Leaching Behavior of Lithium from Bauxite Residue Using Acetic Acid

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Abstract

Bauxite residue (red mud) is an alkaline waste derived from the industrial process for extraction of alumina from bauxite ore. Some bauxite residues contain considerable concentrations of critical metals, for example, rare earth elements. Lithium in red mud was also reported as a potential resource. In the current study, a Li-rich red mud was leached using acetic acid to investigate the leaching behavior of lithium followed by neutralizing with hydrochloric or oxalic acid. By neutralizing red mud using diluted hydrochloric acid (0.01 mol/L), 58.04–60.27% of lithium was leached using 25% acetic acid at 95 °C for 60 min. In the meantime, more than 95% of sodium and 85% of calcium were also dissolved in the leachate solution. While oxalic acid (0.5 mol/L) was used to neutralize red mud prior to acetic acid leaching, lithium performed lower leaching efficiencies (42.41–46.88%) in comparison of using of hydrochloric acid–neutralized red mud. Nevertheless, these leaching efficiencies of lithium were close to that of calcium and much higher than those of sodium under the condition. In this study, 25% (v/v) of acetic acid, at 85 °C for 60 min, was demonstrated as optimum conditions for lithium extraction from oxalic acid–neutralized red mud using acetic acid. The results would be useful and of interest for lithium recovery and purification from red mud.

Keywords Lithium · Red mud · Leaching · Acetic acid · Oxalic acid

1 Introduction

Lithium resources are crucial raw materials for various industrial demands, and lithium plays a vital role in energy market and economy [1–3]. With the explosive development of lithium-ion battery in mobile phones, laptops, especially electric vehicles, the demand of lithium brings about a significant competition on lithium resources [1, 2]. Natural sources used for producing lithium can be grouped as two main categories, brines and minerals, such as spodumene and lepidolite [2]. In addition, lithium-bearing clays are also developed as future lithium sources. For example, Egyptian montmorillonite-type clays are bentonite clays included with 40–60% montmorillonite and Li_2O was as high as 1.2% in weight percent [4].

Spent lithium-ion battery waste is considered as the most prominent secondary source of lithium from the various secondary resources, and can be economically recycled [2, 5]. Owing to concomitant of raw ores, some industrial wastes contain large quantities of lithium and might be important lithium resources. For example, coal fly ash from northern China was reported for lithium recovery due to the markedly enrichment in lithium, approximately 0.2 wt% of Li_2O [6]. Another industrial waste, bauxite residue (BR), more known as red mud (RM), also contains lithium, which is associated in bauxite and transferred and enriched in red mud in the process of alumina refining. It is reported that lithium was hosted within the Li-bearing claystone in the bauxite deposit [7].

Bauxite residue is generated during the Bayer process of alumina recovery from bauxite ore. The chemical compositions of bauxite residue are associated with the alumina process and the raw bauxite ores. The broad ranges of contents are reflected in both the 6–8 main components and more than 50 trace elements. Red mud can be regarded as a "polymetallic raw material" or an "artificial ore." The main components of red mud, for example, aluminum [8], sodium [8], iron [9], and titanium [10], were extracted or separated as resources. The leaching behavior of these elements from red mud was

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investigated [11]. Meanwhile, trace valuable elements in red mud including rare earth elements [12, 13], scandium [14], gallium [11, 15], vanadium [11, 16], and lithium [17], were also reported as potential resources. Concentrations of lithium in different red mud samples were determined as a trace/minor component with a wide range from several tens to hundreds μg/g [18–20] depending on the raw material, bauxite ore [21]. It has been reported that red mud samples from China contain broad ranges of 6.0–224 μg/g of lithium [17].

Distribution and leachability of main or trace metals from red mud were reported under different conditions [22]. Unlike the other valuable trace metals [8–16, 23], seldom work focused on the lithium recovery from red mud. Metal release from red mud should be carefully assessed when red mud is alternatively used to neutralize [22]. The objective of the current study is to investigate the leaching behavior of lithium from a high Li-bearing red mud using acetic acid based on the previous work. Lithium can be well leached in the fourth extraction step of Tessier's sequential extraction procedure [17], which was based on a neutralization treatment using sodium acetate solution with a pH 5 adjusted by acetic acid. Various acids have been introduced to deal with red mud for neutralization treatment [24, 25]. In this study, before acetic acid leaching process, diluted hydrochloric acid and oxalic acid were respectively selected to neutralize the alkaline red mud sample and to remove the soluble metals like sodium and calcium. Meanwhile, effect parameters including acetic acid concentration, reaction temperature, and reaction time were investigated. Knowing the leaching behavior of lithium from red mud would be useful for lithium recovery and purification for further processing.

