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ACIDIC LEACHING OF CADMIUM FROM ZINC PLANT RESIDUE

Mahdi GHARABAGHI, Mehdi IRANNAJAD, Amir Reza AZADMEHR

Department of Mining and Metallurgical Eng., Amirkabir University of Technology, Hafez Ave., Tehran, Iran, email: iranajad@aut.ac.ir, Tel:+98-21-64542900

Abstract. In the present paper cadmium leaching from zinc plant residue using sulphuric acid was examined. The zinc plant residue is a hazardous waste which is produced in the hydrometallurgical zinc plant and it contains considerable amounts of metals such as zinc, cadmium and nickel. The effects of sulphuric acid and other important factors such as reaction time, solid-to-liquid ratio, particle size, stirring speed and temperature on cadmium recovery were investigated. The concentration of cadmium in solution was observed to increase with the increase of the reaction time, acid concentration, stirring speed and temperature. Decreasing of solid/liquid ratio and particle size were also beneficial for cadmium recovery. The largest cadmium leaching recovery (97%) was obtained after 30 minutes of treatment at 25°C using H₂SO₄ (8% (v/v)) at solid-to-liquid ratio of 0.10. XRD and SEM analyses of the residues obtained after leaching showed that the cadmium containing phase had been decomposed in the leaching residues. The results indicated that it is possible to use this waste as a secondary resource for cadmium recovery.

keywords: hazardous waste, leaching, cadmium extraction, zinc plant residue

1. Introduction

Cadmium is an important metal, which is extensively used in solar batteries, electronics, electroplating, metallurgical industries, synthetic chemicals, ceramics, etc. (Arntz et al., 1999; Cheremisinoff, 1995; Kumar, et al., 2009). Cadmium is relatively rare metal in the earth's crust and occurs naturally as a minor constituent of base metal ores (Habashi, 1997). Cadmium is obtained as a by-product from base metals production or recovered from other secondary resources such as Ni-Cd batteries, cadmium-tellurium solar cells, zinc sinter plant fume, cadmium-containing alloys etc. (Butterman, 2002; Freitas and Rosalém, 2005; Kumbasar, 2009; Nogueira and Margarido, 2004; Reddy and Priya, 2006). Hydro and pyrometallurgical processes have employed for the recovery of metals from these resources.

In the hydrometallurgical zinc extraction, waste material containing cadmium is generated in the form of solid residue. This waste contains considerable amounts of metals values such as zinc, cadmium and nickel. The disposal of the zinc plant residue

causes not only environmental pollution (cadmium and its compounds are highly toxic and carcinogenic in nature) but a loss of resources (Agrawal and Sahu, 2006; Altundoğan, 1998; Elyahyaoui and Bouhlassa, 2001; Kumar et al., 2009; Turan, 2004; Safarzadeh, 2008). Contributing to metal losses and potential environmental pollution, it is possible to manage zinc plant residue by using this waste as cadmium secondary resource. Recycling of metals will conserve the natural resources to meet the future demand of materials and also reduce the environmental pollution. The extraction of metal from hazardous wastes has coincidence with the legislations assigned to protect the environment in the world and can mitigate unfavourable environmental impacts (Chen et al., 2011).

In the recent years, a lot of research has been performed for recovery of cadmium and nickel from some waste as secondary resources (Bernardes, 2004; Cortina, 1996; Freitas and Rosalém, 2005; Huang, 2007; Kumbasar, 2009; Nogueira and Margarido, 2004; Reddy et al., 2005; Safarzadeh, 2007, 2009). Zinc plant residue contains high concentration of cadmium, and it can be used for cadmium extraction. In addition, this waste contains a desirable amount of nickel and other valuable metals, and it can be considered as a secondary resource for these metals. This waste composition is varied due to the origin of zinc concentrate and process conditions which have been used for zinc production. The average cadmium content in these wastes, which are produced in different zinc plants in the world, are shown in Table 1.

Table 1. Cadmium content in zinc plant wastes

Country	Cadmium concentration (%)	Reference
Turkey	3.0	(Kumbasar, 2009)
Iran	13-16	(Safarzadeh, 2009)
Turkey	1.33	(Kul, 2008)
Yugoslavia	2-6	(Stanojevic et al., 2000)
India	6-7	(Singh, 1996)
Brazil	26	(Gouvea and Morais, 2007)

As can be seen in the Table 1, there are considerable cadmium concentrations in these wastes, and they can be considered as cadmium secondary resources. Recycling of these wastes for other industries is a good approach for minimizing environmental problems. Recycling of hazardous waste becomes more important in recent years due to the shortage of high quality ore reserve.

