PRODUCTION OF ZINC POWDER FROM Co-Zn PLANT RESIDUE USING SELECTIVE ALKALINE LEACHING FOLLOWED BY ELECTROWINNING

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Abstract: Annually, gigagrams of cobalt residues, called hot filter cake (HFC), are produced from the Co neutralization step at Iranian zinc plants. With respect to the composition of HFC (i.e., 15-25% Zn, 0.5–1.5% Co, 3–8% Mn), it can be used as a secondary source of zinc, cobalt and manganese. In the present study, for the first time, treatment of HFC for separation and recovery of zinc has been studied. The residue was treated by employing selective alkaline leaching, in order to recover the maximum amount of zinc, followed by zinc electrowinning process. As a results, a solution was obtained from alkaline leaching under the optimum condition of 75 °C, sodium hydroxide of 8 M, solid-to-liquid ratio dm^3 of 1:10, and stirring speed of 600 rpm, having zinc recovery of 88.5 %. In the following step, the electrowinning process, under the optimum working conditions being current density 350 A/m^2 and time 10 hours, was carried out to produce a zinc powder with high purity of 99 percent. Finally, a simple and effective conceptual flow diagram was proposed for the process.

Keywords: zinc powder, HFC, alkaline leaching, sodium hydroxide, alkaline electrowinning

Introduction

A part of zinc is recovered from different secondary resources, namely zinc ash, zinc dross, flue dusts of electric arc furnace and brass smelting, automobile shredder scrap, rayon industry sludge etc., which contain different levels of impurities depending on their sources. These secondary metal resources could be used to extract metallic values or it may be disposed of. However, the disposal of such materials is now becoming expensive because of increasingly stringent environmental protection regulations.

Furthermore, the chemical nature of these dust particles is such that they are classified as hazardous wastes (Safarzadeh et al., 2009). Therefore, the extraction of metallic values from these resources instead of their disposal seems to be economical.

In the zinc production plant Zanjan in Iran, because of the chemical composition of the ores, removal of some impurity such as cobalt is necessary to recover zinc product (Safarzadeh et al., 2009). The solid residue of the cobalt precipitation step, known as hot filtercake (HFC), is obtained after filtration of pulp. This residue, with the composition of 15-25% Zn, 0.5-1.5% Co and 3–8% Mn, can be considered as a secondary resource of zinc in Iranian zinc plants.

This paper is the first work in which the HFC waste containing Co and Zn and the other heavy metals was treated to recovery and produce metals. Therefore, the emphasis of this paper is on treating the Co-waste produced in zinc plants. The hot filter cake (Co-waste), that was a waste is now is a potential and considerable source of zinc, cobalt etc. The hazardous high-cobalt composition of the filter cake, poses unavoidable risk to human health and environment. Metal content extraction from the HFC and controlling the hazard of it, is a great concern among Iranian researchers not only for environmental reasons but also for economic aspects (Safarzadeh et al., 2008, 2009). Therefore, a cost-effective process is necessary to extract the metals from the waste sources such as HFC by hydrometallurgical treatment.

Some studies have been carried out in the world to recover valuable metals from zinc plant residues (ZPR). The extraction of zinc, cobalt and manganese from the Iranian HFC of National Iranian Lead and Zinc Company (NILZ) by solvent extraction has been reported. In such solvent extraction routs, the separation of zinc, manganese and cobalt was accomplished using 20% D2EHPA+5% TBP in kerosene, 298 K and at O:A ratio of 1:1and pH of 1.5-2 (Eivazi et al., 2008ab; Jha et al., 2001). Despite the recovery of zinc from acidic media, zinc leaching from ZPR in alkaline medium has been developed. Alkaline agents such as sodium hydroxide, ammonium hydroxide, and sodium carbonate can selectively bring the desired metal into solution, leaving Fe and other metals such as Ni and Mn. Alkaline leaching of zinc-carbon batteries has been reported in literature (Kim et al., 2008; Shin, 2008). Kim et al. reported the alkaline leaching of zinc-carbon batteries in 4 M NaOH and obtained a solution with recovery of 64% Zn and 0.1% Mn (Kim et al., 2008). This solution can be followed by electrowinning (EW) of alkaline leaching solution to produce zinc powder with 99.9% purity (Gurmen and Emre, 2003). Zhao and Stanforth (2000) also investigated alkaline treatment of smithsonite ores followed by zinc powder production. In some investigations, different alkaline leaching techniques were tested in order to dissolve zinc present in an electric arc furnace (EAF) steel dust (Dutra et al., 2006). Furthermore, alkaline leaching of sphalerite by feeding lead carbonate, illustrates leaching zinc out as $Na_2Zn(OH)_4$ (Chenglong and Youcal, 2009). The alkaline leaching of metals from various resources in different investigations has been summarized in Table 2. Among the reports summarized in Table 2, no research has been done on selective extraction of zinc from cobalt waste followed by EW.