2 Materials and Methods

2.1 Raw Materials and Characterization Methods

The red mud sample used in this study was HN, a lower iron diaspore Bayer red mud obtained from Henan, China, as described in previous literatures [17, 26]. HN is one of the typical bauxite residues generated in Henan province, China, containing 224 μg/g of lithium. As reported previously [17], the main chemical composition of HN is listed as follows (wt%): Al_2O_3: 22.54, SiO_2: 19.70, Fe_2O_3: 9.69, CaO: 17.65, TiO_2: 6.82, Na_2O: 4.12, K_2O: 2.10, and MgO: 1.44.

Guaranteed reagent grade hydrochloric (36.0–38.0 wt/v %, Sinopharm Chemical Reagent Co., Ltd), analytical reagent grade oxalic (≥ 99.5 wt%, Tianjin Yongda Chemical Industrial Co., Ltd), and analytical reagent grade acetic (≥ 99.5 wt%, Tianjin Kaixin Chemical Industrial Co., Ltd) acids were used for neutralization or leaching process in this study.

Powder X-ray diffraction measurements were performed using a PANalytical Empyrean diffractometer with Cu Kα

radiation. Each sample was prepared by compaction into a silicon sample holder and a 2θ range between 5° and 70° was scanned.

2.2 Leaching Experiment Process

Raw bauxite residue of HN was dried at 80 °C for 24 h and then ground and sieved through a 100-mesh sifter for subsequent neutralization process.

2.2.1 Acid Neutralization Procedure

Hydrochloric acid and oxalic acid were used for HN red mud neutralization treatment, respectively. Specifically, 500 mL of 0.01 mol/L hydrochloric acid was prepared by adding 75 g HN red mud into a 1000mL conical flask. The reaction lasted for 60 min at room temperature accompanying with slight swing oscillation. After filtration and washing with water for 3 times, the pH of final leachate solution was measured (pH 8.4). The leaching residue was labeled as hydrochloric acid–neutralized HN. It was dried at 80 °C for 24 h and was ground homogeneously for acetic acid leaching procedure.

In the case of oxalic acid neutralization procedure, 500 mL of 0.5 mol/L oxalic acid was prepared by adding 75 g HN red mud into a 1000-mL conical flask. The reaction was conducted at 60 °C for 30 min using a thermostatic water bath with slight swing oscillation. After filtration and washing with water for 3 times, the pH of final leachate solution was measured (pH 7.5). The leaching residue obtained here was labeled as oxalic acid–neutralized HN. Similarly, oxalic acid–neutralized HN was also dried and ground for acetic acid leaching procedure. In addition, 1.0 g original HN sample was also leached with 6.7 mL oxalic acid of various concentrations (0.1, 0.25, 0.5, 0.75, and 1.0 mol/L) under the same condition, and the obtained leachate solutions were determined by ICP-OES.

2.2.2 Acetic Acid Leaching Procedure

Hydrochloric acid–neutralized HN and oxalic acid–neutralized HN were respectively leached using acetic acid solution. According to Tessier's sequential extraction procedure, fraction bound to Fe–Mn oxides was investigated using 0.03 M hydroxylamine hydrochloride ($HONH_2HCl$) in 25% (v/v) acetic acid [17]. To know the function of hydroxylamine hydrochloride, neutralized HN samples were leached using acetic acid solutions with different amount of hydroxylamine hydrochloride inside. The leaching procedure was performed using 2.0 g neutralized HN by adding 20 mL solution into a 50-mL centrifuge tube. Various concentrations of hydroxylamine hydrochloride (0, 0.02, 0.04, and 0.06 mol/L) were prepared in the solution of 25% (v/v) acetic acid. Reactions were

