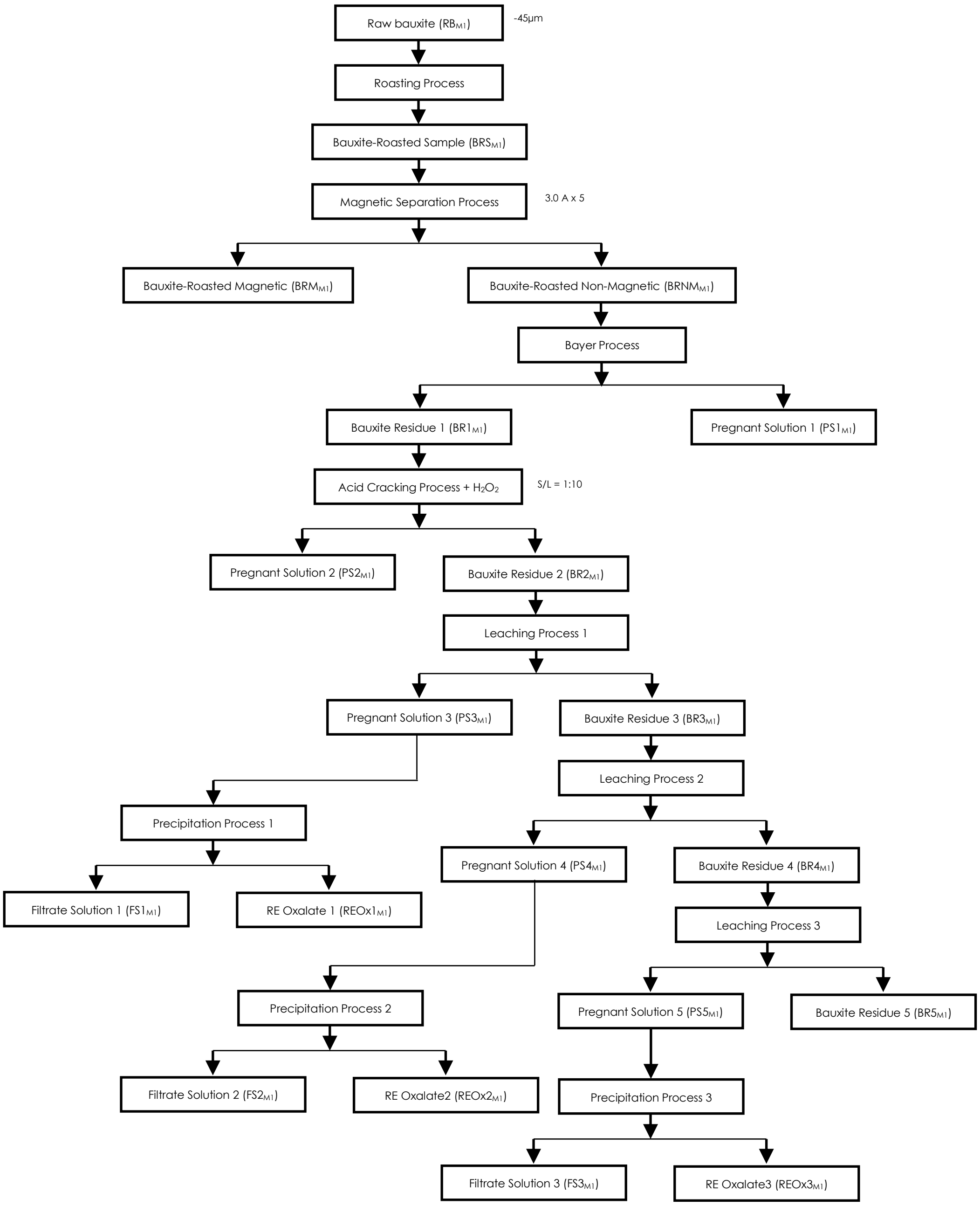


Figure 2. Process flow chart of method 1 (Roasting-Magnetic Separation-Bayer-Acid cracking with H_2O_2-Leaching processes).