Furthermore, as seen in this table, the novelty is the usage of a specific material (EAF dust, ores etc.) that leaching has been carried out on it. For example, in one of the latest investigations carried out by Mohapatra et al. (2014), a flowsheet has been developed for extraction of zinc from spent catalyst by alkaline leaching as the flowsheet of this study developed for zinc extraction from HFC. This research shows that developing a process for metal recovery from various resources is currently attractive. Therefore, one of the differences of this paper from the others is usage of HFC in an advanced process in which some products were obtained. Using this advanced process, a main product (zinc powder) and a by-product (Co-Mn concentrate) have been produced.

In our previous research, selective zinc alkaline leaching optimization and cadmium sponge recovery by electrowinning from residue obtained from Ni-Cd neutralization step (cold filter cake, CFC) was carried out (Moradkhani et al., 2012). In the present study, selective leaching of zinc from the hot filter cake by using sodium hydroxide (NaOH) was conducted. Through this method, zinc leaves most of the impurities such as Co, Mn, Ca etc. largely unaffected in the residue. As the scope of the present work, the effective leaching parameters such as caustic soda (NaOH) concentration (M), temperature (°C), liquid to solid ratio (L/S) and stirring rate (rpm) were investigated and the optimum condition were obtained. After leaching HFC with caustic soda, a strongly alkaline filtrate solution was obtained and stored. Finally, zinc was recovered as an active powder from this solution by alkaline electrolysis.

Experimental methods

Materials and reagent

The investigation was carried out involving hot filter cake (HFC) residue sampled from a disposal site of Iranian Zinc Mine Development Company (IZMDC). The filter cake was dried at 110 °C for 24 h and then was comminuted to a size below 100 μm. The composition of the sample was analyzed by the atomic absorption method and the result was shown in Table 1. Furthermore, the characterization of minerals was performed by X-ray powder diffraction (XRD) under the condition of Cu Kα at 40 kV and 30 mA. Sodium hydroxide with the purity of 98% from Merck (Germany) was used in leaching experiments.

Table 1. Chemical composition of filter cake produced in cobalt purification step

