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LEACHING OF LEAD FROM SPHALERITE CONCENTRATE DIRECT LEACHING RESIDUE BY HYDROCHLORIC ACID AND SODIUM CHLORIDE FOR PREPARATION OF LEAD OXIDES

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Abstract

The purpose of this study was to select and propose an applicable method for extracting lead from sphalerite concentrate direct leaching residue. A large number of experiments were conducted to extract lead from sphalerite concentrate direct leaching residue by hydrochloric acid and sodium chloride solution as leachates. The main optimum parameters were determined, such as a liquid-solid ratio of 17.5-1, a reaction temperature of 85 °C, an initial hydrochloric acid concentration of 1.3 mol/L, an initial sodium chloride concentration of 300 g/L, and a reaction time of 60 min. Ninety-five percent of the zinc, 96.0% of the iron, and 93.7% of the lead were extracted into leachate at the optimum conditions. The lead in the leachate was in the form of $[PbCl_4]^{2-}$. After the leachate was purified to remove impurities, it was converted into lead oxalate by sodium oxalate as a precipitant. Finally, lead oxalate was decomposed to obtain lead oxide powders via a high-temperature calcination process.

Keywords: *Sphalerite concentrate direct leaching residue; Hydrochloric acid and sodium chloride; Lead oxalate; Lead oxide; Leaching*

1. Introduction

Lead and zinc are indispensably important metal elements in the development of the national economy and are widely used in human life and industrial production [1]. The lead and zinc waste products generated during the smelting process are classified as dangerous, and they also pose a great threat to public health and ecosystems [2]. Traditional open-air stockpiling or simple landfill treatment of lead and zinc waste residue occupies a large area of land. Moreover, toxic elements in waste residue, such as copper, cadmium, lead, and zinc are easily released into the natural environment by natural weather cycles and leaching processes, causing serious pollution and potential harm to the surrounding ecological environment, such as soil, surface water, and underground water, and ultimately directly endangering human life and health [3].

Environmental pollution problems, such as emissions of soot, sulfur dioxide, and arsenic hydride, have always existed in traditional zinc hydrometallurgy processes [4]. Overall, the Trail Zinc refinery (BC, Canada) has developed a hydrometallurgical direct leaching process to avoid air pollution problems caused by high-temperature roasting [5]. Yunnan Metallurgical Group Co., Ltd. (China) introduced the technology and conducted an oxygen-pressure acid leaching test on zinc sulfide concentrate at the end of the 20th century [6]. Since then, the production of the direct leaching process has expanded rapidly [7-9]. As a result, increasingly more direct leaching residue from zinc sulfide concentrates has been produced as metal production has increased. Direct leaching residue mainly contains elemental sulfur and other harmful metals, such as zinc and lead, which pose a great threat and challenge to the ecological environment [10]. The recovery of elemental sulfur from direct leaching residue is usually carried out by the hot filtration method, the flotation method, etc., while the recovery of other harmful metals is less understood [7, 10].

Zinc leaching residue usually involves the traditional pyrometallurgy method, which consumes a large amount of energy and produces a large amount of sulfur dioxide gas, posing a threat to the environment [11]. Since legislation of ion emissions has become increasingly strict, the recovery of direct leaching residue has become increasingly important with alternative hydrometallurgical technology [12,

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13]. Since sodium carbonate (Na_2CO_3) and sodium hydroxide (NaOH) can convert lead sulfate ($PbSO_4$) into easily treated lead carbonate ($PbCO_3$) and lead hydroxide ($Pb(OH)_2$), they are often used to leach waste residue containing lead sulfate [13-16]. However, the price of these leaching agents is relatively high, so low-cost sodium chloride (NaCl) is proposed and developed as a leaching agent. Lead-bearing waste residue has been leached using brine, and a satisfactory metal recovery rate was achieved [17, 18]. However, most studies have focused on lead paste or lead slag containing lead sulfate, while less research has occurred on sphalerite concentrate direct leaching residue.

This paper proposes a hydrometallurgy leaching technique suitable for sphalerite concentrate direct leaching residue. The samples were leached by hydrochloric acid and sodium chloride solution to obtain a lead-containing solution. After the impure ions were removed, the leachate reacted with the saturated sodium oxalate solution to obtain lead oxalate. Then, the lead oxalate was calcined at different temperatures to prepare lead oxide powders.

2 Experimental procedure

2.1. Sample preparation and characterization

In this paper, sphalerite concentrate direct leaching residue was the research object and provided by Kunming Metallurgical Research Institute, Yunnan Province, China. The samples were dried at 60 °C for 48 hours and then rod milled for 30 min before analysis and leaching. The chemical composition of the samples was determined by XRF and is shown in Table 1. The samples mainly consisted of plumbojarosite ($PbFe_6(SO_4)_4(OH)_{12}$) and silica (SiO_2), followed by iron pyrite (FeS_2) and calcium sulfate dihydrate ($CaSO_4{\bullet}2H_2O$) (Fig. 2(a)).