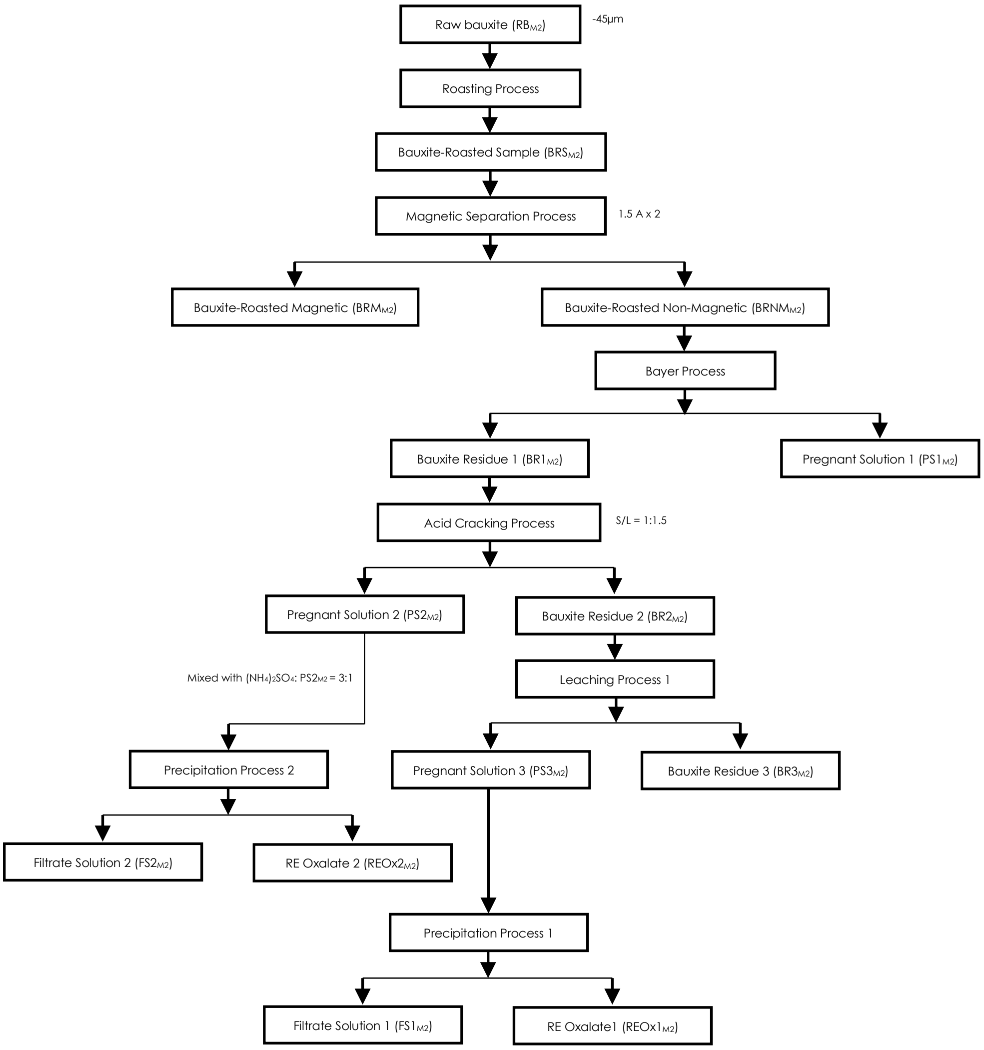


Figure 3. Process flow chart of method 2 (Roasting-Magnetic Separation-Bayer-Acid Cracking-Leaching Processes).

$$\text{REE recovery (\%)} = \frac{m_{\text{REE of PS or REOx}}}{m_{\text{TREE of BR1}}} \text{ x } 100\% \tag{1}$$

$$\text{RE metal purity (\%)} = \frac{C_{\text{RE metal of REOx}}}{C_{\text{TREE of REOx}}} \text{ x } 100\% \tag{2}$$

Where m is the mass of the REE in the sample and C is the concentration of REE in the sample.

3. Results and Discussion

3.1. Sample Characterization of Raw Bauxite (RB)

3.1.1. X-ray fluorescence (XRF) analysis result

The chemical compositions of the RB for M1 and M2 are presented in Table 1. The Fe_2O_3 was the major compound with 44.6643 wt%, whereas the Al_2O_3, TiO_2 and SiO_2 were 34.1519 wt%, 9.6104 wt% and 8.3937 wt%, respectively. Other trace compounds were also detected however, all were found to be less than 1.0 wt%. The data also shows that the RB used in this study was classified as a low-grade bauxite ore where Al_2O_3 content was less than 40% and the SiO_2 content higher than 5%, respectively [36]. Moreover, the ratio of Al_2O_3 to SiO_2 calculated was around 4.07, indicating pre-treatments are required prior to Bayer process implementation.

Table 1. Chemical compositions of RB from Felda Bukit Goh, Kuantan, Pahang, Malaysia by XRF.

	Fe_2O_3	**Al_2O_3**	**TiO_2**	**SiO_2**	**F**
Chemical composition (wt%)	44.6643	34.1519	9.6104	8.3937	1.8378
	SO_3	**Na_2O**	**MnO**	**P_2O_5**	**ZrO_2**
	0.2709	0.2289	0.2241	0.2212	0.1803
	Cr_2O_3	**CuO**			
	0.1300	0.0866			

3.1.2. X-ray diffraction (XRD) analysis result

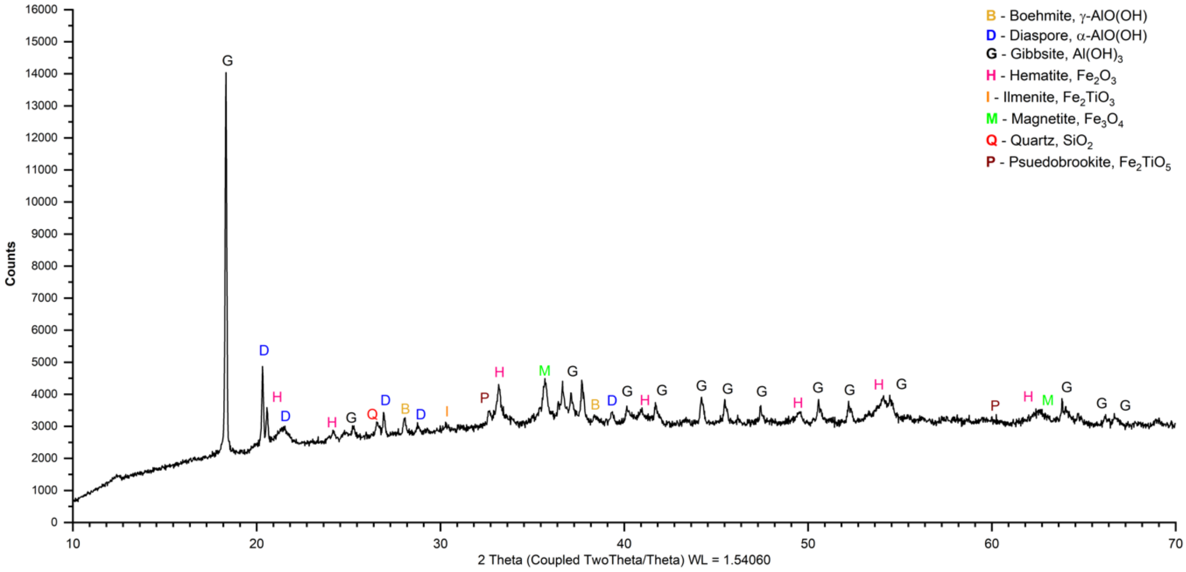


Figure 4. Mineral phases of RB from Felda Bukit Goh, Kuantan, Pahang, Malaysia by XRD.