Elements	Zn	Co	Mn	Ca	Cd	Ni	Fe
wt,%	17.51	1.06	6.33	11	0.13	0.04	0.6

Table 2. A brief summary of alkaline leaching of metals from various materials

Research	Reagents	Material	Aim	Ref.
Leaching of oxidized zinc ore in various media	Sodium hydroxide	Oxidized zinc ore	The extraction of zinc from oxidized zinc ore	Frenay, 1975
Extraction of antimony from sulfide ores	Sodium hydroxide	Sulfide ores	Extraction of antimony	Olper, 1997
Caustic soda leach of electric arc furnace dust	Sodium hydroxide	Electric arc furnace	The extraction of zinc and lead from electric arc furnace dust	Mordodan et al. 1999
Production of Zn powder by alkaline treatment of smithsonite Zn–Pb ores	Sodium hydroxide	Smithsonite Zn–Pb ores	Production of Zn powder	Zhao and Stanforth, 2000
Pressure oxidative leaching of molybdenite in alkaline media	NaOH–NH$_4$OH or NaOH–NaOCl	Molybdenite	Recovery of molybdenum from molybdenite	Mirvaliev and Inoue, 2001
Leaching and cementation of heavy metals from electric arc furnace dust in alkaline medium	Sodium hydroxide	Electric arc furnace	The extraction of zinc from electric arc furnace dust	Orhan, 2005
Oxidative alkaline leaching of americium from simulated high-level nuclear waste sludge	Sodium hydroxide	Nuclear waste sludge	Leaching of americium	Reed et al., 2005
Alkaline leaching of zinc from electric arc furnace steel dust	Sodium hydroxide	Electric arc furnace steel dust	Zinc recovery from the EAF dust	Dutra et al., 2006
Separation of zinc from spent zinc-carbon batteries by selective leaching	Sodium hydroxide	zinc-carbon batteries	Separation of zinc from spent zinc-carbon batteries	Shin, 2008
Preparation of Mn–Zn ferrite from spent zinc–carbon batteries by alkali leaching, acid leaching and co-precipitation	Sodium hydroxide	zinc–carbon batteries	Preparation of Mn-Zn ferrite	Kim et al., 2008
Mechanochemical leaching of sphalerite in an alkaline solution containing lead carbonate	Mechanochamical leaching by Sodium hydroxide	Sphalerite	The extraction of zinc from sphalerite	Chenglong and Youcai, 2009
Alkaline leaching Zn and its concomitant metals from refractory hemimorphite zinc oxide ore	Sodium hydroxide	Refractory hemimorphite zinc oxide ore	Separation of zinc and silica	Chen et al., 2009
The kinetics of gibbsite dissolution in NaOH	Sodium hydroxide	Gibbsite	Al dissolution	Pereira et al., 2009

Metal extraction from spent sulfuric acid catalyst through alkaline and acidic leaching	Sodium hydroxide	Spent sulfuric acid catalyst	Vanadium extraction	Ognyanova et al., 2009
Pretreatment of copper anode slime with alkaline pressure oxidative leaching	Sodium hydroxide	Copper anode slime	Prepare a sample for acidic leaching	Liu et al., 2014
Desilication from titanium–vanadium slag by alkaline leaching	Sodium hydroxide	Titanium–vanadium slag	Desilication from titanium–vanadium slag	Chen et al., 2013
Ligand mediated eco-friendly leaching of zinc from spent catalyst in alkaline media	NaOH and EDTA	Spent catalyst	The extraction of zinc from spent catalyst	Mohapatra et al., 2014

The results of XRD analysis revealed that $CaSO_4 \cdot 2H_2O$, $ZnSO_4 \cdot H_2O$, $CoSO_4 \cdot 4H_2O$ and $Ca_3Mn(SO_4)_2$ were the major mineralogical phases in the filter cake (Fig. 1).
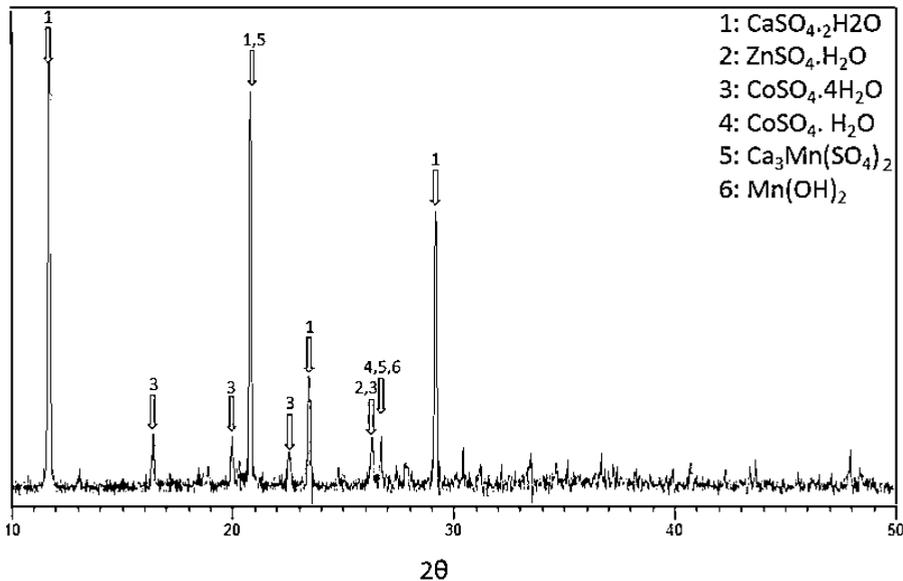
Fig. 1, X-ray diffraction analysis of hot filter cake (HFC)