The XRD patterns of samples were recorded using X-ray diffractometer (Bruker D8 Discover) with CuKα radiation (40 kv, 40 mA). The particle size distributions of samples were researched with a laser particle size analyzer (Mastersizer 3000). SEM images of samples were completed using scanning electron microscope (SU8000). Thermal analysis was conducted on a simultaneous thermal analyzer (Netzsch STA 449F3). 31.007 mg of the sieved sample was heated in pure nitrogen under atmospheric pressure, with a heating rate of 5 °C/min from room temperature to 800 °C.

Table 1. *Chemical composition of sphalerite concentrate direct leaching residue by XRF*

Elements	S	Fe	Pb	SiO_2	Ca	Zn	Ba
Content (wt.%)	15	24	5.73	12.62	3.59	1.74	1

2.2. Experimental flow and leaching procedure

The flow chart of the entire experimental process is shown in Fig. 1. The sphalerite concentrate direct leaching residue was leached by chloride, and then, the pH value of the leachate obtained was adjusted to remove iron ions. The leachate was successively treated with sodium oxalate and ethyl alcohol for the preparation of lead oxalate. Then, the lead oxalate was calcined at different temperatures to prepare lead oxide powders.

In the chloride leaching experiments, the samples were leached in a thermostatic water bath. A certain concentration of hydrochloric acid and sodium chloride solution was added to the flask and then placed into a thermostatic water bath. The solution was heated to the set temperature, and the stirring rate was set at 500 rpm. When the temperature reached the set value, a certain amount of sample was quickly added into the flask. The leaching reaction began. After the leaching was completed, solid-liquid separation and sampling were performed for chemical analysis. The leaching efficiency (η) of zinc, iron, and lead was calculated by equation (1).

$$\eta = \frac{CV}{mx} \times 100\% \qquad (1)$$

where C is the concentration of the elements in the leachate, g/L; V is the volume of the leachate, L; m is the quantity of the samples used in each experiment, g; x is the content of the elements in the samples, %.

Since the pH value of the solution was 3.7, Fe(III)

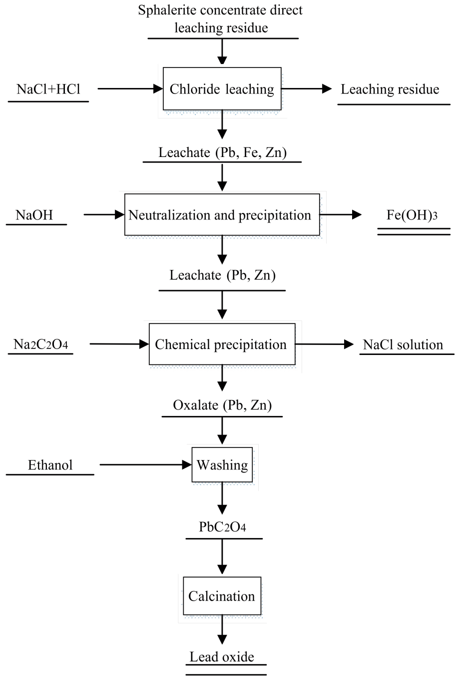

Figure 1. *The flow chart of the whole experiment*

was completely precipitated. Thus, the leachate was adjusted to a pH of 4 - 4.5 by a dropwise addition of sodium hydroxide solution (5 mol/L). After the precipitation was complete, Fe(III) was converted into iron hydroxide, and lead was still present in the form of $[PbCl_4]^{2-}$ in the solution. Then, lead oxalate was prepared by adding the saturated sodium oxalate solution due to its lower solubility product constant (4.8×10^{-10}). To remove other impurities, the lead oxalate was repeatedly washed with ethanol at 60 °C and then dried in a vacuum drying chamber. Finally, the lead oxalate was calcined in air for 2 hours at different temperatures and lead oxide powders were obtained.

3. Results and discussion

3.1 Chloride leaching

3.1.1 Characterization of direct leaching residue and chloride leaching residue

The XRD profiles of the direct leaching residue and chloride leaching residue samples are shown in Fig. 2. As shown in Fig. 2(a), the direct leaching residue sample was mainly composed of plumbojarosite ($PbFe_6(SO_4)_4(OH)_{12}$) and silica (SiO_2), followed by iron pyrite (FeS_2) and calcium sulfate dihydrate ($CaSO_4{\bullet}2H_2O$). As shown in Fig. 2(b), the XRD profile of the leaching residue after 60 min of chloride leaching at 85 °C was far different from that of the original direct leaching residue, which was mainly composed of silica (SiO_2), iron pyrite (FeS_2), and elemental sulfur (S). This result showed that the plumbojarosite was decomposed in the chloride leaching process. The size distribution curves of the direct leaching residue and chloride leaching residue samples are shown in Fig. 3. The peak for the leaching residue after 60 min of chloride leaching was obviously situated to the left of the peak for the direct leaching residue. This was precisely because the plumbojarosite decomposed in the chloride leaching process, leading to small leaching residue particles. This result is consistent with the XRD results, where the peak of plumbojarosite was not found after chloride leaching for 60 min (Fig. 2(a) and (b)).