The result of the XRD analysis on the RB is illustrated in Figure 4. Eight mineral phases were identified, such as boehmite, diaspore, gibbsite, hematite, ilmenite, magnetite, quartz and psuedobrookite. Commonly, gibbsite, $Al(OH)_3$, is the predominant mineral found in bauxite ore, and it is the main resource for alumina production.

3.1.3. Rare earth elements (REE) contents in methods 1 and 2

It is noted that Sc does not belong to any of the REE groups as the chemical properties of Sc are different from those of the other REE, but are often associated with REE. However, in this study the Sc group together with La to Eu in the light rare earth elements (LREE), whereas Y, Gd to Lu are classified as heavy rare earth elements (HREE) [37]. Tables 2a and 2b show the results of REE concentrations using the same RB but a different ICPMS model and brand. The data in the tables are also plotted in Figure 5. The decision to send the sample to another vendor was because the ICPMS UMP was under maintenance and each experiment was conducted a different year. Even so, the pattern of the result was quite similar, where the LREE was more abundant than the HREE, the Ce was the major element in LREE, whereas Y was the major element in HREE. The total rare earth elements (TREE) from Table 2a were higher than Table 2b, which were 344.53 mg/L and 231.43 mg/L, respectively. This difference was because of the sample preparation used. For ICPMS BPT, the solid sample was not digested but prepared in a fused bed method meanwhile, the sample preparation for ICPMS UMP was based on the open digestion method.

Table 2a. REE concentrations of RB in M1 from Felda Bukit Goh, Kuantan, Pahang by ICPMS, BPT.

	Sc	**La**	**Ce**	**Pr**	**Nd**	**Sm**	**Eu**		
LREE (mg/L)	60.00	62.10	104.00	14.90	64.90	8.80	4.01		
					318.71				
	Y	**Gd**	**Tb**	**Dy**	**Ho**	**Er**	**Tm**	**Yb**	**Lu**
HREE (mg/L)	11.30	7.84	0.71	3.08	0.56	1.06	0.15	0.98	0.14
					25.82				
TREE (mg/L)					**344.53**				

Table 2b. REE concentrations of RB in M2 from Felda Bukit Goh, Kuantan, Pahang by ICPMS, UMP.

	Sc	**La**	**Ce**	**Pr**	**Nd**	**Sm**	**Eu**		
LREE (mg/L)	14.50	40.80	74.50	9.70	40.40	8.20	2.10		
					190.20				
	Y	**Gd**	**Tb**	**Dy**	**Ho**	**Er**	**Tm**	**Yb**	**Lu**
HREE (mg/L)	20.40	6.50	0.94	5.50	1.02	3.03	0.45	2.98	0.42
					41.23				
TREE (mg/L)					**231.43**				

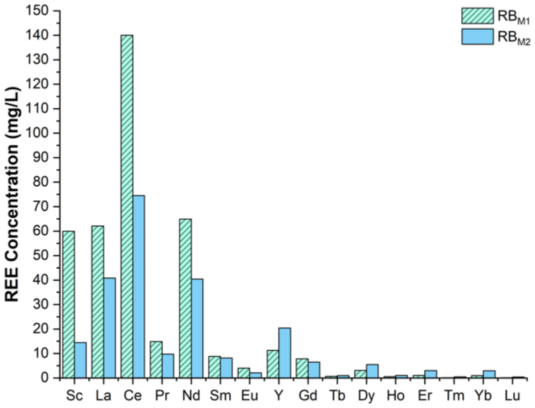


Figure 5. REE concentrations of RB_{M1} and RB_{M2}.

3.2. Comparison Methods of REE Extraction

3.2.1. REE concentrations after roasting and magnetic separation in M1

The concentrations of REE after roasting and magnetic separation are shown in Figure 6a. The TREE of BRS_{M1}, BRM_{M1} and $BRNM_{M1}$ detected were 301.31 mg/L, 275.97 mg/L and 300.18 mg/L, respectively, which are considered to have slight changes. However, compared with the RB, approximately 12.5% of the TREE was lost after roasting in BRS_{M1}. Among the REE concentrations, Ce was found to be the highest loss, about 36% followed by samarium (Sm) and europium (Eu), about 31% each. The REE loss may be due to volatilization or decomposition of the minerals for instance cerium (III) acetate hydrate decomposed around 250°C to 550°C [38]. In addition, after roasting, the Fe_2O_3 content increased from 44.66 wt% to 51.32 wt% similar to TiO_2 content, whereas the Al_2O_3 and SiO_2 contents decreased as displayed in Figure 6b. The gibbsite minerals started to decompose at around 220ºC [39] which may affect the content reduction. In Figure 6c, after roasting some of the hematite minerals in RB (Figure 4) were converted to magnetite minerals in BRS_{M1}.