performed with a liquid-to-solid ratio of 10 mL: 1 g, at 95 °C on a thermostatic water bath with gentle swing oscillation for a period of 60 min.
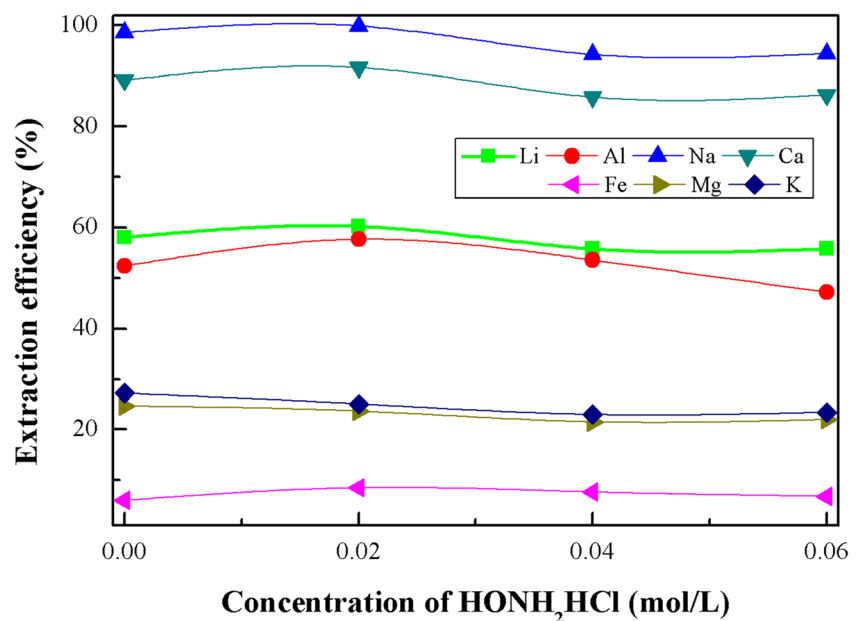
Oxalic acid–neutralized HN was also used to investigate experimental conditions of acetic acid concentration (without adding hydroxylamine hydrochloride), reaction temperature, and reaction time. Each group for condition experiments were conducted on a thermostatic water bath with slight swing oscillation with a liquid-to-solid ratio of 10 mL:1 g. Some details for each conditional experiment are as follows: Reactions for acetic acid concentrations tests were performed at 60 °C for 60 min and 1, 5, 15, 25, 35, and 50% (v/v) of acetic acid were investigated. Temperatures tests were 25, 45, 60, 75, 85, and 95 °C using 25% (v/v) acetic acid for a reaction period of 60 min. Reactions time was controlled for 5, 30, 60, 90, and 120 min using 25% (v/v) acetic acid leaching at 60 °C.

The leaching efficiency of a given element in this study was calculated according to the following equation.

$$\varepsilon\,(M,\%) = \frac{V \times c}{m \times w} \times 100 \tag{1}$$

where ε (M, %) represents the leaching efficiency of metal (including Li, Al, Na, Ca, Fe, Mg, and K); V (L) is the total volume of leaching solution merged with washing solution; c (g/L) is the concentration of metal (including Li, Al, Na, Ca, Fe, Mg, and K) ion in the solution; m (g) is the weight of the original HN red mud sample; and w (%) is the content of metal (including Li, Al, Na, Ca, Fe, Mg, and K) in the original HN red mud sample.

3 Results and Discussion

3.1 Hydrochloric Acid for Neutralization

Lithium in the red mud was thought to be existing as bound to Fe–Mn oxides [17], and hydroxylamine hydrochloride was used as a reductant in the acetic leaching process [17, 22]. To know the effect of the hydroxylamine hydrochloride, hydrochloric acid–neutralized HN was leached with 25% (v/v) acetic acid solution included a different amount of hydroxylamine hydrochloride. With changing the amount of hydroxylamine hydrochloride, the extraction efficiencies of lithium as well as the common main elements from red mud are shown in Fig. 1. As demonstrated in Fig. 1, the leaching efficiencies of lithium as well as the main elements from hydrochloric acid–neutralized HN did not change with increasing the amount of hydroxylamine hydrochloride. It can be inferred that hydroxylamine hydrochloride was not the contributing reagent for elements leaching. The results indicate that lithium in red mud may not be as Fe–Mn oxides form and can mainly be extracted by acetic acid solution.