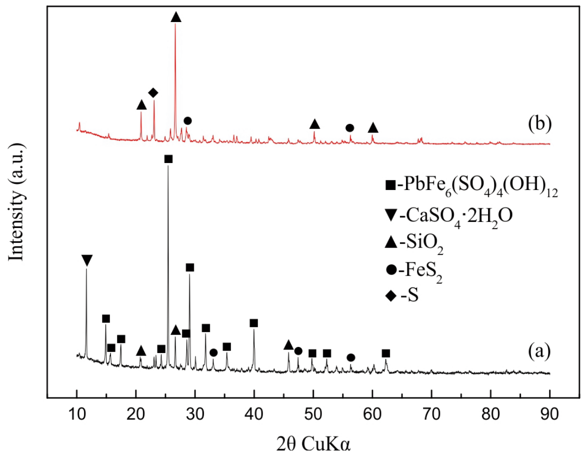

Figure 2. *XRD patterns of samples: (a: sphalerite concentrate direct leaching residue and b: leaching residue after 60 min chloride leaching at 85 °C)*

3.1.2 Effect of liquid-solid ratio

The effect of the liquid-solid ratio on the extractions of zinc, iron, and lead was studied with different liquid-solid ratios from 7.5-1 to 20-1 at a reaction temperature of 85 °C, an initial hydrochloric acid concentration of 1.2 mol/L, an initial sodium chloride concentration of 250 g/L and a reaction time of 60 min. As shown in Fig. 4, the leaching

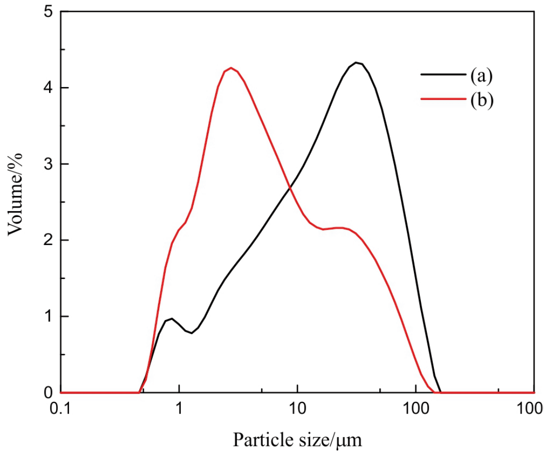

Figure 3. *Size distribution curves of the samples: (a: sphalerite concentrate direct leaching residue and b: leaching residue after 60 min chloride leaching at 85 °C)*

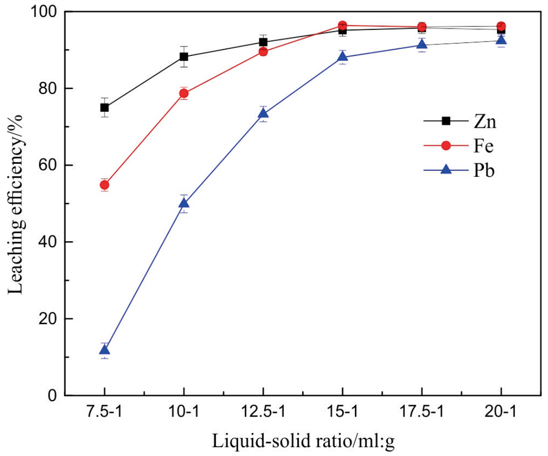

Figure 4. *Effect of liquid-solid ratio on the extractions of zinc, iron and lead from sphalerite concentrate direct leaching residue (temperature: 85 °C; initial hydrochloric acid concentration: 1.2 mol/L; initial sodium chloride concentration: 250 g/L; time: 60 min)*

efficiency of zinc, iron, and lead increased significantly with the increasing liquid-solid ratio. Only 75.0% of the zinc, 54.9% of the iron, and 11.7% of the lead were extracted at a liquid-solid ratio of 7.5-1, and the leaching efficiencies increased to 95.7%, 96.0%, and 91.3%, respectively, with the increase in the liquid-solid ratio to 17.5-1. In addition, the leaching efficiency of zinc, iron and lead slowly increase to a certain value when the liquid-solid ratio increased from 17.5-1 to 20-1. A suitable solid-liquid ratio of 17.5-1 for the extractions of zinc, iron, and lead was obtained for the experiments.