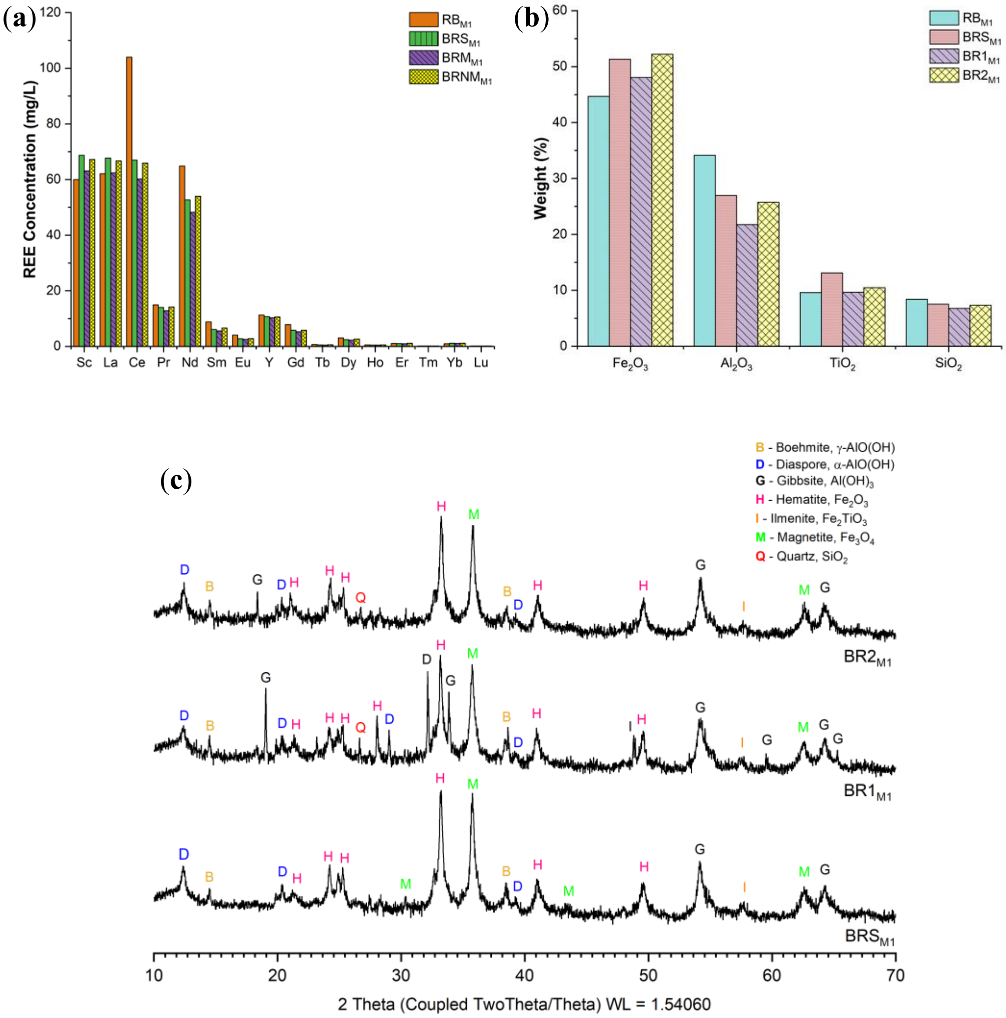


Figure 6. (a) REE concentrations of RB_{M1}, BRS_{M1}, BRM_{M1} and $BRNM_{M1}$ **(b)** major compounds in RB_{M1} and BRS_{M1} **(c)** mineral phases in BRS_{M1}, $BR1_{M1}$ and $BR2_{M1}$.

3.2.2. REE concentrations after Bayer process and acid cracking in M1 and M2

The concentrations of TREE after the Bayer process and acid cracking in M1 and M2 are shown in Figures 7a and 7b, respectively. In M1, the TREE of $BR1_{M1}$ detected was at 346.81 mg/L and $BR2_{M1}$ at 319.79 mg/L, this indicates that performing the Bayer process in M1 will not affect the REE concentrations but will only target the removal of alumina. The result also shows that increments of TREE about 16%, whereas after acid cracking some of the REE may have been leached out into $PS2_{M1}$ leading to some reduction of REE concentrations. Even after the Bayer process and acid cracking the Fe_2O_3 contents were above 48 wt% (Figure 6b), whereas other major compounds also showed similar trends. As for the mineral phases, no major changes were observed in $BR1_{M1}$ and $BR2_{M2}$ however the concentration of the minerals (1100 to 1600 counts) were decreased (Figure 6c). For M2, it was observed that after the Bayer process and acid cracking about 48% and 80% of TREE in $BR1_{M2}$ (119.34 mg/L) and $BR2_{M2}$ (46.08 mg/L) were declining, respectively. It was suspected that some of the REEs leached out in the $PS1_{M2}$ as before the Bayer process the REE may be loss during roasting and magnetic separation. REE are paramagnetic under room temperature [40] which requires high current intensity for separation however it may associate with other oxide minerals such as iron oxide in the bauxite. After acid cracking the TREE in $PS2_{M2}$ detected around 20.65 mg/L where most of it was Ce. The purpose of acid cracking was to liberate the REE that may interlock within the mineral host.

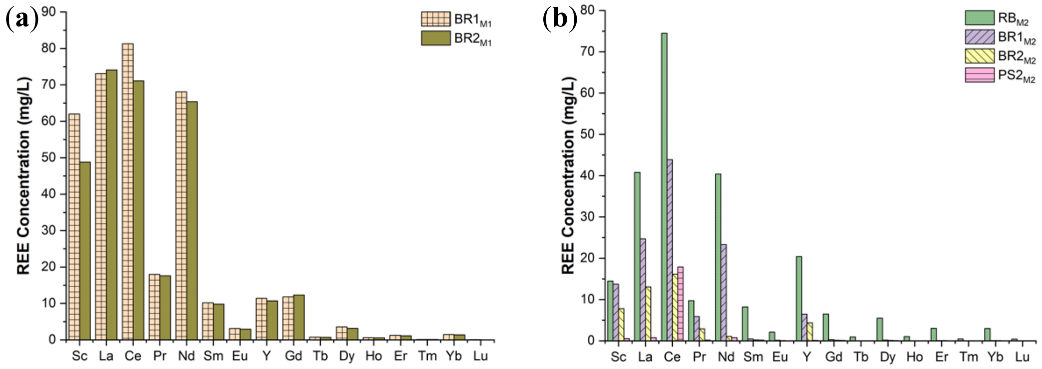


Figure 7. REE concentrations of **(a)** BR1$_{M1}$ and BR2$_{M1}$ **(b)** RB$_{M2}$, BR1$_{M2}$, BR2$_{M2}$ and PS2$_{M2}$.