In this study, the leaching of cadmium in aqueous sulphuric acid solution was investigated. The effects of the acid concentration, reaction time, particle size, solid-to-liquid ratio, reaction temperature and stirrer speed on the dissolution rate have been

evaluated. In addition, optimum conditions which provided maximum cadmium extraction using a hydrometallurgical process were determined. The results of this study are important from the environmental point of view in the treatment and recovery of cadmium from wastes.

2. Experimental

The zinc plant residue which was used in the study come the Calcimin zinc plant in Zanjan, Iran. The sample was ground and sieved by ASTM standard sieves to obtain the nominal particle size fractions of -850+425, -425+250, -250+75, -250, -75 μm in diameter. The chemical composition of the sample is given in Table 2. As shown in Table 2, the sample contained high amounts of cadmium and zinc. The XRD results indicated that metals were in the oxide form.

In the leaching experiments, sulphuric acid (Merck, Germany) and distilled water were used. Leaching experiments were carried out in a 500 ml Pyrex reactor equipped with a stirrer motor for mixing and reflux condenser to prevent losses by evaporation. The reactor was inserted into a temperature controlled water bath for controlling the reaction temperature. 100 cm^3 of sulphuric acid at pre-determined concentrations (v/v) was transferred into the reactor, brought to the desired temperature and then 10 g of dried sample was added onto the acid solution. The leaching was continued for the pre-determined periods and the content of reactor was filtered as soon as the process finished. The cadmium concentration in solution was analyzed using Atomic Absorption Spectrophotometer (AAS) of Perkin-Elmer.

Table 2. The chemical analysis of the sample

Component	ZnO	CdO	NiO	CuO	PbO	SO ₃	CaO	Fe ₂ O ₃	MgO	Al ₂ O ₃	LOI*
Amount, %	38.92	16.56	4.21	1.99	1.38	12.10	2.61	0.44	0.20	0.34	20.54

*Loss on ignition (LOI) is the sample weight reduction after being ignited

3. Results and discussion

The solubilisation of metals can be predicted from Eh-pH (or Pourbaix) diagrams and also the Cd, Zn, Ni and Cu sulphates solubility products can be obtained from thermodynamic data (Nogueira and Margarido, 2004; Pourbaix, 1978). In the leaching system, the concentration of metals depend on the equilibrium pH of the solution and equilibrium solubility constant of the metals sulphate.

The Eh-pH diagram, drawn using the Medusa software for the Cd-S-H₂O system, is shown in Fig. 1. From Fig. 1 it is obvious that SO₄⁻² ion increases the solubility of Cd ion in the leaching system and the following complexation reaction occurs through sulphuric acid leaching:



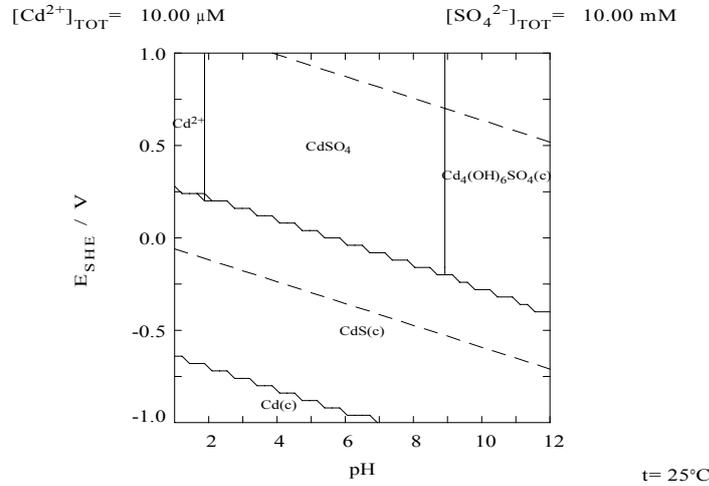


Fig. 1. Eh-pH diagrams of the Cd-S-H₂O system, Medusa software (Royal Institute of Technology, Sweden, <http://www.kemi.kth.se/medusa>)

