




Recovery of Calcium from Reaction Fly Ash

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Abstract: Reaction fly ash contains a large number of harmful substances, so it is usually solidified and buried in landfills. To improve the problem of insufficient landfill space, this study recovers CaOH from reaction fly ash to achieve mass and volume reduction. The leachate obtained by leaching the reaction fly ash with de-ionized water and 2N hydrochloric acid was used in the experiments, respectively. The volume reduction with 2N hydrochloric acid had better performance than de-ionized water, representing more than 90%. The leaching efficiency of Ca reached 21.06% with de-ionized water for 20 min at a condition of 25 °C and 7 mL/g pump density. The chemical precipitation with NaOH was conducted immediately after the completion of the leaching experiment, with a precipitation efficiency of CaOH reaching 98.55%. The leaching efficiency of Ca reached 70.26% with 2N hydrochloric acid for 30 min at a condition of 25 °C and 10 mL/g pump density. The chemical precipitation with NaOH and ion exchange with IRC748 were conducted, respectively, after the completion of the leaching experiment. After two precipitations, the precipitation efficiency of CaOH was 99.93%. The precipitation efficiency and purity of Ca after ion exchange separation were 99.90% and 98.91%, respectively. This work provided an effective approach to recover CaOH from reaction fly ash and accomplished volume reduction at the same time.

Keywords: fly ash; hydrometallurgy; calcium recovery; ion exchange; chemical precipitation



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1. Introduction

With the world's population growth and economic development, we have produced a large amount of Municipal solid waste (MSW). At present, the global urban MSW production is about 2.01 billion tons per year, and it is expected that the MSW production will increase to 3.40 billion tons by 2050 [1,2]. MSW generally consists of two types of waste. The first mainly comes from plants and wet litter [3]. The other is dominated by recycled materials such as paper, cardboard, plastic, glass and metal [4].

Many waste treatment technologies are available today, including composting, recycling and incineration. Among these treatment technologies, incineration can reduce the volume of MSW by 85–90% and the mass by 80% [5–7]. Therefore, incineration has gradually become a method for dealing with MSW.

The biggest disadvantage of incinerated municipal solid waste is the formation of toxic and harmful by-products. The incineration process produces a large amount of municipal solid waste incineration fly ash (MSWI-FA) (fly ash accounts for about 3–15% of the total waste incinerated), bottom ashes (BA) [8], and toxic organic compounds such as polychlorinated dibenzodioxins and polychlorinated dibenzofurans [9]. Heavy elements in MSWI-FA are easily leached [10], leading to contamination of soil and groundwater. In addition, MSWI-FA can also spread through the air, causing environmental pollution [11]. Therefore, how to deal with fly ash is a major issue.

At present, MSWI-FA treatment methods include carbonization, washing, leaching, chemical stabilization, electrocoagulation, and electrodialysis [12–19]. The treatment method of waste incineration fly ash in Taiwan is the stabilization/solidification (S/S) process. However, the S/S process usually increases the mass of fly ash [20], resulting in a serious shortage of landfill capacity. After considering the disposal methods that will not

harm the environment and recycle resources. It is believed that volume reduction of fly ash can best solve the problem of insufficient landfill. At present, fly ash volume reduction and weight reduction research are underway. Quina et al. [21] investigated the leaching efficiency of heavy metals in fly ash. Finally, the dissolution efficiency of heavy metals will change with the change in liquid-to-solid ratio and pH value. Xia et al. [22] investigated the leaching efficiency of calcium compounds. The leaching efficiency will vary with pH value, time, and temperature. In terms of leaching acid solution selection, Weibel et al. [23] proposed that hydrochloric acid is the best leaching solution, which can dissolve most of the metals in fly ash. There has been extensive research on leaching fly ash using various reagents. Whereas most of the literature focuses on the leaching of metals from MSWI-FA and discusses the effects of experimental conditions such as reaction time, temperature, leaching reagents used, and liquid-to-solid ratio.

In this study, a new process for reducing the mass of waste incineration fly ash and recovering metals is proposed. Fly ash usually contains metals such as Ca, Al, Cd, Cu, Fe, Mn, Pb, and Zn, among which calcium metal has the highest content. Therefore, if the calcium in fly ash can be recovered, the quality and volume of fly ash can be effectively reduced. In this study, three different methods were used to recover metallic calcium from fly ash and the volume reduction of each method was discussed. In addition, we look for the most efficient way to recycle calcium.

2. Materials and Methods

2.1. Materials

The materials used in this study were the fly ash collected from Tainan Waste Incineration Plant (Tainan, Taiwan). Table 1 lists the metal composition and content of fly ash. A chelating cation exchange resin IRC748 obtained from Rohm and Haas was used in this study.