Several studies have demonstrated that using H_2SO_4 as pre-treatment before REE leaching can enhance the REE extraction such as Sc up to 80% [41] with purity of 40 wt. % [42] however a high iron content is also associated together with the Sc extraction. Note that, after acid cracking with the addition of H_2O_2, the liquid texture of BR2$_{M1}$ became too viscous compared to without H_2O_2 was observed in BR2$_{M2}$. Moreover, the BR2$_{M2}$ became too hard as well as intact after drying and required mortar crushing before the next process was implemented. The reaction of H_2O_2 on concentrated H_2SO_4 produces highly activated and oxidizing peroxymonosulfuric acid (H_2SO_5), also refers as Caro's acid [43]. This may result in the REE contents remaining in the BR. According to Alan *et al.* [44] the presence of H_2SO_4 will lead to the production of titanium peroxide sulphate ($TiOSO_4$) and with mixed of H_2O_2 it can form the soluble compound TiO_2SO_4. In this present study, the concentration of H_2O_2 is way too high than Alan and co-workers demonstrated, perhaps this may contribute to the formation of REE complex compounds that hinder the REE leaching performance.

3.2.3. REE concentrations after leaching in M1 and M2

The concentrations of REE, major compounds and mineral phases after leaching in M1 and M2 are illustrated in Figure 8. In Figure 8a, the TREE in BR3$_{M1}$, BR4$_{M1}$ and BR5$_{M1}$ were decreased after multistage of leaching at 338.92 mg/L, 272.33 mg/L and 269.08 mg/L, respectively. Even so, most of the REE was unable to be leached out and remained in BR, in fact, the TREE in PS3$_{M1}$, PS4$_{M1}$ and PS5$_{M1}$ were also decreased at 1.60 mg/L, 0.56 mg/L and 0.31 mg/L, respectively (Figure 8b). On the contrary, in M2 the TREE of PS3$_{M2}$ (1.01 mg/L) detected was 1.66 times higher than BR3$_{M2}$ (0.61 mg/L) where Nd and Ce were most leached as shown in Figure 8c. After leaching it was also observed that all the four major impurities contents remained in BR$_{M1}$ increased in each leaching stage as shown in Figure 8d. The lixiviant may not dissolve directly the iron oxide minerals into the PS but may assist the precipitation. As evident that the Fe_2O_3 contents in the BR increased from 45.31 wt% to 50.98 wt%, this perhaps contributed by the reaction of $(NH_4)_2SO_4$ with iron oxide minerals in BR to form magnetite, Fe_3O_4 [45] or ammonium jarosite, $(NH4)Fe_3(SO_4)_2(OH)_6$ [46]. The pattern of the mineral phases of the BR illustrated in Figure 8e seemed to be no major changes yet the concentrations were slightly higher (1000 to 2000 counts) than in the previous residues (Figure 6c).