3.1. Effects of leaching time

Using a constant sulphuric acid concentration (8% (v/v)), the effect of leaching time on the cadmium extraction was investigated at a temperature of 25°C, stirring speed of 500 rpm and solid-to-liquid ratio of 0.10. The percentage extraction of cadmium vs. time was plotted and presented in Fig. 2. The results clearly demonstrate that cadmium extraction was fast and reached equilibrium within 30 minutes. Within the first 10 minute, 78% cadmium extraction was achieved. This increased to 93% after 20 minutes dissolution. After an initial fast reaction rate, cadmium concentration in leach solution increased slowly with time. For example, after an initial increase of cadmium extraction up to 93% within 20 minutes, cadmium concentration slowly increased and reached 97% after 30 minutes. This can be attributed to the increase in solution pH with time because of the consumption of acid as the leaching proceeds.

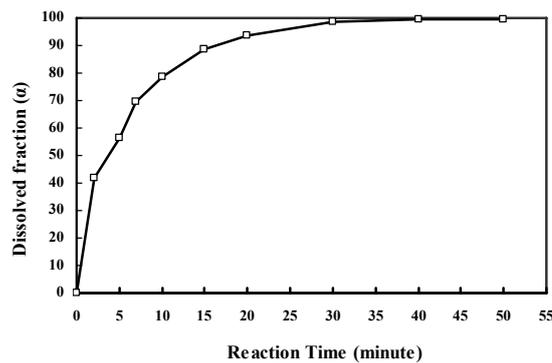


Fig. 2. The effects of reaction time on cadmium extraction (8% acid concentration (v/v), S/L=0.10, T=25°C, 500 rpm, -250 μm)

The results clearly demonstrate the effects of leaching time on the cadmium extraction. With the leaching time of 30 minutes, the leaching efficiency reached about 97% at 25 °C using the solid-to-liquid ratio of 10%. When the leaching time was above 30 minutes, there was not any significant difference on leaching efficiencies. Therefore, 30 minutes was the optimum leaching time and extended leaching times had no significant effects on cadmium extraction.

3.2. Effects of acid concentration

The effects of sulphuric acid concentration on the dissolution of cadmium for 40 minutes of leaching time are given in Fig. 3. Experiments were performed with five different acid concentrations (v/v) at a reaction temperature of 25 °C, a stirring speed of 500 rpm, a particle size of -250 µm and solid-to-liquid of 10%. There was low cadmium extraction efficiency for low acid concentration, namely 2 and 5% (v/v). By increasing the acid concentration, the extraction values were greatly increased and reached 99.0% at 15% (v/v) sulphuric acid concentration. At the 2 and 5% (v/v) acid concentration, the amount of acid was not adequate to extract all of leachable cadmium. As observed from the experimental results given in Fig. 3, using 8% (v/v) acid concentration, the cadmium contents increased and reached its maximum by increasing the leaching time up to 30 minutes, and then remained almost constant.

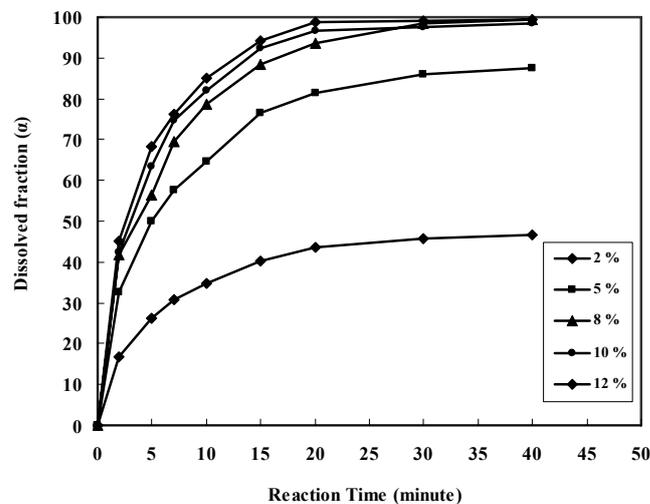


Fig. 3. Effects of acid concentration on cadmium extraction from waste (25°C; 500 rpm, S/L=0.10, particle size -250 µm)

Increasing cadmium extraction, as acid concentration increases, is due to acid concentration effect on increasing the H^+ activity that result in further dissolution of cadmium containing material. Similarly (Babu et al., 2002), also confirmed that the zinc extraction from zinc sulphide concentrate is a function of sulphuric acid

concentration. Increasing metal extractions by increasing leaching agent concentration were also observed by Bodas (1996) and Souza et al. (2007).