3.1.3 Effect of reaction temperature

The effect of the reaction temperature on the extractions of zinc, iron, and lead was studied at different reaction temperatures from 75 to 95 °C. During the leaching process, other leaching conditions remained constant, such as the liquid-solid ratio of 17.5-1, initial hydrochloric acid concentration of 1.2 mol/L, initial sodium chloride concentration of 250 g/L, and reaction time of 60 min. As Fig. 5 shows, increasing the reaction temperature enhanced the leaching efficiency of lead but not zinc and iron. The leaching efficiency of lead increased from 68.1% to 91.3% as the reaction temperature increased from 75 to 85 °C, while for zinc and iron, the leaching efficiency increased less than 4% under the same conditions. In addition, the further increase in the reaction temperature showed almost no significant increase in the leaching efficiencies of zinc, iron, and lead. Considering the relatively increased leaching efficiency of zinc, iron, and lead, a reaction temperature of 85 °C was suitable.

3.1.4 Effect of initial hydrochloric acid concentration

The experiments were carried out at the initial hydrochloric acid concentration range of 0.6 to 1.4 mol/L, maintaining liquid-solid ratio of 17.5-1, a reaction temperature of 85 °C, an initial sodium chloride concentration of 250 g/L, and a reaction time of 60 min. The effects of the initial hydrochloric acid concentration on the leaching efficiency of zinc, iron, and lead are presented in Fig. 6. The leaching efficiency of zinc, iron, and lead first increased and then slowed to a constant value. The leaching efficiency of zinc, iron and lead increased from 78.2%, 56.5%, and 22.6% to 95.3%, 96.2%, and 92.4%, respectively, when the initial hydrochloric acid concentration increased from 0.6 to 1.3 mol/L, respectively. In addition, the further increase in the initial hydrochloric acid concentration did not significantly promote leaching.

3.1.5 Effect of initial sodium chloride concentration

The effect of the initial sodium chloride concentration on the leaching of zinc, iron, and lead was investigated within the range of 150 to 300 g/L under constant conditions. The results are shown in Fig. 7. The variable concentration of initial sodium chloride had a significant influence on the leaching of zinc, iron, and lead. The leaching efficiencies of zinc, iron, and lead were 86.1%, 87.8%, and 68.4%, respectively, when the initial sodium chloride concentration was 150 g/L. However, 95.5% of the zinc, 96.0% of the iron, and 93.7% of the lead were extracted when initial sodium chloride concentration

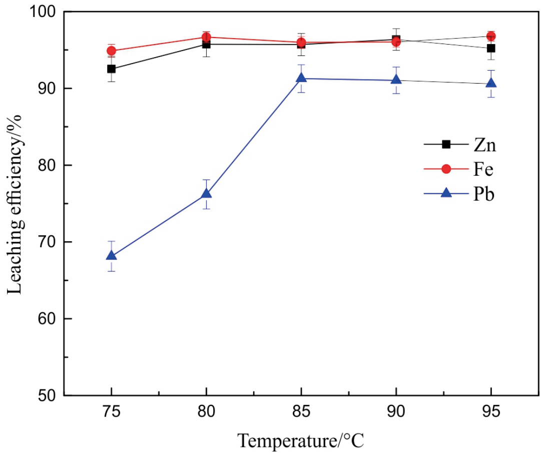

Figure 5. *Effect of the reaction temperature on the extractions of zinc, iron and lead from sphalerite concentrate direct leaching residue (liquid-solid ratio: 17.5-1; initial hydrochloric acid concentration: 1.2 mol/L; initial sodium chloride concentration: 250 g/L; time: 60 min)*

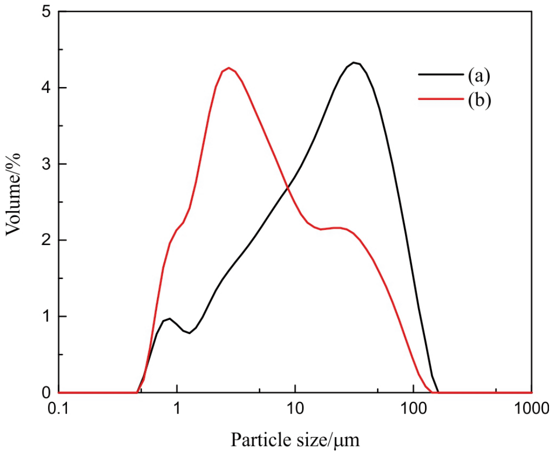

Figure 6. *Effect of the initial hydrochloric acid concentration on the extractions of zinc, iron and lead from sphalerite concentrate direct leaching residue (liquid-solid ratio: 17.5-1; temperature: 85 °C; initial sodium chloride concentration: 250 g/L; time: 60 min)*

was 300 g/L, which was obviously different from the leaching efficiency at lower concentrations of sodium chloride. Considering the relatively increased leaching efficiency of zinc, iron, and lead, an initial sodium chloride concentration of 300 g/L was suitable.