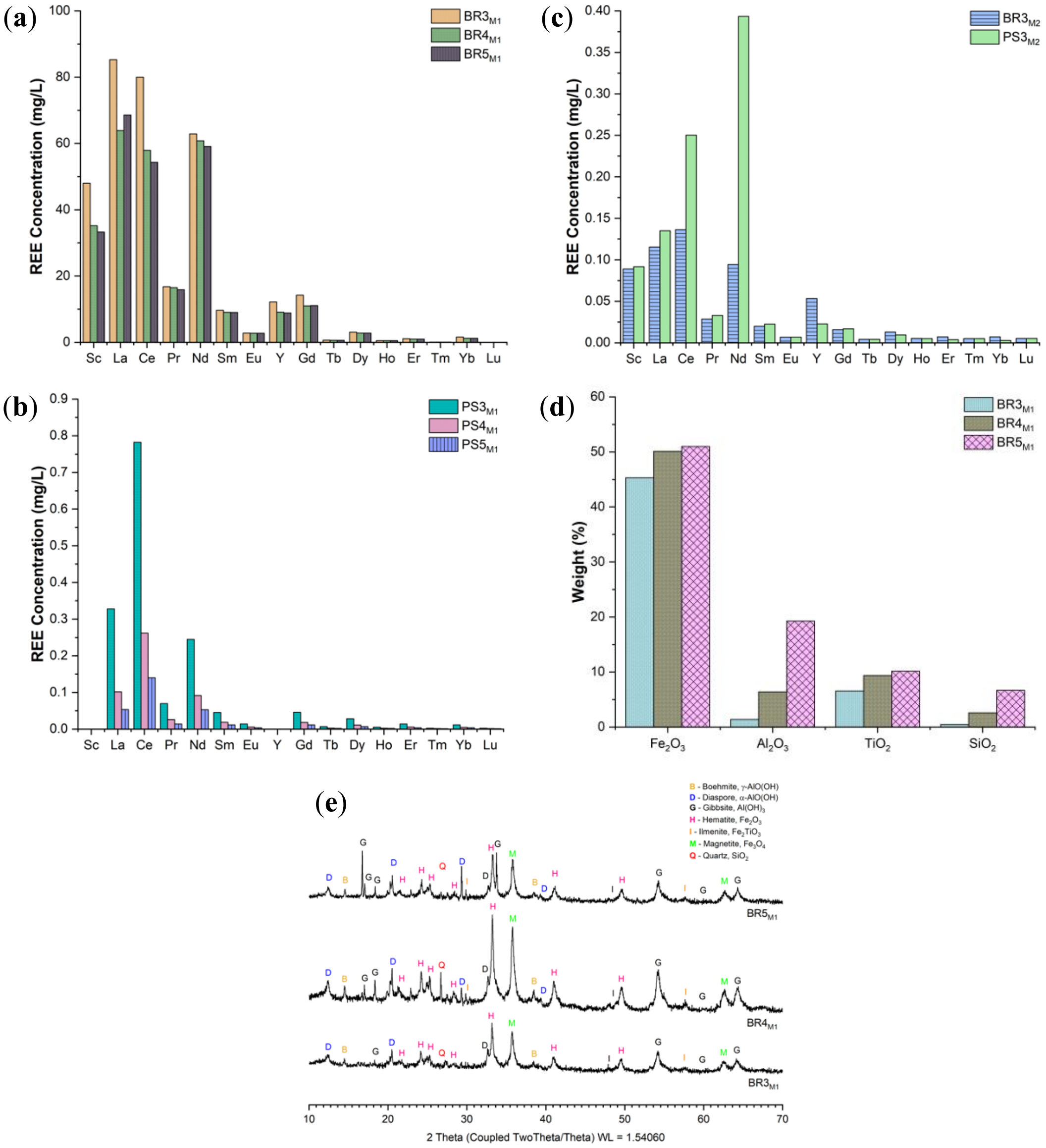


Figure 8. REE concentrations of **(a)** BR3$_{M1}$, BR4$_{M1}$ and BR5$_{M1}$ **(b)** PS3$_{M1}$, PS4$_{M1}$ and PS5$_{M1}$ **(c)** BR3$_{M2}$ and PS3$_{M2}$ **(d)** major compounds in BR3$_{M1}$, BR4$_{M1}$ and BR5$_{M1}$ **(e)** mineral phases in BR3$_{M1}$, BR4$_{M1}$ and BR5$_{M1}$.

3.2.4. REE concentrations after precipitation in M1 and M2

The concentrations of REE, major compounds and mineral phases after precipitation in M1 and M2 are presented in Figure 9. From Figure 9a, the Sc dominantly precipitated in each precipitation stage around 1.71 mg/L to 2.45 mg/L, followed by Ce, La, Y, Nd and Sm. Note that the Sc concentrations in the PS of M1 were not included in the analysis, so the concentration may be high in the PS3$_{M1}$, PS4$_{M1}$ and PS5$_{M1}$. Meanwhile, in M2, the concentration of REE specific to Ce and Ne from REOx was higher compared to the rest as shown in Figure 9b. The concentration of Ce through the precipitated Ce in the form of cerium oxalate, $Ce_2(C_2O_4)_3$ was up to 702.62 mg/L alone in REOx1$_{M2}$, whereas the Nd was detected at 21.50 mg/L and 33.00 mg/L in REOx1$_{M2}$ and REOx2$_{M2}$, respectively. In contrast, the other REEs (Figure 9c) were detected at lower than 0.5 mg/L. The findings here revealed that Ce and Nd tend to precipitate as compared to Sc and other REE. Without the presence of impurities (e.g., Fe^{2+}, Fe^{3+}, Al^{3+} and Ca^{2+}) cerium oxalate precipitate recovery can be attained as high as 56% when the pH increased from 1.5 to 2.5 [47] and during the precipitation process, the final pH recorded was between 1.8. to 1.9. Even so, the presence of impurities as displayed in Figure 9d can affect the REE recovery. In addition, note that in this study pH solution was not adjusted thus, using a high concentration of oxalic acid perhaps can elevate the REOx precipitation as reported elsewhere [48].

After precipitation, it was observed that the Fe_2O_3 was not detected nevertheless, the impurities such as SiO_2, Al_2O_3, P_2O_5 and CaO were the four major compounds that co-precipitated together with REOx (Figure 9d). Referring to its phase minerals in Figure 9e, the types of impurities that co-exist during precipitation were dominated by silicate-based minerals namely potassium aluminum silicate ($KAlSi_4$), clinochlore ($(Mg, Fe, Al)_6(Si, Al)_4O_{10}(OH)_8$) and diopside ($CaMgSiO_6$) with high concentration (24000 to 145000 counts) than RB. Besides that, whewellite ($CaC_2O_4.H_2O$) and phosphorus oxide (P_4O_7) compounds were also detected.