Figure 1 also shows that approximately 60% lithium (58.04–60.27%) can be leached from hydrochloric acid–neutralized HN. The leaching efficiencies were obviously higher than that of HN (45%) from Tessier's sequential extraction procedure [17]. As previously reported, approximately 10% lithium was leached prior to acetic acid leaching as fraction exchangeable and bound to carbonates. Diluted hydrochloric acid used in this study only extracted 0.52% lithium in the process of neutralization treatment. Therefore, a higher

Fig. 1 Extraction of elements from hydrochloric acid–neutralized HN using 25% (v/v) acetic acid included different concentrations of HONH$_2$Cl (liquid-to-solid ratio of 10 mL:1 g, 95 °C, 1 h)

leaching efficiency of lithium was obtained based on hydrochloric acid–neutralized HN. However, with regard to the main elements, sodium followed by calcium performed a higher extraction efficiency than that of lithium under each condition (Fig. 1). The preferential leaching of sodium and calcium might bring difficulties to subsequent lithium separation.

XRD comparisons of original HN red mud, hydrochloric acid–neutralized HN, and acetic acid leaching residue from hydrochloric acid neutralized HN red mud are presented in Fig. 2. The main phases in HN red mud from the XRD peaks did not change after neutralization treatment using diluted hydrochloric acid. For example, sodium-bearing phases (such as hydroxycancrinite) and even carbonates were not dissolved. Peaks of hydroxycancrinite, calcite, and kotaite disappeared in the XRD pattern of the final residue after acetic acid leaching. The results indicate that sodium and calcium were not leached during the diluted hydrochloric acid neutralization process, and would be dissolved in the acetic acid leaching process. Therefore, the acetic acid leachate had a higher leaching efficiency of sodium and calcium.

Concentrated hydrochloric acid can dissolve iron, aluminum, and even titanium form red mud [27], while diluted hydrochloric acid was too mild to remove sodium or calcium, resulting in huge gaps between lithium and other main metals like sodium and calcium in the subsequent acetic acid leaching process. Therefore, oxalic acid was proposed to neutralize red mud to remove some of the main elements before acetic acid leaching.
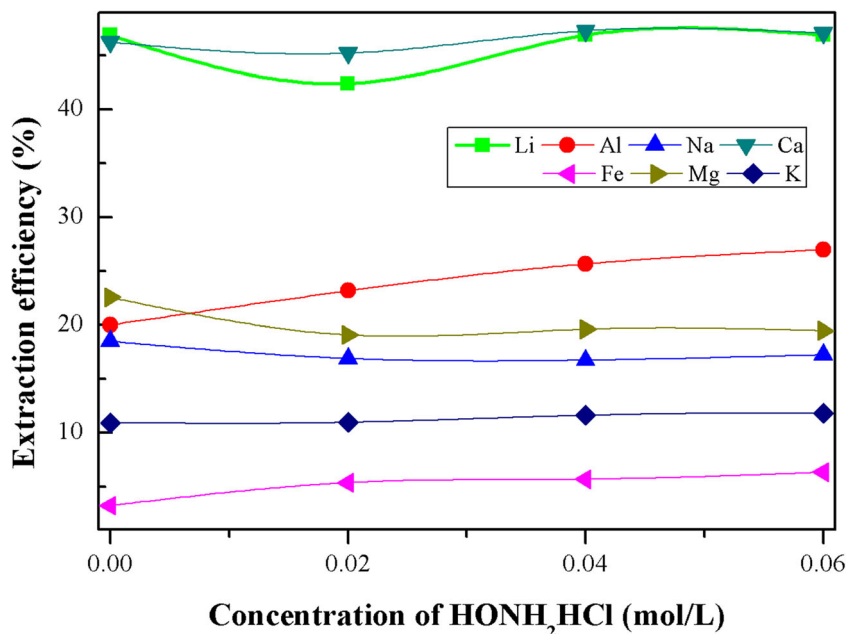
3.2 Oxalic Acid for Neutralization

Oxalic acid was attempted to neutralize HN red mud prior to acetic acid leaching process. To investigate the leaching behavior of lithium from oxalic acid–neutralized HN, acetic acid leaching process was conducted using 25% (v/v) acetic acid solution included a different amount of hydroxylamine hydrochloride. Figure 3 shows the leaching efficiencies of lithium from oxalic acid–neutralized HN. Lithium efficiencies ranged 42.41–46.88%, which were a bit lower than those from hydrochloric acid–neutralized HN as shown in Fig. 1. Lithium and the main elements' leaching efficiencies performed a narrow range with increasing the amount of hydroxylamine hydrochloride. As discussed above, it seems that hydroxylamine hydrochloride did not affect elements' leaching efficiencies.