After 30 minutes the reaction reached equilibrium and the formation of solid products at the system prevented further leaching of cadmium. In addition, the precipitation of gypsum or other insoluble minerals, which were present in the leaching system, prevented further dissolution. However, there were no beneficial effects upon increasing acid concentration to more than 8% (v/v) for 30 minutes of leaching.

3.3. Effects of solid-to-liquid ratio

In order to evaluate the influence of solid-to-liquid ratio on the cadmium leaching recovery, experiments were carried out for 40 minutes at 25°C and acid concentration of 8% (v/v). The results, presented in Table 3, indicate that the cadmium concentration in the solution increased with a decrease in the solid-to-liquid ratio. Cadmium extraction rises from 71.2% at S/L 0.30 to 98.65% at S/L 0.05. However, the cadmium recovery was 97.5% at S/L 0.05. When S/L was lower than 0.10, cadmium leaching recovery increased slowly. The improvement of leaching results for low solid content can be attributed to the fact of the amount of reagent to each particle increases with decreasing solid amounts in the leaching process. Therefore, in the diluted pulp it was expected to achieve an increasing dissolution rate. Similar results for dissolution were found in other studies (Zhang and Nicol, 2010).

Lower S/L leads to dilute solutions which are unfriendly to filtration. Thus, solid to liquid 0.10 is considered the optimal.

Table 3. Effect of solid-to-liquid ratio on cadmium extraction (25 °C; 500 min⁻¹, 30 minutes, acid concentration 8% (v/v))

Test No.	Solid-to-liquid ratio	Cadmium extraction (%)
1	0.05	98.65
2	0.10	97.50
3	0.15	86.15
4	0.20	79.10
5	0.30	71.20

3.4. Effects of particle size

To investigate the effect of the particle size on the cadmium dissolution rate, experiments were carried out using five different particle sizes of the sample (-850+425, -425+250, -250+75, -250, -75 μm) at a reaction temperature of 25°C, stirring speed of 500 rpm and acid concentration of 8% (v/v). As can be seen in Table 4, as the particle size decreases, the cadmium dissolution rate increases. After 5 minutes of leaching time, 48.2 % cadmium was extracted using sample size -850+425

micron, and it was improved to 63.5% using sample size finer than 75 micrometers. The cadmium leaching recovery is kept at the level between 97% and 99% when the samples size was lower than 250 μm . This situation can be attributed to the increasing contact surface of the samples as the particle size decreases. Basing on the obtained results, we have considered the -250 μm particle size as the optimum.

Table 4. Variation of cadmium extraction with particle size

Fraction	Particle size (μm)	Cadmium extraction (%)
1	-850+425	89.15
2	-425+250	92.12
3	-250+75	96.52
4	-250	97.61
5	-75	99.19

3.5. Effects of stirring speed

To observe the effect of stirring speed on the dissolution rate, experiments were carried out at particle size of -250 μm , reaction temperature of 25 $^{\circ}\text{C}$ and acid concentration 8% (v/v). As shown in Table 5, the stirring speed had a significant influence on the cadmium dissolution rate and higher extraction values were achieved at higher stirring speed. The cadmium extraction increased more than 25% when the stirring speed increased from 100 to 400 rpm. A further increase of the stirring speed had a little effect on the cadmium extraction. The maximum extraction values reached under such condition was more than 97%. The experimental results showed that the dissolution rate is practically independent of the stirring speed after 500 rpm. So the stirring speed of 500 rpm was considered as the optimum agitating rate.

Table 5. Effect of stirring speed on cadmium leaching (8 % (v/v) acid, 25 $^{\circ}\text{C}$, 500 min^{-1} , 30 min, S/L=0.10)

Test No.	Stirring speed (rpm)	Cadmium extraction (%)
1	0	32.11
2	100	74.94
3	200	85.64
4	300	93.31
5	400	96.57
6	500	97.21
7	600	98.83

3.6. Effects of temperature on the leaching

The effect of the temperature on the dissolution rate was studied using four different reaction temperatures from 25 to 75 $^{\circ}\text{C}$ for particles size of -250 μm , stirring speed of 500 rpm and acid concentration of 8% (v/v). Figure 4 shows that increasing

reaction temperature increases the dissolution rate, as expected from the exponential dependence of the rate constant in the Arrhenius equation. More than 88% of the cadmium present in the sample was extracted after 15 minutes at the reaction temperature of 25 °C and it improved to 97% at 75°C. However, the obtained results show that by increasing the leaching time, the effects of temperature decreases. This behaviour was observed in previous works carried out by Abdel-Aal and Rashad (2004), Mulak et al. (2005) as well as Nogueira and Margarido (2004).