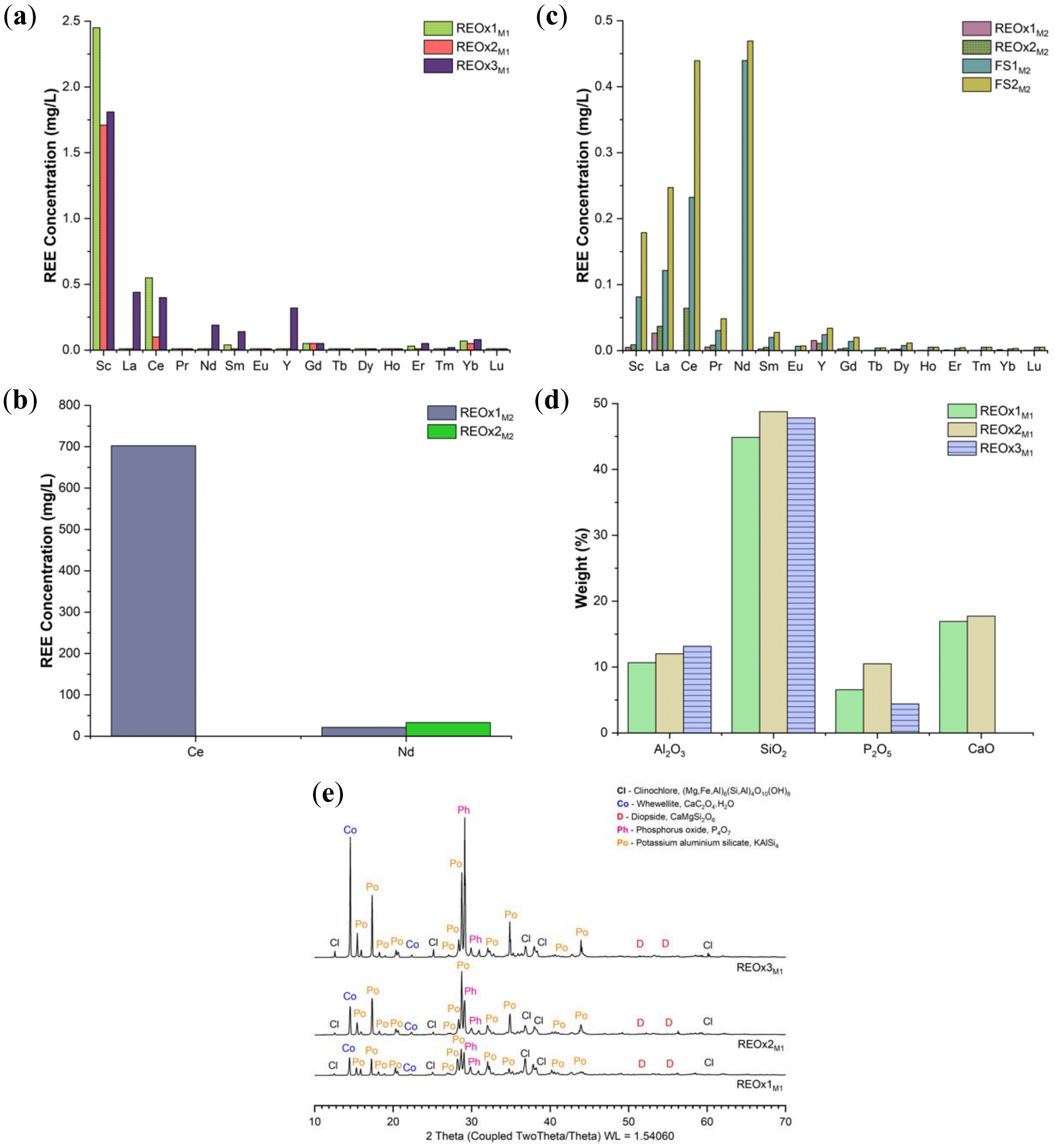


Figure 9. REE concentrations of **(a)** REOx1$_{M1}$, REOx2$_{M1}$ and REOx3$_{M1}$ **(b)** REOx1$_{M2}$, REOx2$_{M2}$, FS1$_{M2}$ and FS2$_{M2}$ **(c)** Ce and Nd from REOx1$_{M2}$ and REOx2$_{M2}$ **(d)** major minerals in REOx1$_{M1}$, REOx2$_{M1}$ and REOx3$_{M1}$ **(e)** mineral phases in REOx1$_{M1}$, REOx2$_{M1}$ and REOx3$_{M1}$.

3.2.5. REE recoveries in M1 and M2

The recoveries of REE by groups and the dominant elements as well as the element's purity percentage are displayed in Figure 10. Among the PS in M1, the LREE, HREE and TREE recoveries in PS3$_{M1}$ showed the highest at 0.91%, 0.73% and 0.89%, respectively (Figure 10a). For PS4$_{M1}$ the REE recoveries were about the same at 0.30%, meanwhile 0.17% for HREE and 0.14% for both LREE and TREE in PS5$_{M1}$. Overall, the recoveries of TREE in REOx in M1 show between 0.04 to 0.08% and among the REOx, the LREE and HREE presented the highest in REOx3 at 0.07% and

0.14%, respectively. On the other hand, in M2 the LREE, HREE and TREE recoveries of PS2$_{\mathrm{M2}}$ achieved up to 99.03%, 22.24% and 94.40%, correspondingly, whereas in PS3$_{\mathrm{M3}}$ the LREE, HREE and TREE recoveries attained at 4.54%, 5.69% and 4.61%, correspondingly as illustrated in Figure 10b. Additionally, the recoveries of LREE and TREE in REOx1$_{\mathrm{M2}}$ were at 24.95% and 23.45%, respectively, while the HREE only showed around 0.01%. For REOx2$_{\mathrm{M2}}$ the recoveries of LREE, HREE and TREE were 8.88%, 0.08% and 8.35%, respectively. In terms of element recovery of Ce and Nd (Figure 10c), the Ce and Nd in REOx1$_{\mathrm{M2}}$ can be recovered through precipitation as high as 61.86% and 3.56%, respectively. As for REOx2$_{\mathrm{M2}}$, Nd can be recovered up to 45.53% however, the Ce recovered was too low at 0.04%. The element purity was also calculated based on the overall REOx in M2 as presented in Figure 10d. The Ce purity was achieved up to 92.78%, whereas Nd purity at 7.20%.