Table 1. Metal content in reaction fly ash.

Element	Ca	Al	Cd	Cu	Fe	Mn	Pb	Zn
weight percentage (%)	48.11	0.602	0.014	0.051	0.633	0.022	0.139	0.775

Hydrochloric acid (HCl) used in the experiments was 37% purity, without further purification (from Shin-Shin Chemical Co. (Tainan, Taiwan)). All the aqueous solutions were prepared in deionized water.

2.2. Fly Ash Washing

Fly ash was washed with deionized water and HCl solutions for comparison. Five grams of fly ash were added to a predetermined volume of washing liquid. Liquid-to-solid ratios (L/S) in mL/g were set at 5, 7, 10, and 15, respectively. Concentrations of HCl were set at 1, 2, and 3N for comparison. The mixture was stirred at 25 °C, 750 rpm for 10–60 min, then filtered. The filtrate was analyzed for Ca^{2+} and heavy metals to investigate the efficiency of washing.

2.3. Resin and Column Pretreatment

The chelating cation exchange resin IRC748 was repeatedly washed with de-ionized water. After washing until it was clean, it was soaked in a 1N HCl aqueous solution of three times the resin volume for 1 to 2 h. De-ionized water was then used to wash the resin again until the pH was 7, after which a column experiment could be conducted. The IRC748 showed a strong affinity for heavy metals. A column with a length of 15 cm and a diameter of 1.5 cm containing 12 mL of resin was used for adsorption experiments.

2.4. Analyst

The concentration of metal ions in the solutions obtained after washing experiments, ion exchange, and chemical precipitation was determined by using Atomic Absorption Spec-

troscopy (AAS, PinAAcle 900F AA Spectrometer, PerkinElmer Inc., Waltham, MA, USA). The pH of the solution was monitored by a pH meter (PL-700PVS, Dogger Science, Taipei, Taiwan). To control the temperature during the experiment, a thermostatic bath with magnetic stirring (Shin-Kwang Precision Industry Ltd., New Taipei, Taiwan) was employed. The crystal structure of the precipitation was analyzed by X-ray diffractometry (XRD, DX-2600, Dandong, China).

2.5. Separation of Ion Exchange and Chemical Precipitation

As shown in Figure 1, the experiment plan in this investigation included chemical precipitation with NaOH and ion exchange with IRC748, which were conducted after the completion of the leaching experiment. The selective precipitation process used NaOH as the reagent to directly separate calcium from the other elements. The IRC748 resin effectively adsorbed heavy metals. The NaOH proved to be an efficient precipitation reagent, resulting in a $\text{Ca}(\text{OH})_2$ precipitate.

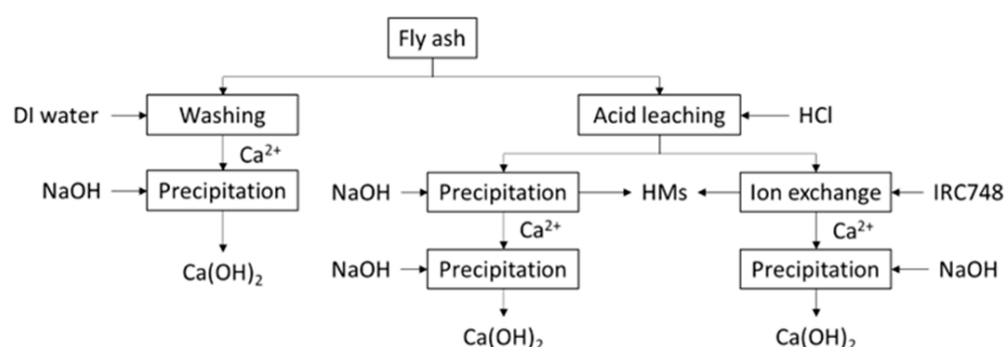


Figure 1. Proposed flowsheet for recovering Ca from municipal Solid Waste (MSW).

2.6. Analysis of Purity

After metal separation experiments, all powders were analyzed for purity. The formula for purity analysis is shown in Formula 1. In the formula, C_a represents the concentration of metal ions (mg/L), V represents the quantitative volume (mL), D represents the dilution factor, F represents the mass conversion factor and W represents the sample weight (g).

Formulas for Purity Analysis:

$$\text{Purity\%} = \frac{C_a \times V \times D \times F}{W \times 10^6} \times 100\% \quad (1)$$

3. Results and Discussion

3.1. Washing with Deionized Water

The effects of the liquid-to-solid ratio on the calcium efficiency of washing and the reaction time on the calcium efficiency of washing were investigated in this section.