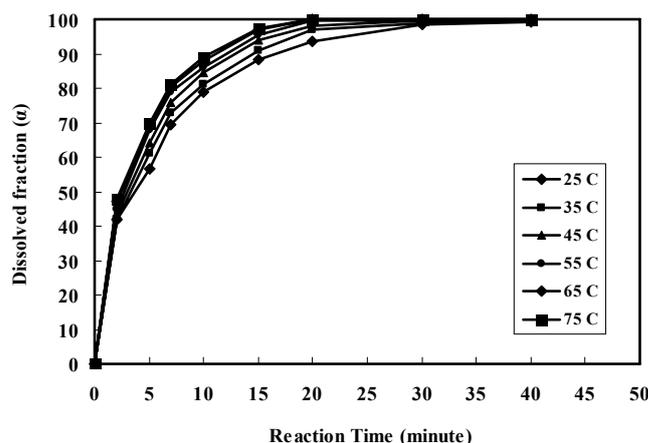


Fig. 4. Effect of reaction temperature on the cadmium extraction (8% (v/v) acid, 25°C, 500 rpm, S/L=0.10, particle size: -250 μm , 500 min^{-1})

4. Characterization of leaching residue

A series of analyses were performed to measure the chemical composition and to determine the mineralogy, morphology and particle size of leaching residues obtained under different leaching time. The chemical composition of the three residues obtained at an acid addition of 8% (v/v) stirring speed 500 rpm, solid to liquid ratio of 0.10, 25 °C after 5, 15 and 30 minutes leaching is shown Table 6.

It can be seen that only about 0.01–0.02% of cadmium remains in the residue. As can be observed in Table 6, by increasing the leaching time, the Cu, Ca and Pb amounts increased markedly while the cadmium content decreased correspondingly. In fact, the amounts of Ca and Pb remained almost the same and increasing of Cu, Ca and Pb amounts in the leach residues were due to a decrease in the Ni, Cd and Zn content.

A XRD analysis of the same residues in Table 6 was also performed. Gypsum, $\text{ZnSO}_3 \cdot 2\text{H}_2\text{O}$, and PbO were identified by XRD and cadmium compounds amounts decreased in residues with increasing leaching time. After 15 minutes of leaching, gypsum, copper oxide and different lead compounds were formed. There was a little amount of cadmium in the leaching residue yet. After 30 minutes of leaching, only copper, lead, and calcite compounds were identified and gypsum was the main

constituent. No cadmium containing materials were detected in the residue. This gives rise to the assumption that all the cadmium had been leached by sulphuric acid.

Table 6. Cadmium amounts in residue after different leaching time

Leaching Time (min)	5	15	30
Nickel (%)	3.01	1.24	0.17
Cd (%)	9.31	3.12	1.13
Zn (%)	21.36	8.73	2.11
Cu (%)	4.12	9.14	12.61
Pb (%)	7.32	15.41	18.46
Ca (%)	12.35	27.72	37.62

The gypsum and lead oxide intensity in the sample increased markedly after leaching, and the nickel and cadmium intensity decreased extremely. It could be expected that nickel and cadmium compound dissolved in the system. Furthermore, the Fe_2O_3 intensity also increased slightly.

Figures 5 to 8 show SEM images the residues obtained under leaching condition. The particle size distributions for these residues are shown in Fig. 5. From Fig. 5 it can be seen that the particles vary in size and they show a wide particle size distribution (Fig. 5a). The sample particles have irregular shapes (Fig. 5b). The micrographs of the leaching residues show a progressive reduction in particle diameter (Fig. 6) and increase in roughness and porosity of the solids as shown in Fig. 7.