Experimental procedure

As mentioned, the comminuted HFC was used in the experiments of this work. In order to separate zinc from other impurities, two-step alkaline leaching followed by electrowinning were proposed. In these experiments, the solution was heated to the desired temperature. Then, HFC sample was added to the alkaline solution. The mentioned pulp was stirred for one hour. For chemical analysis, some samples were taken from the pulp at different time intervals. The samples were immediately

vacuumed filtered, diluted and analyzed for zinc and other elements. The obtained filtrate from the optimum condition experiment was stored for electrowinning experiments. All the experiments of this study were conducted in triplicate and 95% confidence interval (CI) was used to calculate the experimental error bar in each figure. In other words, the responses are the mean ± 95% confidence intervals of three independent experiments.

First alkaline leaching tests

Alkaline leaching experiments were carried out in a beaker in thermo-controlled water bath equipped with a mechanical stirrer. The effects of sodium hydroxide concentration (M), liquid to solid ratio (L/S), temperature (°C) and stirring rate (rpm) on these leaching experiments were studied. In order to carry out the leaching experiments the desired amount of HFC was added to specific volume of caustic soda solution at the desired temperature and stirring rate (with respect to the designed parameters). The condition of parameters applied in the alkaline leaching experiments is illustrated in Table 3. In order to obtain and store the solution for the next step, an experiment was performed under the optimum condition. Hence, 50 g solid residue was dissolved in 0.5 dm^3 of 8 M sodium hydroxide (L/S of 10:1) at 75 °C and 600 rpm for one hour. After completion of the leaching, the hot slurry was filtered. The concentrations of Zn^{2+}, Mn^{2+} and Co^{2+} in the leaching filtrate and solid residue were characterized by the atomic absorption analysis.

Table 3. The parameter values for alkaline leaching of cobalt filter cake

Parameter	Value
NaOH concentration (M)	4, 6, 8, 10
Temperature (°C)	25, 50, 60, 90
Phase ratio (v/w)	4, 6, 8, 10
Steering speed(rpm)	400, 600, 800, 1000

Second alkaline leaching tests

The aim of second alkaline leaching is to reduce the amount of zinc in residue as much as possible and increase the overall zinc recovery (Moradkhani et al., 2012). Under optimum conditions obtained in the first alkaline leaching experiments, the solid residue obtained from the first stage containing 3-5% Zn was again treated with NaOH solution. In the second alkaline leaching experiments, 50 g solid residue was dissolved in 0.5 dm^3 of 8 M sodium hydroxide at 95 °C and 600 rpm for 1 h. The concentration of Zn^2, Co^{2+} and Mn^{2+} in the leaching liquor and solid residue were characterized by atomic absorption analysis. It is noteworthy that in lab-scale experiments, the first alkaline leaching solid residue is treated with fresh NaOH solution. However, in the larger scale operations, the spent electrolyte of zinc alkaline electrowinning process is

recycled to the second leaching step to recover the remaining zinc of the residue. Furthermore, in pilot-scale operations the alkaline filtrate obtained from the second leaching step is also recycled to the first alkaline leaching step. Therefore, the second alkaline leaching filtrate solution is contacted with HFC in the first alkaline leaching tank. At the end of the laboratory experiments, the zinc alkaline solution is stored for next studies.

Electrowinning experiments

Based on the reported investigations (Gurmen and Emre, 2003; Kim et al., 2008; Zhao and Stanforth, 2000), zinc present in the highly alkaline filtrate solution can be recovered in an active powder form by zinc alkaline electrowinning. Alkaline electrowinning has many advantages versus sulfate electrowinning such as lower electricity consumption and production of a highly active zinc powder (Moradkhani et al., 2012). In order to perform the electrowinning experiments, the first and second alkaline leaching were combined. In this step, under the optimal conditions, the zinc powder was produced with high purity. Stainless steel electrodes formed the anode and cathode in this study. Finally, the deposited zinc metal powder could easily be removed from the electrode. It was then washed with warm distilled water and dried at 50°C, weighed and analyzed.