3.1.6 Effect of reaction time

To investigate the leaching efficiency of zinc, iron, and lead during different periods of the chloride leaching process, time-series experiments were implemented at liquid-solid ratio of 17.5-1, a reaction temperature of 85 °C, an initial hydrochloric acid concentration of 1.3 mol/L and an initial sodium chloride concentration of 300 g/L. The leaching efficiencies of zinc, iron, and lead as a function of time are displayed in Fig. 8. The leaching efficiency of zinc, iron, and lead sharply increased with increasing reaction time. The leaching efficiency of zinc, iron, and lead was 95.5%, 96.0%, and 93.7%, respectively, when the leaching time was 60 min. However, the further increase in reaction time to 90 min did not significantly impact the leaching of sphalerite concentrate direct leaching residue.

3.2 Preparation and calcination of lead oxalate

3.2.1 Characterization of lead oxalate

The XRD pattern of the lead oxalate samples obtained from the sphalerite concentrate direct leaching residue is shown in Fig. 9. As seen from the XRD pattern, all the peaks of lead oxalate obtained matched the standard XRD pattern of anorthic structure lead oxalate (PDF 00-014-0803). The SEM images of the lead oxalate samples are shown in Fig. 10. We can see that lead oxalate has a flaky shape with irregular or sharp-edged structure. In addition, its surface is extremely smooth, crystal clear, without a trace of impurities. The size distribution curve of the lead oxalate samples is shown in Fig. 11. The lead oxalate particles were small with a uniform size distribution, which was beneficial for the precursor to the preparation of lead oxide powders.

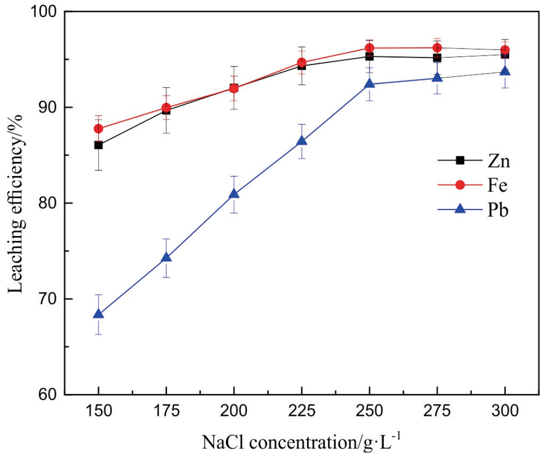

Figure 7. *Effect of the initial sodium chloride concentration on the extractions of zinc, iron and lead from sphalerite concentrate direct leaching residue (liquid-solid ratio: 17.5-1; temperature: 85 °C; initial hydrochloric acid concentration: 1.3 mol/L; time: 60 min)*

3.2.2. Calcination of lead oxalate and characterization of lead oxides

The TG and DSC curves are illustrated in Fig. 12. There was only one obvious stage of weight loss in the TG curve. As the lead oxalate decomposed, the weight decreased at temperatures ranging from 327 to 407 °C. The total weight loss of the samples was approximately 25.00%, which was close to the

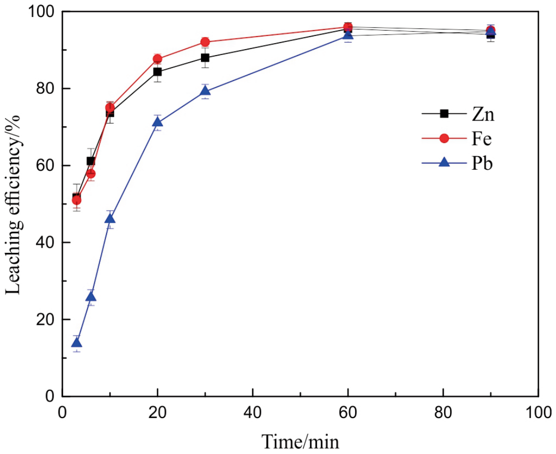

Figure 8. *Effect of the reaction time on the extractions of zinc, iron and lead from sphalerite concentrate direct leaching residue (liquid-solid ratio: 17.5-1; temperature: 85 °C; initial hydrochloric acid concentration: 1.3 mol/L; initial sodium chloride concentration: 300 g/L)*