It is worth mentioning that in the acetic acid leaching process based on oxalic acid–neutralized HN, most of the main elements, especially sodium and aluminum, performed lower leaching efficiencies in comparison to those based on hydrochloric acid–neutralized HN. This was ascribed to the proper acidity and the chelating ability that oxalic acid presented during the neutralization process [28]. Figure 4 shows XRD comparisons of original HN, oxalic acid–neutralized HN, and acetic acid leaching residue from oxalic acid–neutralized HN. After the oxalic acid neutralization process, hydroxycancrinite and calcite disappeared, and meanwhile, whewellite was generated. Whewellite could be obviously observed after acetic acid leaching process, implying that calcium in the form of whewellite will not enter into the acetic acid leachate together

Fig. 2 XRD patterns of (a) original HN red mud, (b) hydrochloric acid–neutralized HN, and (c) acetic acid leaching residue from hydrochloric acid–neutralized HN
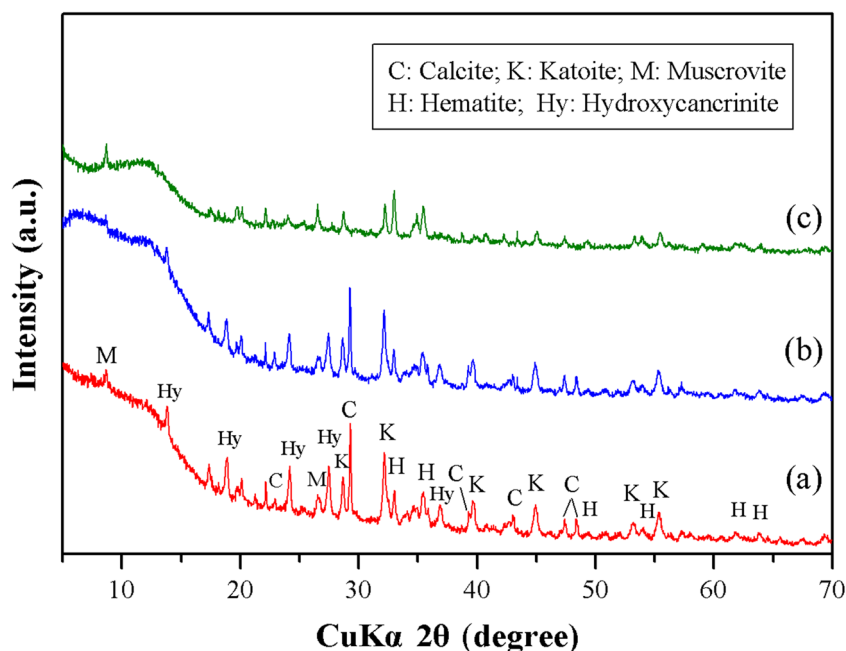
Fig. 3 Extraction of elements from oxalic acid–neutralized HN using 25% (v/v) acetic acid included different concentrations of $HONH_2Cl$ (liquid-to-solid ratio of 10 mL:1 g, 95 °C, 60 min)

with lithium. The above results suggest that oxalic acid can release sodium and aluminum from the solid phases into solution, and relatively reserved lithium.

To know metals' dissolution behavior from original HN in oxalic acid neutralization process, oxalic acid leaching experiments were conducted with different concentrations. Figure 5 shows the removal efficiencies of the elements from red mud using different oxalic acid concentrations. Oxalic acid can effectively remove sodium and its leaching efficiency can be higher than 60% when the concentration achieved 0.25 mol/L. With the oxalic acid concentration increased to 0.5 mol/L, more than 89% sodium can be dissolved. In the process of

changing acetic concentration from 0.25 to 0.5 mol/L, the loss of lithium was stable around 13.4%. However, further adding the oxalic acid concentration to 0.75 mol/L, the loss of lithium was near 50% and was not suitable for neutralization treatment. In terms of calcium, the amount of calcium extracted by oxalic acid was limited using different oxalic acid concentrations due to the insolubility of calcium oxalate. Calcium oxalate is insoluble in the acetic acid leaching process, which can be explained by the weak acidity of acetic acid. It can be concluded that oxalic acid is artfully used to treat red mud in this study for it is acidic enough for removal of sodium and can precipitate calcium.