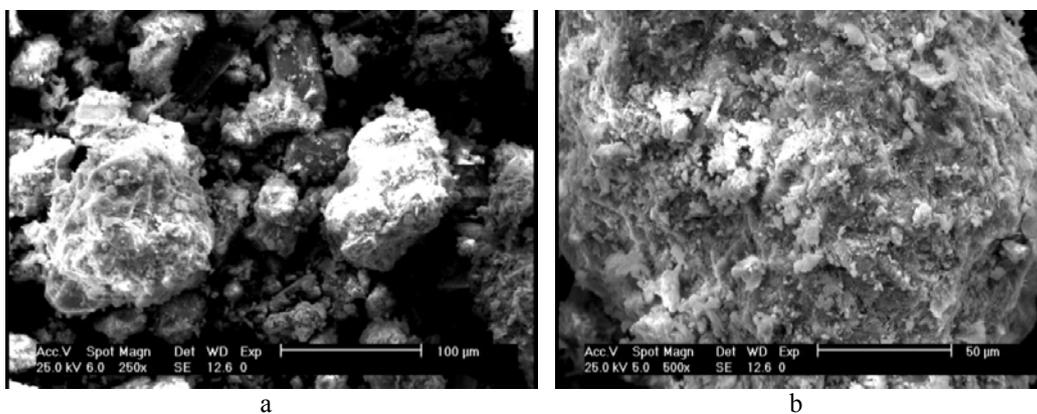


Fig. 5. (a, b). Particles of the sample before leaching

There was a large area of erosion at the sample surface (Fig. 7) and the surface was very rough and loose, approaching a porous structure. The EDAX analysis showed the amount of cadmium in the leaching residue decreased and amount of calcite and gypsum increased by increasing leaching time. This situation may be because the cadmium compounds were dissolved first. After 30 minutes leaching, other compounds such as calcite and gypsum were remained in the residue (Fig. 8).

The characterization study showed that the cadmium containing phases progressively disappeared as indicated by the XRF results. Fewer soluble calcite and lead containing phases predominated at later stages and their grains are also observed in the leaching residue (Fig. 8).

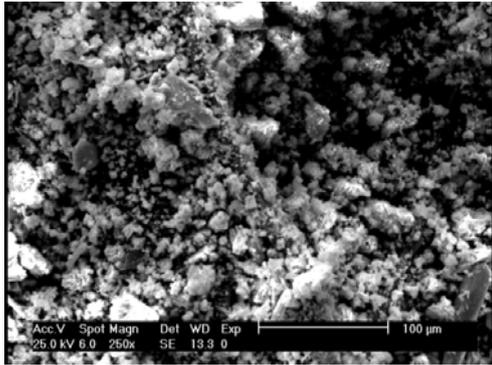


Fig. 6. Particles of the sample after 15 minutes of leaching. Leaching conditions: 8% acid concentration (v/v), 25°C, 500 min⁻¹, 100 g/dm³ solids

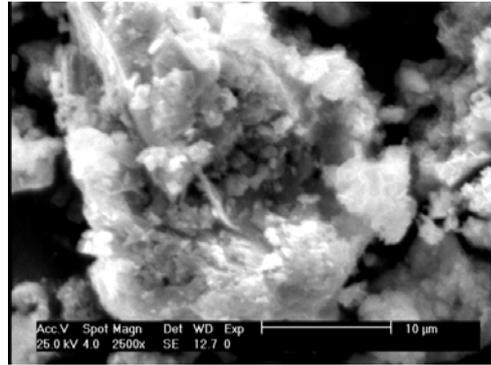


Fig. 7. Surface of particle after 20 minutes of leaching. Leaching conditions: 8% acid concentration (v/v), 25°C, 500 min⁻¹, 100 g/dm³ solids

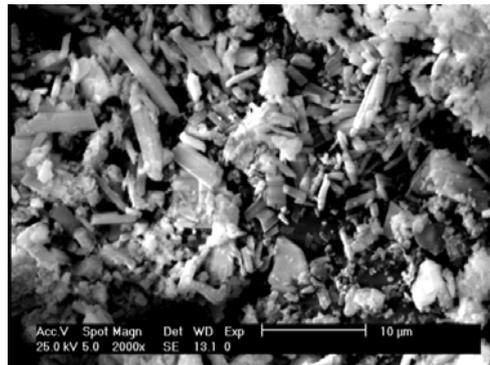


Fig. 8. Particles of the samples after 30 minutes of leaching. Leaching conditions: 8% acid concentration (v/v), 25°C, 500 min⁻¹, 100 g/dm³ solids

5. Final optimum experiment and process flowsheet

By optimization of the leaching variables summarised in Table 7, the final test was performed. The results showed with using these optimum parameters, it was possible to extract more than 97% of cadmium in the sample. The process flowsheet for cadmium and other metals recovery from this waste is presented in Fig. 9.