As shown in Figure 2a, when the liquid-to-solid ratio was 5 mL/g, the insufficient amount of liquid led to uneven stirring and a low water washing rate. When the liquid-to-solid ratio was 7 mL/g, the water washing rate reached the highest. However, adding liquid again cannot effectively increase the washing rate. Probably, the solubility equilibrium for the metal compounds present was the limiting factor [24]. As more water was added, the fly ash leached more metals. However, Ca^{2+} and anions were also more prone to re-precipitation.

Figure 2b showed the effect of time on the washing rate. The washing rate was the highest in 20 min. If the washing time was prolonged, the washing rate would decrease due to the re-precipitation of salts. This result was similar to that of Chen et al. [25]. This was attributed to the formation of $\text{Ca}^{2+}\text{X}^{2-}$ precipitation when the concentrations of anions and Ca^{2+} were higher than the K_{sp} value.

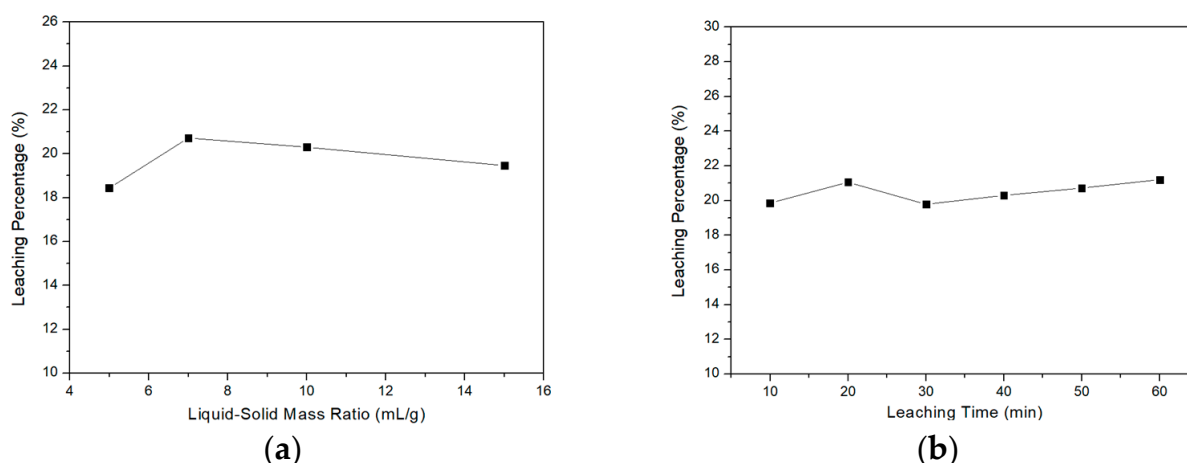


Figure 2. The effect of (a) liquid-to-solid ratio and (b) time on the efficiency of calcium washing.

As shown in Figure 3, the volume reduction of washing increased from 31.65% to 39.13% as the liquid-to-solid ratio increased from 5 to 15 mL/g. Considering the water consumption and washing efficiency, the liquid-to-solid ratio of 7 mL/g was selected as the optimal parameter.

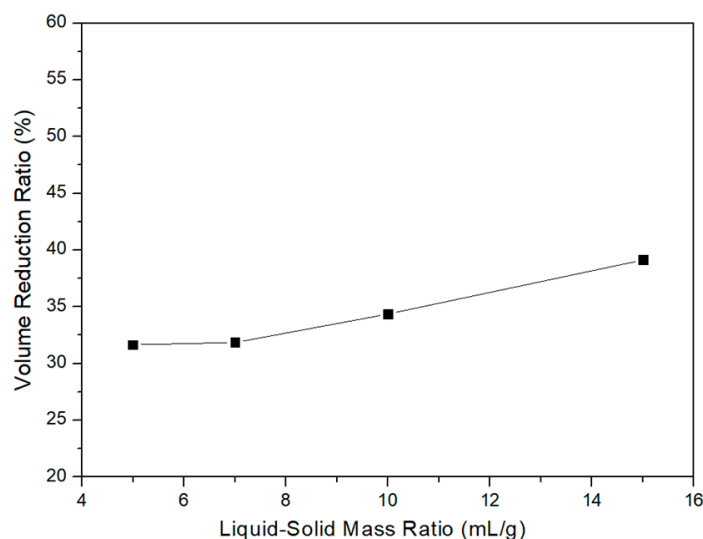


Figure 3. Volume reduction ratio of water washing.