Fig. 4 XRD patterns of (a) original HN red mud, (b) oxalic acid–neutralized HN, and (c) acetic acid leaching residue from oxalic acid–neutralized HN
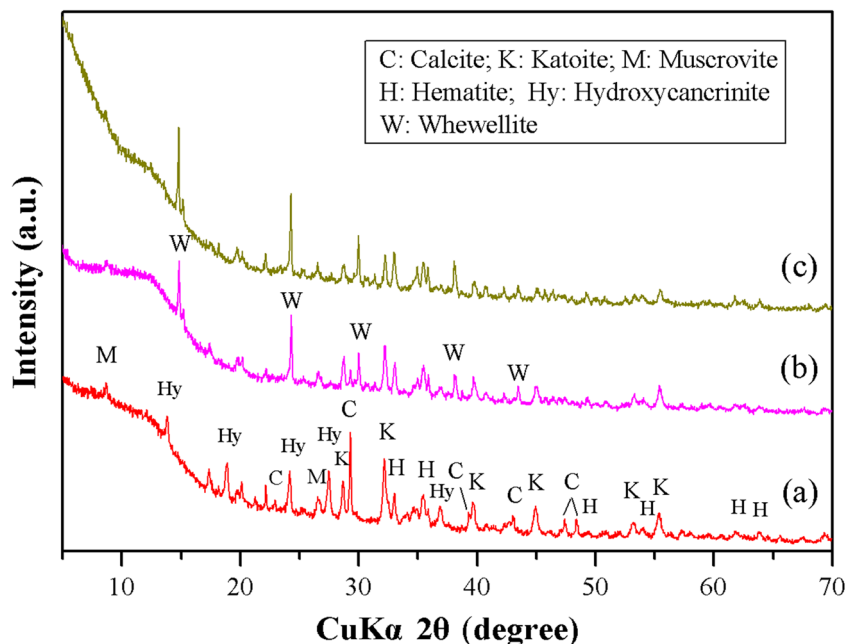
Fig. 5 Effect of oxalic acid concentration on extraction of metals from original HN red mud (liquid-to-solid ratio of 10 mL:1.5 g, 60 °C, 30 min)
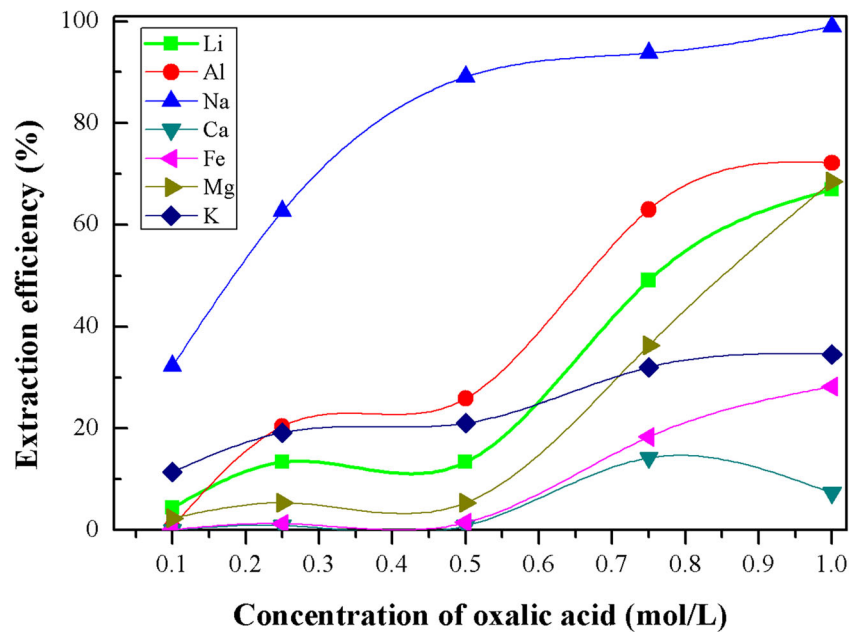

3.3 Effect of Reaction Temperature

Prior to acetic acid leaching process, oxalic acid pretreatment has since been demonstrated to be effective widen the gap of leaching efficiency between lithium and main elements from red mud. Meanwhile, hydroxylamine hydrochloride added in acetic acid solution does not take effect as discussed above. It is necessary to discuss the variation in leaching behavior under different experimental conditions, such as acetic acid concentration, reaction temperature, and reaction time.