Result and discussion

Alkaline leaching experiment and recovery of zinc

Alkaline leaching with sodium hydroxide can be exploited in separating cations, since only certain metal oxides such as zinc oxide and aluminum oxide show amphoteric behavior and dissolve in it (Moradkhani et al., 2012). With respect to this behaviour, sodium hydroxide was chosen as the selective alkaline agent because it thermodynamically leaches zinc from the solid state. However, it cannot dissolve nickel and cadmium under atmospheric condition. In this study, the effects of NaOH concentration, leaching temperature, L/S, leaching time and stirring rate on the alkaline leaching extraction of zinc were investigated. As mentioned before, the zinc content of highly alkaline filtrate solution can be recovered in the active powder form by zinc alkaline electrowinning. In order to perform the electrowinning experiments, the first and second alkaline leaching were combined. In the subsequent steps, a filtrate solution obtained from the first alkaline leaching step containing about 20 g/dm^3 Zn was electrolyzed to recover zinc in an active powder form.

Effect of concentration of sodium hydroxide

In order to investigate the effect sodium hydroxide, four concentrations (4, 6, 8, and 10 M) were used in the experiments. The metal recovery percentage and its concentration leached out into solution vs. sodium hydroxide concentration were

showed in Fig. 2. It is noteworthy that the recovery of impurities such as cobalt and manganese due to approximately zero recovery of them is not shown in the figures of this study.
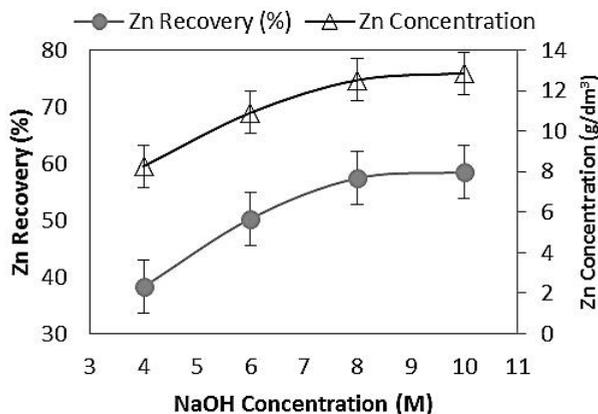
Fig. 2. Effect of sodium hydroxide concentration on metal leaching extraction in the first step (leach cond. temperature 25 °C, L/S 10:1, rpm 600, time 1 h)

The results showed that the zinc dissolution depends on NaOH concentration and increases with its enhancement. This result indicate high selective alkaline leaching of Zn over Co and Mn. This was because in the highly alkaline media, zinc exists in solution as a complex anion of ZnO_2^{2-}. Nevertheless, NaOH cannot bring Co and Mn from solid state to the solution and form water-soluble complexes.

Based on the experimental result, the zinc extraction efficiency after 1 h at different sodium hydroxide concentrations in the first leaching step was found to be 38.2, 50.6, 57.4 and 58.4%, respectively. Therefore, the NaOH concentration of 8 M is selected as the optimum condition. Furthermore, with respect to the result obtained in the second alkaline leaching under the condition applied in the first leaching, the overall zinc extraction efficiency of two steps was found to be 88.5 %.

Effect of L/S

Figure 3 shows the zinc recovery as a function of L/S at 25 °C, NaOH concentration of 8 M, rpm of 600 for 1 h. These results showed the obvious effect of L/S ratio on the zinc leaching recovery. In other words, with increasing ratio of NaOH solution to solid in the sample, dissolution of zinc can be facilitated. As shown in this figure, the zinc recovery enhanced from 38.8% at L/S of 4 to 57.4% at L/S of 10. Therefore, L/S=10:1 with the maximum zinc recovery is considered as the optimum phase to liquid ratio.
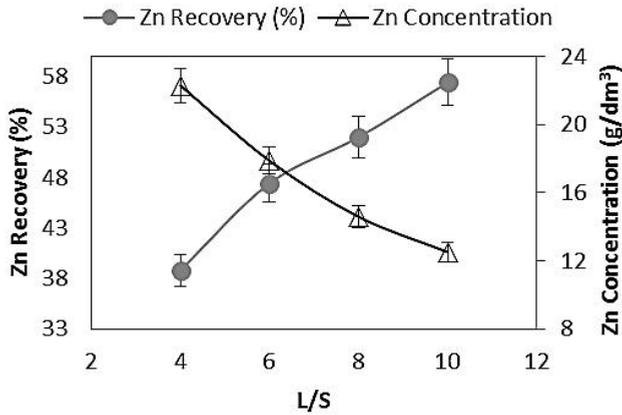
Fig. 3. Effect of L/S on Zn leaching recovery (leaching conditions: temperature 25 °C, NaOH concentration 8 M, time 1 h)