According to the above results, the optimum washing conditions were the liquid-to-solid ratio of 7 mL/g and a reaction time of 20 min. The calcium efficiency of washing of 21.06% and a volume reduction of 31.85%. This result was similar to other research results [26–28].

3.2. Acid Leaching

The effects of acid concentration, liquid-to-solid ratio, and reaction time on the calcium efficiency of leaching were investigated in this section.

Most of the K, Na, Al, Ca, Cd, Cu, Pb, and Zn-containing compounds dissolved at pH = 1 when the HCl solution was used, leading to an ash weight reduction of ~80% [29]. Therefore, this study investigated the effect of hydrochloric acid on fly ash leaching. As shown in Figure 4a, the leaching efficiency of calcium increased from 60.72% to 72.02% with the concentration of hydrochloric acid increasing from 1N to 2N and stagnated thereafter with a further increase in the concentration of hydrochloric acid. This result showed that 2N hydrochloric acid leached all metals that can be leached.

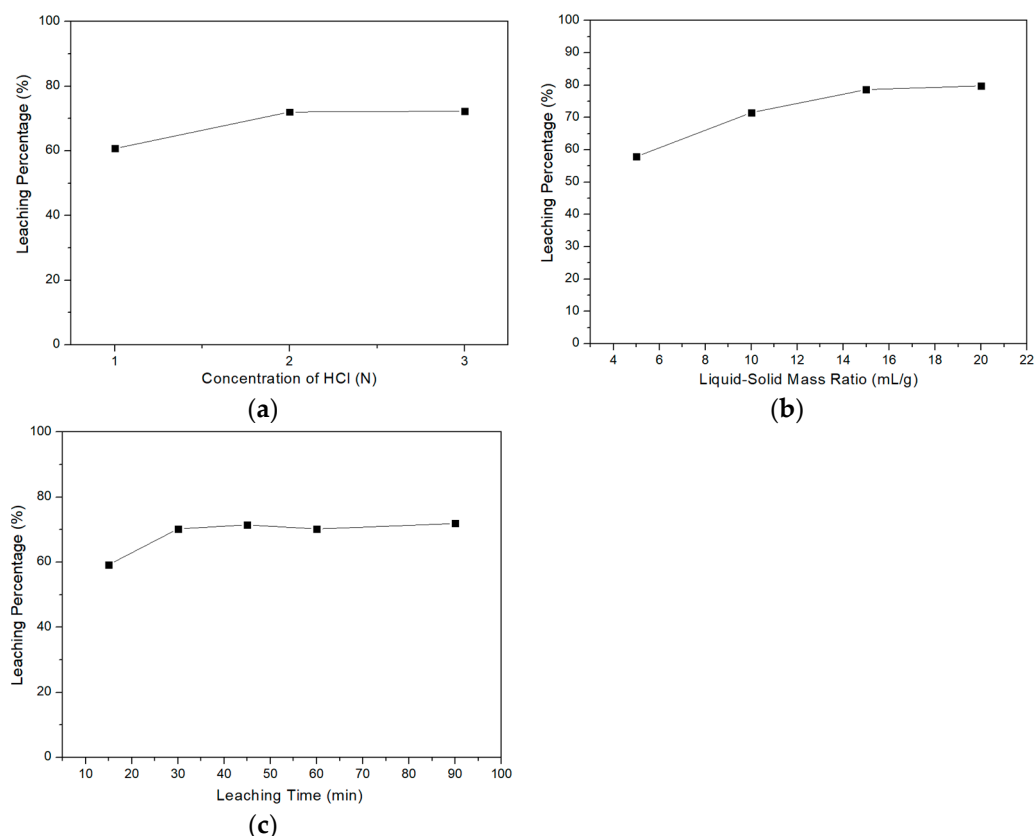


Figure 4. The influence of (a) acid concentration, (b) liquid-to-solid ratio, and (c) time on the efficiency of calcium leaching with hydrochloric acid.

As shown in Figure 4b, the leaching efficiency of calcium increased from 57.88% to 71.49% with the liquid-to-solid ratio increasing from 5 to 10 mL/g. When the liquid-to-solid ratio was raised to 15 mL/g, the leaching efficiency was not significantly improved. While a higher L/S ratio was beneficial to the dissolution of salt in fly ash, considering both leaching efficiency and volume reduction, the recommended L/S ratio was 10 mL/g.

The effect of time on the leaching efficiency is shown in Figure 4c. The leaching efficiency was rather slow, taking about 30 min to complete the reaction. The salts did not re-precipitate during acid leaching. Therefore, the leaching rate did not decrease when the reaction time was extended.