The effect of reaction temperature was firstly investigated to compare the dissolution of metals from oxalic acid–neutralized HN. Figure 6 shows the variation in elements leaching behavior from oxalic acid–neutralized HN with leaching temperature of 25, 45, 60, 75, 85, and 95 °C. The results show that with respect to increase of reaction temperature, the dissolution of lithium and most of the investigated metals increased. Peculiarly, a sharp increase in lithium leaching was observed from 25 to 85 °C. A slight decline was also observed for all the metals when the temperature was over 85 °C, which is due to volatilization of acetic acid at high temperature. In this study, 85 °C was thought to be the optimal reaction temperature for lithium leaching.

Fig. 6 Effect of reaction temperature on extraction of metals from oxalic acid–neutralized HN (25% (v/v) acetic acid, liquid-to-solid ratio of 10 mL:1 g, 60 min)
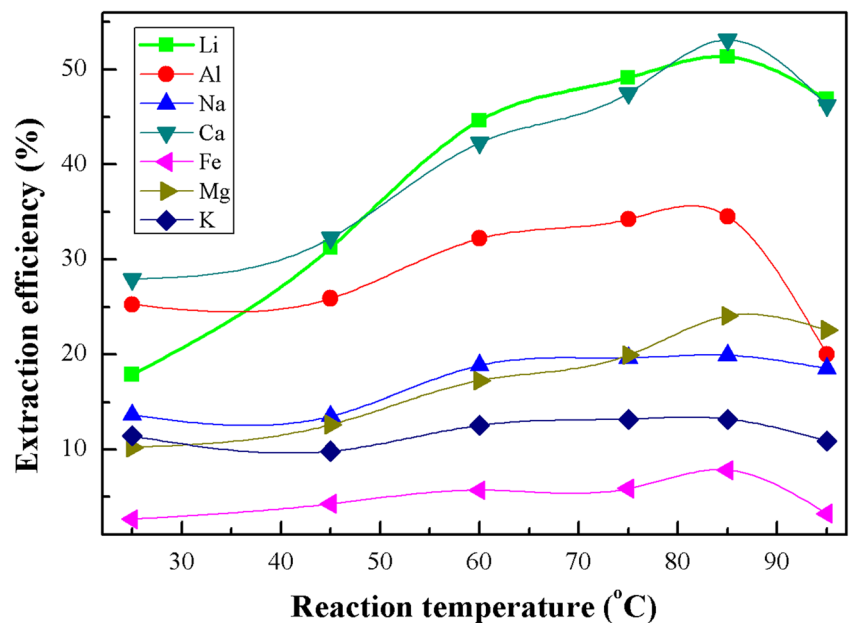
Fig. 7 Effect of acetic acid concentration on extraction of metals from oxalic acid–neutralized HN (liquid-to-solid ratio of 10 mL:1 g, 60 °C, 60 min)
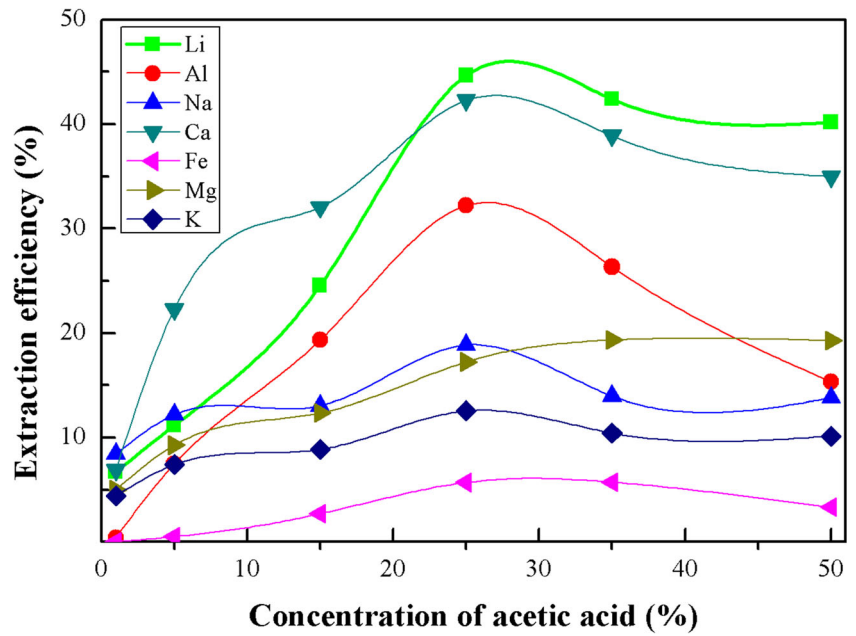
## 3.4 Effect of Acetic Acid Concentration