Effect of leaching temperature

The plot of the temperature vs. metal dissolution is illustrated in Fig. 4. As seen in this figure, the recovery of zinc increased as the temperature increased from 25 to 95°C. These results show that the temperature has strong effect on the Zn leaching recovery. The Zn leaching recovery increased from 57.4% at 25 °C to 88.5% at 95 °C. Despite the high recovery of zinc at 95°C, some amount of impurities such as Co is dissolved. Therefore, to prevent them entering the electrowinning process, 75°C was chosen as the optimum temperature of the first leaching step. Furthermore, the solution at 95 °C is more viscose to such extent that filtration of slurry is difficult and slow (Moradkhani et al., 2012).
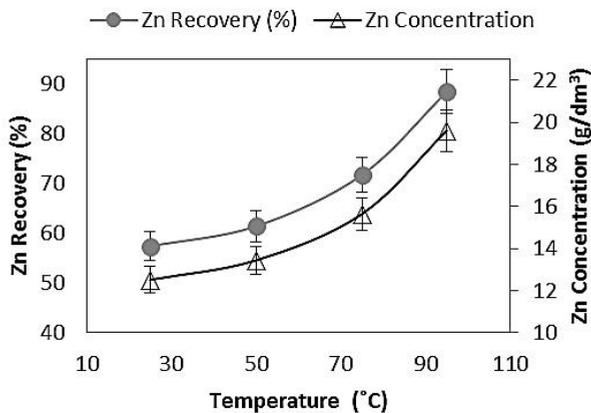
Fig. 4. Effect of temperature on Zn leaching recovery (leach conditions: NaOH concentration 8 M, L/S 10:1, rpm 600, time 1 h)

Effect of stirring rate

In order to evaluate the effect of stirring speed on the zinc leaching, a series of experiments were carried out under the condition: NaOH concentration 8 M, temperature 95°C, time 1 h and L/S 10:1. The effect was studied at four levels; 400, 600, 800 and 1000 rpm. The obtained results are shown in Fig. 5. As shown in this figure, the stirring rate had not considerable effect on zinc dissolution. On the other hand, zinc leaching recovery and concentration was enhanced from 85.1% and 18.7 g/dm^3 at 400 rpm to 88.6% and 19.8 g/ dm^3 at 600 rpm, and no significance changes occurred between 600 rpm (88.5% recovery) and 1000 rpm (89.1% recovery). Therefore, 600 rpm was selected as the optimum stirring speed to confirm enough pulp mixing. Hence, with respect to the results of pervious sections, the optimum conditions of alkaline leaching are found to be sodium hydroxide concentration 8 M, temperature 95°C, time 1h, stirring rate 600rpm and L/S 10:1, with the zinc leaching recovery of 88.5%.
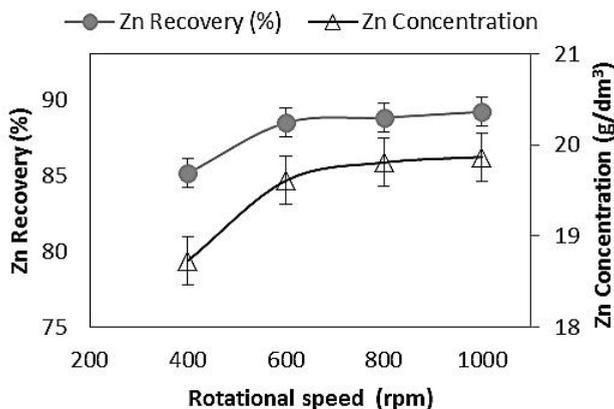
Fig. 5. Effect of stirring rate on Zn leaching recovery (leach conditions: temperature 95°C, NaOH concentration 8 M, time 1 h, L/S 10:1)