As shown in Figure 5, the volume reduction of leaching increased from 70.26% to 90.44% as the liquid-to-solid ratio increased from 5 to 10 mL/g. Considering the water consumption and leaching efficiency, the liquid-to-solid ratio of 10 mL/g was selected as the optimal parameter.

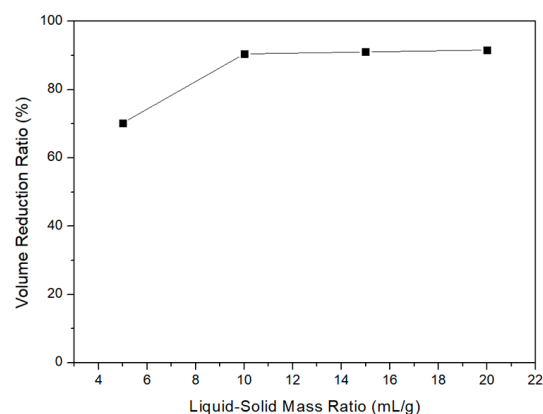


Figure 5. Volume reduction ratio of leaching with hydrochloric acid.

According to the above results. When the acid concentration was 2N, the liquid-to-solid ratio was 10 mL/g and the time was 30 min, the calcium leaching efficiency reached 71.49% and the volume reduction reached 90.44%. Compared with the study of Seham et al. [30], we leached more calcium under similar acid concentrations, L/S ratio, and time. Furthermore, we obtain good volume reduction ratios.

3.3. Selective Chemical Precipitation of Water Washing

After the leaching experiment, we discuss how to recover calcium effectively and obtain the highest purity. The additional amount of NaOH played a significant role in Ca precipitation when the reaction temperature and time were fixed at 25 °C and 30 min, respectively. The Ca precipitation reached 98.55% (Figure 6) with the NaOH to Ca molar ratio fixed at 2:1. As shown in Figure 7, XRD analysis confirmed the presence of a calcium hydroxide crystal phase remaining after precipitation. However, after purity analysis, it was found that the purity of calcium hydroxide was only 66.73%. Therefore, the follow-up will discuss the process of separating other metals and then proceeding to precipitation. It is desirable to obtain calcium of high purity.

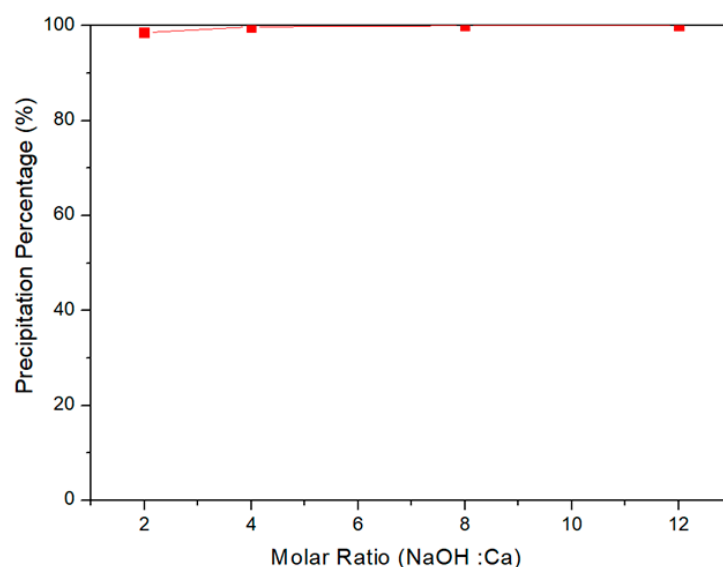


Figure 6. Influence of molar ratio in water washing on the efficiency of calcium precipitation.

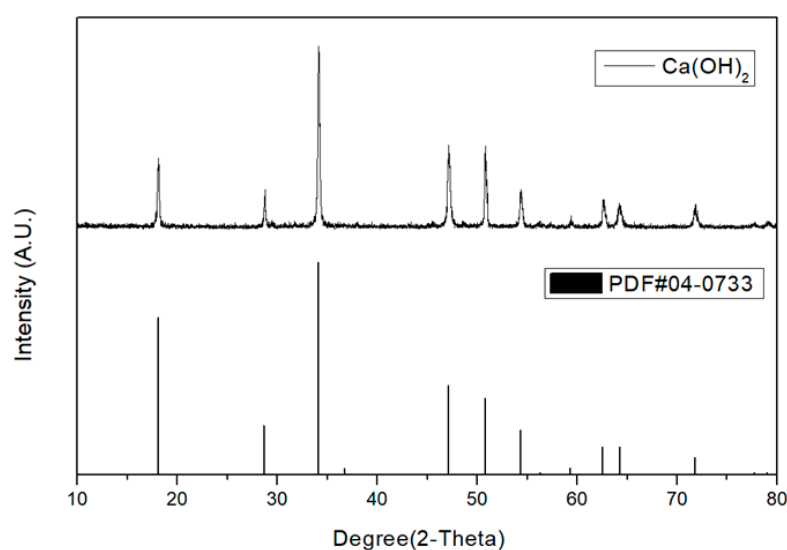


Figure 7. XRD pattern of sodium hydroxide precipitated from washing liquid.