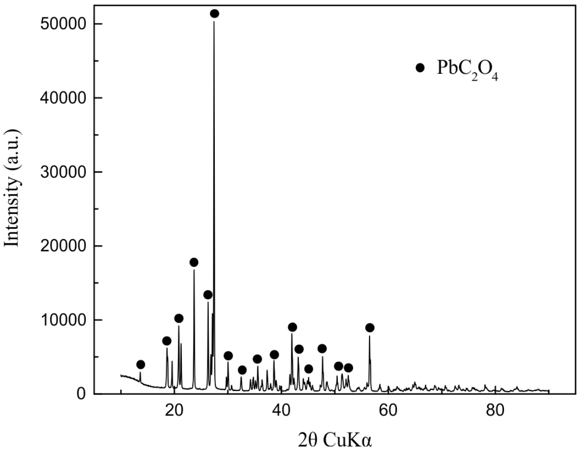

Figure 9. *XRD pattern of the lead oxalate samples*

theoretical value of 24.39 wt.% [11]. There was also one endothermic event resulting from the thermal effect of lead oxalate decomposition in the DSC curve. Consequently, the lead oxalate calcination decomposition equation was determined as follows:

$$PbC_2O_4 = PbO + CO\uparrow + CO_2\uparrow \qquad (2)$$

The XRD patterns of the lead oxides obtained by calcination at 300 °C, 350 °C, 400 °C, 450 °C, 500 °C, and 550 °C are shown in Fig. 13. As shown in Fig. 13(a), the main composition of the combustion product was lead oxalate at 300 °C, indicating that the lead oxalate did not decompose, which was consistent with the result of TG-DSC analysis. When the calcination temperature increased to 350 °C, the combustion product was mainly composed of α-PbO and a small amount of incomplete lead oxalate. When the calcination temperature continued to increase to 400 °C, the lead oxalate decomposed completely, and the combustion product was mainly composed of α-PbO. When the temperature was further increased to 450 °C, the decomposition product was mainly composed of α-PbO and β-PbO, which indicated that the crystallinity of the lead oxide began to change. When the calcination temperature finally reached 500 or 550 °C, β-PbO was obtained.

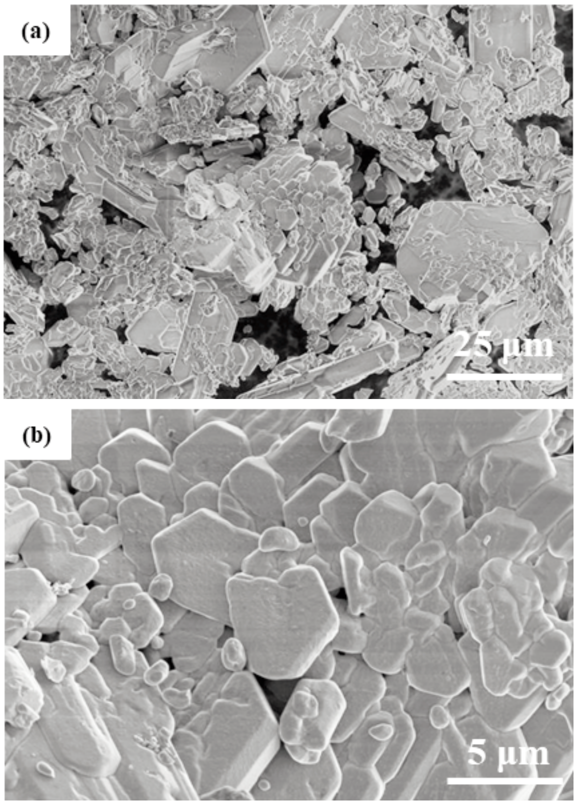

Figure 10. *SEM images of the lead oxalate samples (a: lead oxalate sample and b: local enlarged image of a)*

The SEM images of the lead oxides obtained by calcination at 350 °C, 400 °C, 450 °C, 500 °C, and 550 °C are shown in Fig. 14. When the calcination temperature exceeded the thermal decomposition

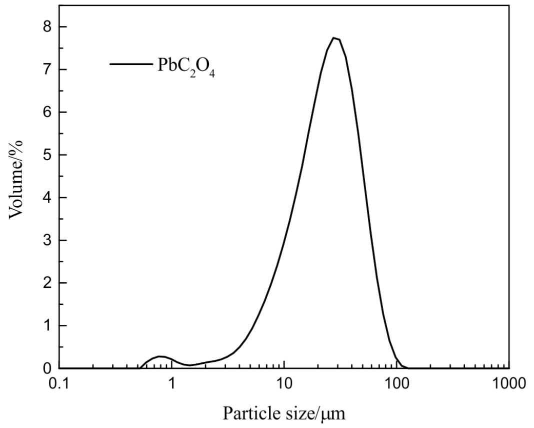

Figure 11. *Size distribution curve of the lead oxalate samples*

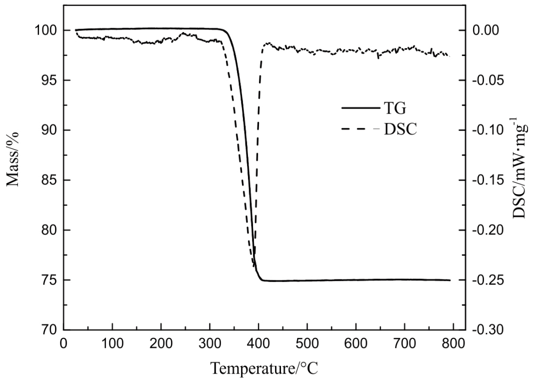

Figure 12. *Simultaneous TG-DSC curves of the lead oxalate samples in a nitrogen atmosphere*