Thermodynamic aspects of zinc dissolution

Some researches, reported that a concentrated NaOH solution could selectively dissolve zinc from solid state and leave behind iron and manganese in the leach residue (Cusanelli et al., 1973; Eacott et al., 1984; Merrill and Lang, 1965; Shin, 2008; Valdez and Dean, 1975). Thermodynamic data (ΔG of -62,76 kJ at 75°C) showed the dissolution of zinc from zinc oxide occurs due to reaction:

$$ZnO + 2NaOH = 2Na^+ + ZnO_2^{-2} + H_2O. \tag{1}$$
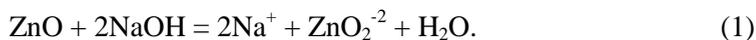
This equation was also used in the Eh-pH diagrams of this study. Figure 6 illustrates the Eh-pH diagrams for the zinc-water system to show the effect of

temperature. As seen in this figure, at 25°C the lines related to the species of $ZnOH_2$ and ZnO_2^{2-} shifts to the right side meaning the need for a higher pH or concentrated NaOH to dissolve a high concentration of zinc(II) in the form of ZnO_2^{2-} ions (Shin, 2008). The same results have been reported by Shin et al. (2009).
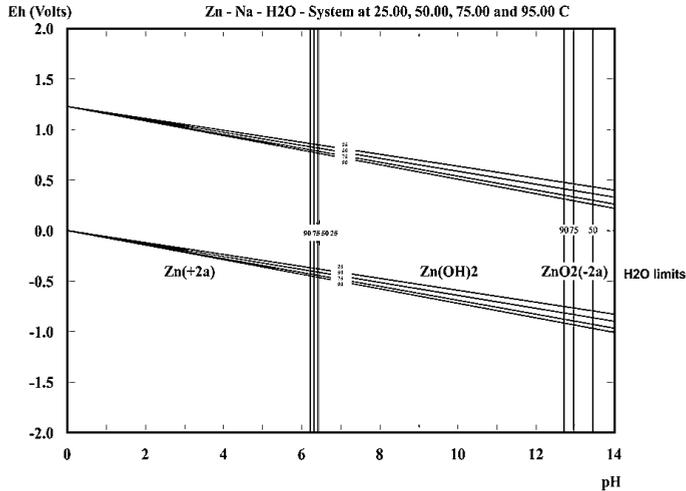
Fig. 6. Potential-pH diagram for zinc-water system in the temperature range 25–95 °C, {Zn} 0.3 M and {NaOH} 8 M (HSC 5.1 software)
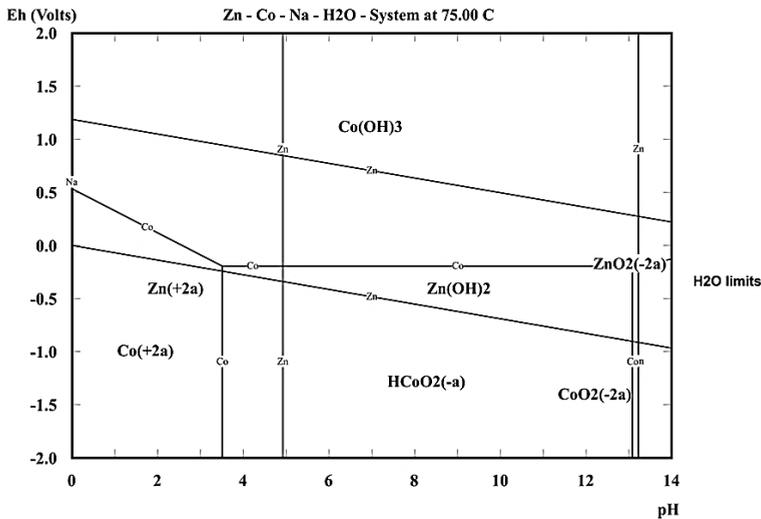
Fig. 7. Potential-pH diagram for zinc-cobalt-water system at 75°C and {Zn} 0.3 M, {Co} 0.01 M and {NaOH} 8 M (HSC 5.1 software)