3.4. Selective Chemical Precipitation of Acid Leaching

The selective chemical precipitation of acid leaching was mainly divided into two parts: The first part was the precipitation of other metals in the leachate. As shown in Figure 8, at a temperature of 25 °C and a rotation speed of 750rpm for 30 min, sodium hydroxide was slowly added to adjust the pH value of the solution. When the pH value was raised from 4 to 10, the co-precipitation rate of calcium increased from 8% to 15%, and the precipitation rate of other metals at pH 10 was 99.99%. This result was the same as that of Monhemius et al. [31]. Calcium was heavily precipitated at pH values above 12, whereas other metals were heavily precipitated at pH 10. Therefore, this study first adjusted the pH to 10 to precipitate other non-target metals.

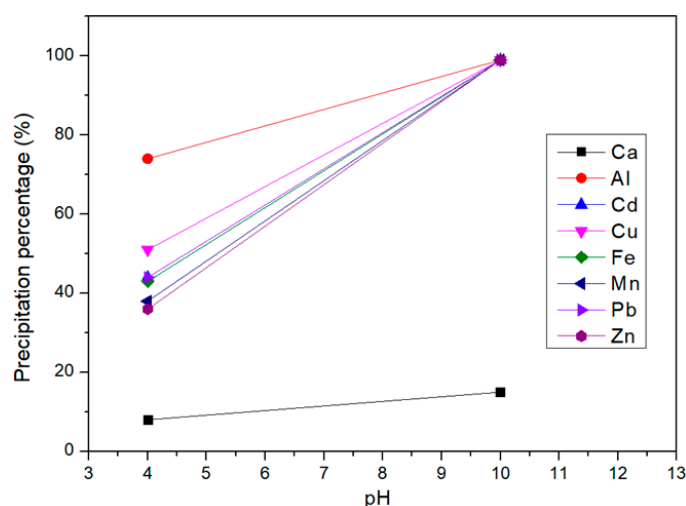


Figure 8. The influence of pH value in acid precipitation solution on the precipitation efficiency of various metal ions.

The second part was the influence of the molar ratio on the calcium leaching rate. It can be seen from Figure 9 that the calcium precipitation rate reached 99.93% when the molar ratio of NaOH to Ca was 2:1. When sodium hydroxide was added again, the calcium precipitation efficiency did not significantly improve. Therefore, a molar ratio of two was selected as the best precipitation parameter. The powder after precipitation was confirmed to be calcium hydroxide in the crystal phase by XRD analysis (Figure 10).

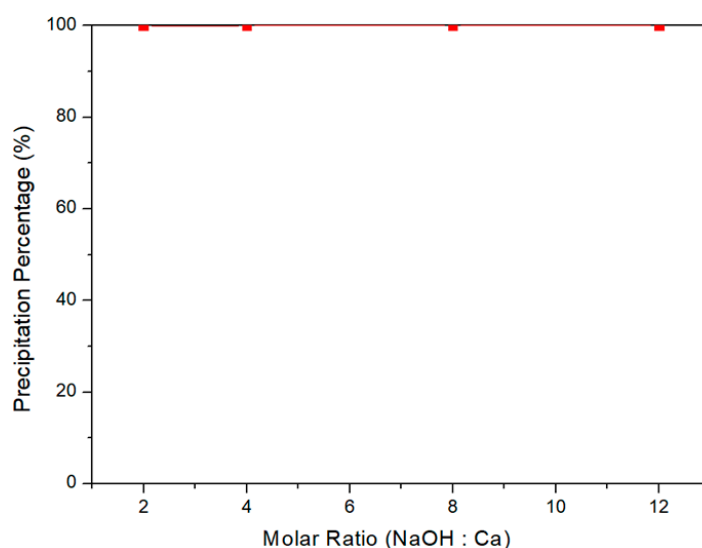


Figure 9. The influence of the molar ratio in an acid precipitation solution on the efficiency of calcium ion precipitation.