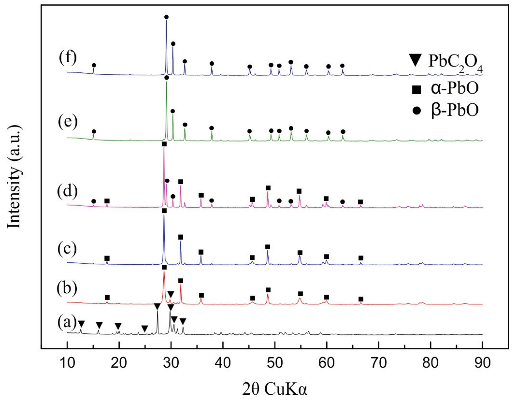

Figure 13. *XRD patterns of the lead oxides obtained by calcination at different temperatures: (a) 300 °C, (b) 350 °C, (c) 400 °C, (d) 450 °C, (e) 500 °C, and (f) 550 °C*

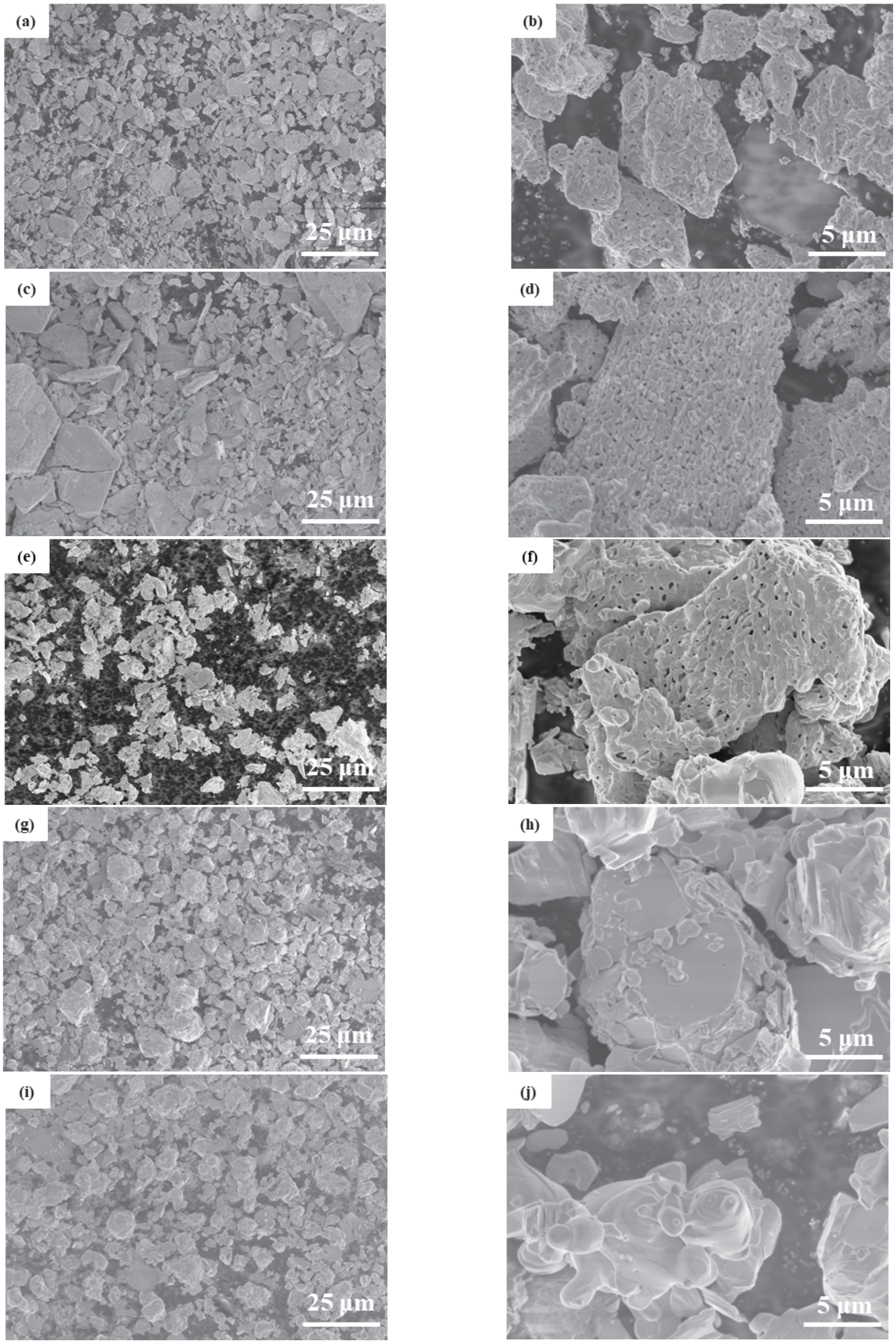

Figure 14. *SEM images of lead oxides obtained by calcination at different temperatures (a: 350 °C; b: local enlarged image a; c: 400 °C; d: local enlarged image c; e: 450 °C; f: local enlarged image e; g: 500 °C; h: local enlarged image g; i: 550 °C; j: local enlarged image i)*