With respect to Fig. 6, the enhancement of the temperature shifts the aforementioned lines to the left side. This shifting means that at relatively low pH values, dissolving of ZnO as ZnO_2^{2-} can easily occur. Furthermore, in order to prove

that the complete dissolution of the impurities such as cobalt, manganese etc. is not possible, the Eh-pH diagram of Zn-Co system in alkaline water was also constructed (Fig. 7). It is noteworthy that because of ignoring the complexity of diagrams and similar results, the cobalt-zinc system was only plotted. As seen in Fig. 7, a very thin area has been dedicated to CoO^{2-} at high pH values indicating a low possibility of cobalt dissolution at 75°C. The same result was obtained for the manganese-zinc system.

Zinc electrowinning

Some studies have been carried out on alkaline zinc electrowinning by Gurmen and Emre (2003) as well as St-Pierre and Piron (1986). They showed that electrowinning of zinc from alkaline solution is economical. In this study, a series of experiments was performed to investigate the electrolysis of zinc from alkaline solution obtained from two-step alkaline leaching. With respect to the aforementioned discussions, due to very low solubility of the metals except zinc in NaOH, the concentrations of the impurities entering the electrowinning cell are in their standard limits. Hence, there was no specific problem in the EW cell.

Figure 8 illustrates the effect of the electrolysis time on Zn(II) concentration in the spent electrolyte of the EW cell. As seen in this figure, the concentration of zinc in the solution falls down indicating that zinc ions deposit on the cathode. Thus, the deposition of zinc was approximately completed within 10 h at 350 A/m^2 for the electrolyte solution containing 19.6 g/dm^3 Zn. Under his condition spongy and powdery zinc deposits was obtained with high purity (over 97%) on the cathode. Subsequently, it was removed easily from the cathode by scraping the metal. The main impurity of this powder is Na_2O resulting from the NaOH usage. To increase the purity of zinc (to over 99%) in the obtained powder, the product was washed in distilled hot water. The aforementioned composition of the zinc powders meets the industrial standards and can be used for gold and silver cementation.
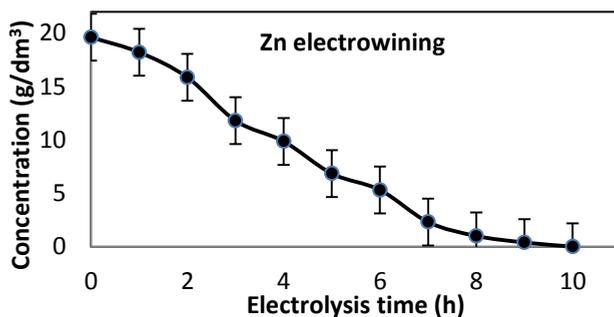
Fig. 8. Concentration versus electrolysis time

Finally, with respect to the stages developed in this study, a simplified flow diagram (Fig. 9) was proposed for the production of zinc powder from the cobalt-zinc plant residue (Co-ZPR), showing the sequential stages according to the conditions obtained in each stage.

Fig. 9. Flow diagram for recovery of zinc from Co filter cake of zinc plant residues

Conclusion

In this study, the production of zinc powder from a Co-Zn plant residue (Co-ZPR) using selective alkaline leaching followed by electrowinning was investigated. With respect to the results of the leaching steps, 88.5% of zinc was selectively leached out in two steps, leaving Co and Mn intact in the solid residue. The condition under which the high leaching recovery of zinc occurred was found to be NaOH concentration 8 M, temperature 75 °C, leaching time 1 h, L/S 10 and rpm 600. Furthermore, a thermodynamic evaluation showed that zinc was leached as an anionic complex (ZnO_2^{2-}) in caustic solution. As a result, the alkaline filtrate solution obtained from the leaching steps could be electrolyzed to produce zinc in a powder form. Therefore, the deposition of zinc was approximately performed in 10 h at 350 A/m^2 with an electrolyte containing 19.6 g/dm^3 Zn. Under this condition, spongy and powdery zinc deposits were obtained. The high purity (over 99%) of zinc powders was obtained after washing the powders. Finally, with respect to the stages developed in this study,

a simplified flow diagram was proposed for the production of the zinc powder from the Co-Zn plant residue.

Acknowledgment

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