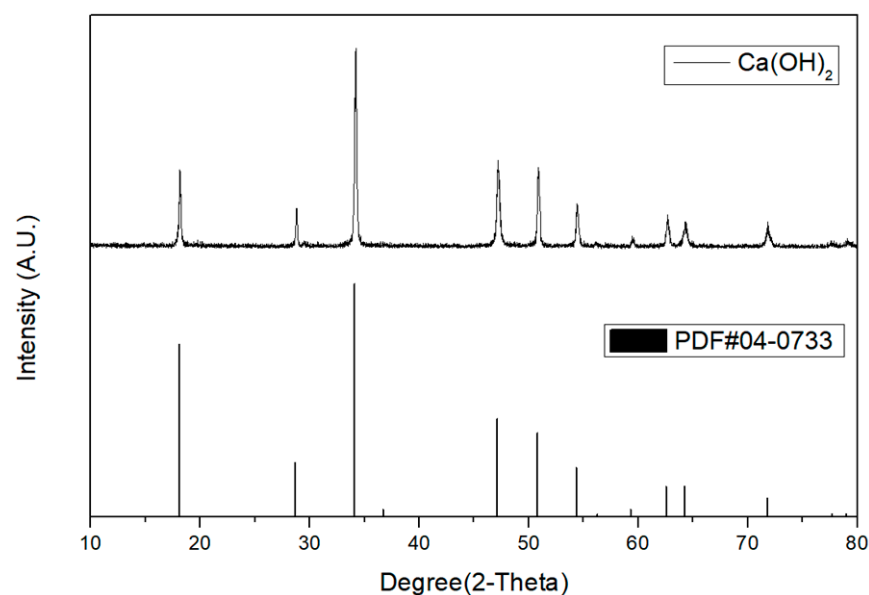


Figure 10. XRD pattern of calcium hydroxide precipitated from a precipitation solution.

This part has been separated by two stages of precipitation, but the purity of calcium was only 82.45%. The purity was still too low to be directly used in industry. Therefore, in the follow-up discussion, after removing non-target metals by ion exchange, precipitation was carried out.

3.5. Ion Exchange for Acid Leaching

According to the findings of Mendes et al. [32], we found that IRC748 was able to adsorb more non-target metals than M4195 and the adsorption amount of non-target metals was the highest at pH = 4. Therefore, we used IRC748 for the adsorption of non-target metals. IRC748 was used to adsorb non-target metals at pH 4, as shown in Figure 11. When the adsorption reached 15 B.V., some copper and manganese were detected, so 1 to 15 B.V. was collected as a calcium enrichment solution.

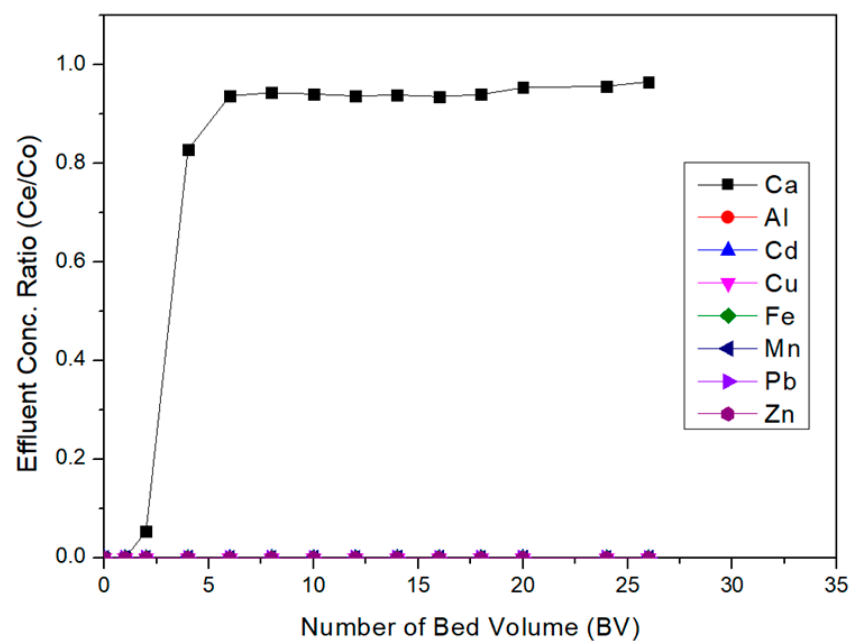


Figure 11. Adsorption curve of each metal with IRC748 of pH = 4.

In the second part, calcium ions in the remaining liquid were precipitated by sodium hydroxide, as shown in Figure 12. When the molar ratio was two, the precipitation rate of calcium reached 99.90%. If sodium hydroxide was added again, the precipitation rate of calcium did not increase significantly. Therefore, a molar ratio of two was selected as the best precipitation parameter. The powder remaining after precipitation was confirmed to be calcium hydroxide crystal phase by XRD analysis (Figure 13). After separating non-target metals by ion exchange, we successfully obtained calcium hydroxide with a purity of 98.91% by the precipitation method. This study was compared with that of Jang et al. [33]. We obtained similar precipitation efficiencies and purities. In addition, this study also explored the volume reduction rate of fly ash.