temperature of lead oxalate, the emissions of gas began, causing the lead oxides to be broken into tiny pieces and porous structures. As the calcination temperature increased, the shape of the lead oxides changed significantly from a rod-like shape to a ball-like shape. This was consistent with the XRD results, in which α-PbO transferred towards β-PbO at the higher calcination temperature. The size distribution curves of the lead oxides obtained by calcination at 350 °C, 400 °C, 450 °C, 500 °C, and 550 °C are shown in Fig. 15. The lead oxide particles obtained were smaller at less 100 μm with a relatively narrower size distribution.

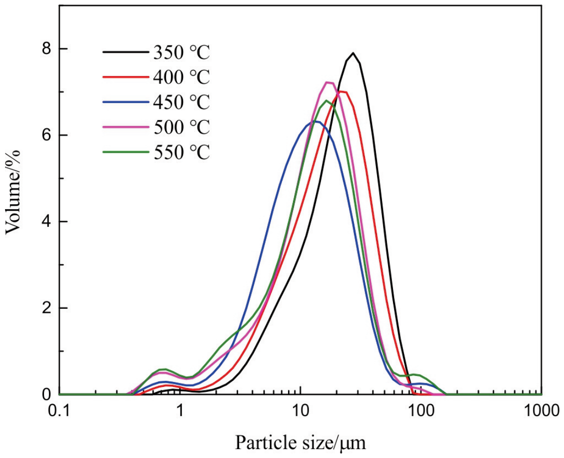

Figure 15. *Size distribution curves of the lead oxides obtained by calcination at different temperatures*

4. Conclusion

A novel hydrometallurgy recovery method combining chloride leaching, oxalate precipitation, and calcination was developed for leaching lead from sphalerite concentrate direct leaching residue to obtain lead oxide powders. Hydrochloric acid and a sodium chloride solution were used as leachates to leach lead from sphalerite concentrate direct leaching residue. The main optimum parameters were determined, such as a liquid-solid ratio of 17.5-1, a reaction temperature of 85 °C, an initial hydrochloric acid concentration of 1.3 mol/L, an initial sodium chloride concentration of 300 g/L, and a reaction time of 60 min. Ninety-five percent of the zinc, 96.0% of the iron, and 93.7% of the lead were extracted into leachate under the optimum conditions. Then, sodium hydroxide was employed as a neutralizer, neutralizing and precipitating to obtain iron hydroxide, which achieved the purpose of removing iron. Finally, sodium oxalate was used as a precursor for the synthesis of the lead oxide powders by a high-temperature calcination process.

Acknowledgements

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LUŽENJE OLOVA IZ TALOGA DOBIJENOG DIREKTNIM LUŽENJEM KONCENTRATA SFALERITA U PRISUSTVU HLOROVODONIČNE KISELINE I NATRIJUM HLORIDA ZA PRIPREMU OKSIDA OLOVA

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Apstarkt

Cilj ovog rada je bio odabir i predlog primenjive metode za ekstrakciju olova iz taloga dobijenog direktnim luženjem koncentrata sfalerita. Izvršen je veliki broj eksperimenata za ekstrakciju olova iz taloga dobijenog direktnim luženjem koncentrata sfalerita u prisustvu hlorovodonične kiseline i natrijum hlorida kao lužitelja. Određeni su optimalni glavni parametri, kao što su odnos tečno-čvrsto od 17,5-1, temperatura reakcije 85 °C, početna koncentracija hlorovodonične kiseline 1,3 mol/L , početna koncentarcija natrijum hlorida 300 g/L i vreme reakcije 60 minuta. Pod optimalnim uslovima je ekstrahovano 95% cinka, 96,0% gvožđa i 93,7% olova u lužni rastvor. Olovo u lužnom rastvoru je dobijeno u obliku [PbCl4]2-. Nakon što je lužni rastvor prečišćen da bi se uklonile nečistoće, isti je pretvoren u olovo oksalat u prisustvu natrijum oksalata kao supstance koja uzrokovala taloženje. Poslednji korak je predstavljao razlaganje olovo oksalata na olovo oksid u prahu putem postupka kalcinacije na visokoj temperaturi.

***Ključne reči**: Talog dobijen direktnim luženjem koncentrata sfalerita; Hlorovodonična kiselina i natrijum hlorid; Olovo oksalat; Olovo oksid; Luženje*