Table 7. The results of leaching under optimum process conditions

Time (minute)	Acid Consumption (v/v) %	Solid-to-Liquid ratio (%)	Temperature (°C)	Stirring Speed (min ⁻¹)	Ni Extraction (%)
30	8	10	25	500	97

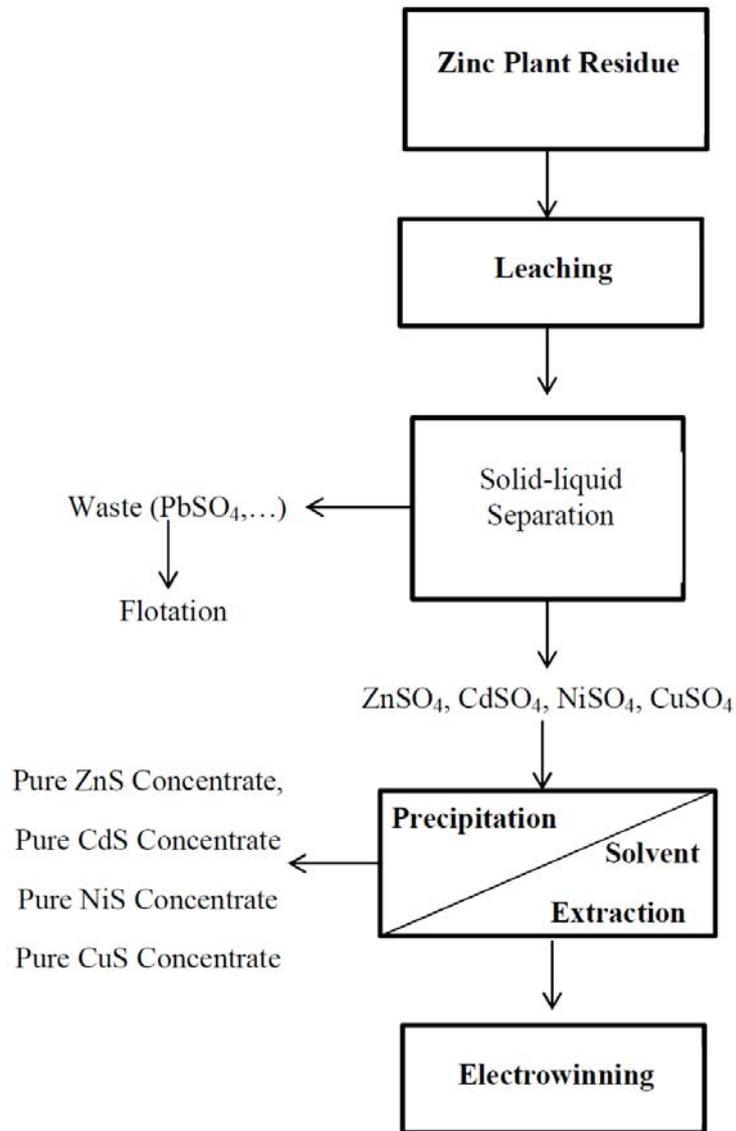


Fig. 9. Flowsheet for the metals recovery from zinc plant residues

6. Conclusions

The results of this study showed that it is possible to extract cadmium content from zinc plant residue and confirmed the previous study (Safarzadeh, 2009). Sulphuric acid leaching tests performed on Iranian zinc plant residue showed that 97% of cadmium can be leached in 30 minutes at 25°C using 8% sulphuric acid solutions. Evaluation of the experimental data showed that the cadmium extraction rate increased with increasing leaching time, acid concentration, stirring speed and decreasing solid-to-liquid ratio. Decreasing particle size was beneficial for leaching performance. SEM and XRD results showed that by increasing leaching time, the cadmium compounds have leached faster than other minerals in the leaching system.

The results indicated that it is possible to extract more than 97% cadmium content using these optimum parameters.

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