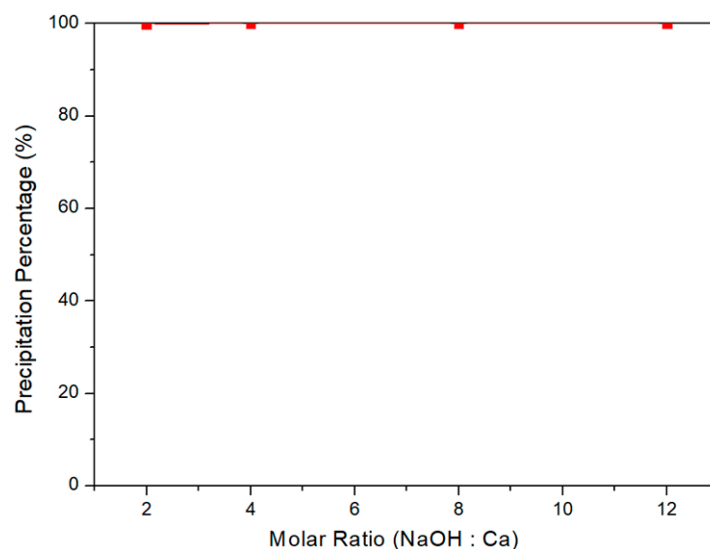


Figure 12. Influence of the molar ratio on the acid precipitation solution after ion exchange on the precipitation efficiency of calcium ions.

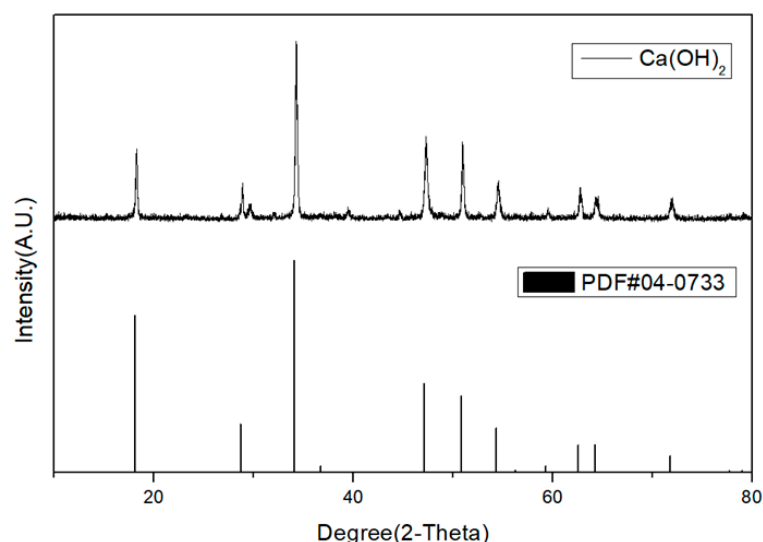


Figure 13. XRD pattern of calcium hydroxide precipitated from ion exchange remaining liquid.

4. Conclusions

First, leach with deionized water and hydrochloric acid. The optimal leaching rates of calcium were 21.06% and 70.26%. A study of calcium hydroxide precipitation would be carried out later. There were mainly three methods.

In the first method, the solution was directly subjected to calcium hydroxide precipitation after washing. The calcium precipitation rate was 98.55% and the purity was 66.73%.

The second method was to precipitate the non-target metals in the solution after acid leaching. Adjust the pH value to 10 to precipitate other non-target metals, and then adjust the molar ratio of sodium hydroxide to calcium to precipitate calcium hydroxide. The precipitation rate was 99.93% and the purity was 82.45%.

The third method was to leach the acid in the solution, adsorb other non-target metals by IRC748, and then adjust the molar ratio to precipitate calcium hydroxide. The precipitation rate was 99.90% and the purity was 98.91%.

Among these three methods, the third method was the best. We used hydrochloric acid for leaching, which can leach 70.26% of calcium and the volume reduction rate reached 90.44%. After conducting ion exchange to remove other heavy metals, the precipitation rate and purity of calcium hydroxide reached 99.90% and 98.91% respectively.

In order to solve the problem of excessive solidification volume of fly ash, in this study, leaching was used to reduce the volume of fly ash and successfully recovered high-purity sodium hydroxide from fly ash.

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