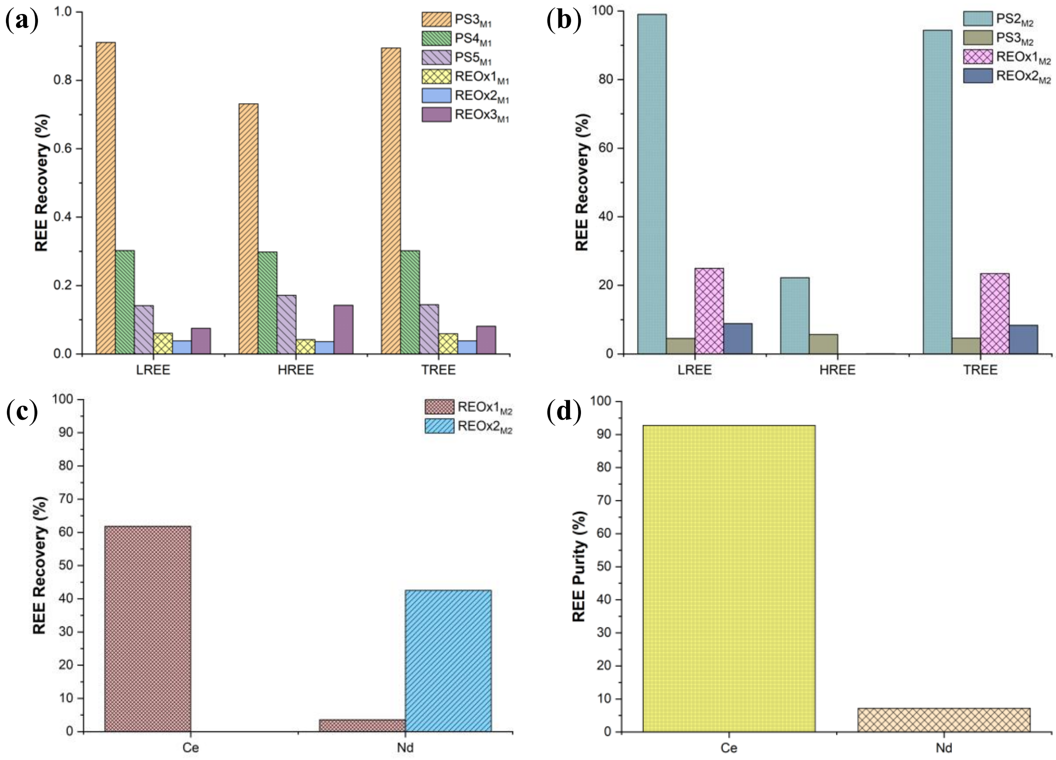


Figure 10. REE recovery of **(a)** PS3$_{\mathrm{M1}}$, PS4$_{\mathrm{M1}}$, PS5, REOx1$_{\mathrm{M1}}$, REOx2$_{\mathrm{M1}}$ and REOx3$_{\mathrm{M1}}$ **(b)** PS2$_{\mathrm{M2}}$, PS3$_{\mathrm{M2}}$, REOx1$_{\mathrm{M2}}$ and REOx2$_{\mathrm{M2}}$; **(c)** Ce and Nd from REOx1$_{\mathrm{M2}}$ and REOx2$_{\mathrm{M2}}$ **(d)** purity of Ce and Nd in REOx$_{\mathrm{M2}}$.

3.3. Comparison of other REE extraction methods from bauxite

The comparison of the proposed method with other REE extraction from bauxite ore is presented in Table 3. From this table, it shows that the results attained from the present study with other reported studies were quite low in terms of REE wt.%. However, in terms of the % recovery the study finding was comparable, as more than 90% of Ce can be recovered. The parameter of each process has not been fully optimized as this result is considered as preliminary results. Further investigation can be done for optimization.

Table 3. Comparison of the proposed method with other REE extraction methods from bauxite ore.

Country	Raw material	Pre-treatments	Leaching	Precipitation	REE Recovery	Reference
Malaysia	Bauxite ore	Roasting, magnetic separation, Bayer process, acid cracking using H_2SO_4	$(NH_4)_2SO_4$ at room temperature	$C_2H_2O_4$	92.78 % Ce (0.0703 wt. %) 7.20 % Nd (0.0055 wt. %)	Present study
China	Bauxite residue	Roasting with $(NH_4)_2SO_4$	Ultrapure H_2O	-	> 90% Sc	[25]
Indonesia	Low-grade bauxite ore	Magnetic separation	$C_2H_2O_4.2H_2O$	-	0.006 wt.% for La, Ce and Y	[32]
India	Red mud	-	Bioleaching using *Gluconobacter oxydans*	-	83% Sc	[49]
Greece	Bauxite residue	Reductive smelting	High-pressure acid leaching using HCl and H_2SO_4	-	90 wt.% Sc (H_2SO_4 at 150°C) 80 wt.% Sc (HCl at 120°C) < 20 wt.% Y, La and Nd (H_2SO_4) > 95 wt.% Y, La and Nd (HCl)	[50]
Indonesia	Low-grade bauxite ore	Roasting	$C_2H_2O_4.2H_2O$	Na_2SO_4 and Na_3PO_4	$C_2H_2O_4.2H_2O$-Na_2SO_4 1.55 wt.% La 0.48 wt.% Ce 0.02 wt.% Y $C_2H_2O_4.2H_2O$ - Na_3PO_4 0.14 wt.% La, 0.17 wt.% Ce and 0.02 wt.% Y	[51]
Canada	Bauxite residue	Microwave	H_2SO_4,	-	64.2 wt.% Sc 78.7 wt.% Nd	[52]
Greece	Bauxite residue	Roasting with H_2SO_4	Agitated and non-agitated Leaching using H_2SO_4	-	> 60wt.% Sc > 80wt.% other REE 60 wt.% Sc	[53]

4. Conclusions

Pre-treatments and leaching processes were performed to investigate the effect of REE leaching performances from Malaysian bauxite. In M1, the pre-treatment via acid cracking with H_2O_2 demonstrated that REE leaching was hindered by the formation of REE complex compounds, resulting in most of the REE contents remaining in BR. Overall, the REE recovery recorded was 1.34% when leached via $(NH_4)_2SO_4$, whereas only 0.17% of REOx can be recovered via $C_2H_2O_4$ precipitation. As for M2, the pre-treatment via acid cracking without H_2O_2 revealed that REE was leachable with REE recovery up to 99.0%, while the REE can be recovered during the precipitation process as high as 31.80%. This study indications that in the absence of H_2O_2 the REE can be easily leached out rather than introducing H_2O_2 into the process. The $(NH_4)_2SO_4$ potentially can be utilized as the lixiviant for REE leaching, as highly concentrated acid, together with roasting, is usually practiced in REE extraction from BR. The present study also shows that instead of Sc, Ce was the predominant element among the REE and can be purified up to 92%. A study of this selective precipitation can be conducted in the future. Performing the Bayer process for alumina extraction is imperative as bauxite is the main ore for aluminum production as well as reducing the disturbance of the REE extraction. Based on these findings, the M2 showed a promising method compared to M1. Even so, the parameters demonstrated herein were not yet fully optimized thus showing only certain REEs that can be leached out. Optimization each of the process parameters can be conducted in the future to achieve a better REE extraction performance.

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Conflicts of Interest

The authors declare no conflict of interest regarding the research and publication of this article.

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