Figure 7 shows the variation in metals leaching behavior from oxalic acid–neutralized HN with acetic acid concentration of 1, 5, 15, 25, 35, and 50% (v/v). The results show that with respect to increase of acetic acid concentration, the dissolution of lithium and most of the investigated metals presented an increase and a decline as the concentration of acid exceeded 25% (v/v). This trend that the percentage of metals dissolved (such as Al and Fe) increased and then decreased with the increase of acetic acid concentration was consistent with the regularities reported by Rubinos and Barral [22]. It is possible

that high concentration of acetic acid dissolved zeolitic aluminosilicates in red mud, and new phases generated would precipitate part of the released metal including lithium. Therefore, the concentration of acetic acid befitting to extract lithium was suggested as 25% (v/v).

3.5 Effect of Reaction Time

Overall, the dissolution of metals from oxalic acid–neutralized HN with respect to time performed a stable trend, expectation of lithium and calcium showing a slightly increase leaching when time went as shown in Fig. 8. At 60 min, the leaching

Fig. 8 Effect of reaction time on extraction of elements from oxalic acid–neutralized HN (25% (v/v) acetic acid, liquid-to-solid ratio of 10 mL:1 g, 60 °C)
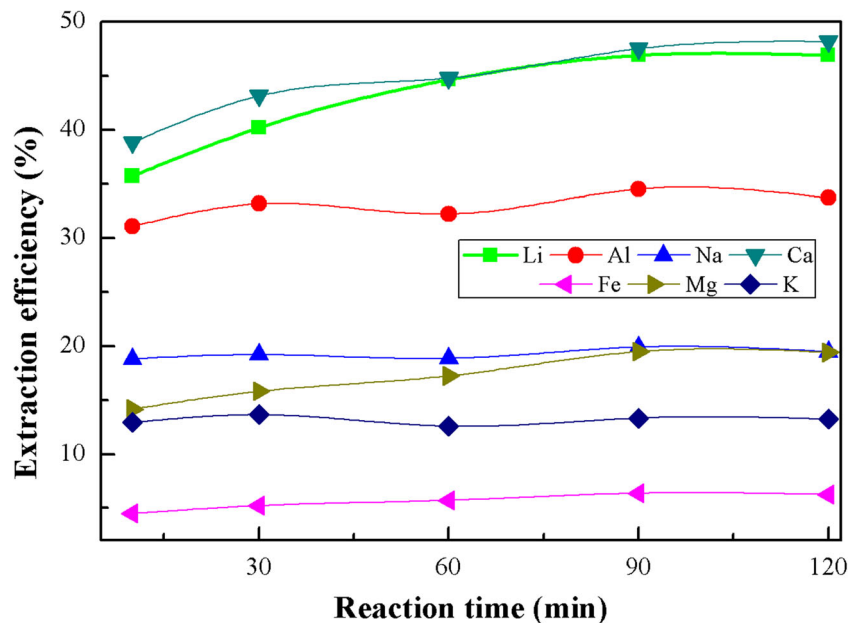
efficiency of lithium was 44.64%, close to calcium 44.79%, while at 90 min, calcium (47.50%) dissolved a little more than that of lithium (46.88%). Therefore, 60 min was suggested to be an optimal condition for lithium leaching in this study.

4 Conclusions

The leaching behavior of lithium from HN red mud using acetic acid was found to be significantly dependent on the neutralization pretreatment. Oxalic acid was effective to remove sodium and make lithium perform a higher leaching efficiency in comparison of diluted hydrochloric acid. While 0.5 mol/L oxalic acid was used to neutralize red mud prior to acetic acid leaching, lithium performed leaching efficiencies of 42.41–46.88% which were close to that of calcium and much higher than those of other main elements like sodium, aluminum, iron, and magnesium. The findings are instructive for subsequent recovery, separation, and purification of lithium from red mud. Concentration of acetic acid was the important factor and hydroxylamine hydrochloride did not work during the leaching process. The optimum conditions for extraction of lithium from oxalic acid–neutralized HN red mud were found to be: acetic acid concentration of 25% (v/v), 85 °C, 60 min.

Compliance with Ethical Standards

Conflict of Interest The authors declare that there is no conflict of interest.

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